

## SPINOSAD (203)

### IDENTITY

Spinosad is a naturally derived fermentation product, which has demonstrated insect control activity against a large number of pests including members of the insect orders Lepidoptera, Coleoptera and Thysanoptera. The product is isolated from actinomycetes *Saccharopolyspora spinosa* and contains a mixture of two structurally similar molecules which are both active insecticidally and have been designated spinosyn A and spinosyn D.

ISO common name: spinosad. Spinosad is a mixture of spinosyn A and spinosyn D.

Chemical name mixture of 50-95% of spinosyn A

IUPAC: (2*R*,3*aS*,5*aR*,5*bS*,9*S*,13*S*,14*R*,16*aS*,16*bR*)-2-(6-deoxy-2,3,4-tri-*O*-methyl- $\alpha$ -L-mannopyranosyloxy)-13-(4-dimethylamino-2,3,4,6-tetra-deoxy- $\beta$ -D-erythro-pyranosyloxy)-9-ethyl-2,3,3*a*,5*a*,5*b*,6,7,9,10,11,12,13,14,15,16*a*,16*b*-hexadeca-hydro-14-methyl-1*H*-8-oxacyclododeca[*b*]-as-indacene-7,15-dione and 50-5% spinosyn D

(2*S*,3*aR*,5*aS*,5*bS*,9*S*,13*S*,14*R*,16*aS*,16*bR*)-2-(6-deoxy-2,3,4-tri-*O*-methyl- $\alpha$ -L-mannopyranosyloxy)-13-(4-dimethylamino-2,3,4,6-tetra-deoxy- $\beta$ -D-erythro-pyranosyloxy)-9-ethyl-2,3,3*a*,5*a*,5*b*,6,7,9,10,11,12,13,14,15,16*a*,16*b*-hexadeca-hydro-4,14-dimethyl-1*H*-8-oxacyclododeca[*b*]-as-indacene-7,15-dione

CAS: (2*R*,3*aS*,5*aR*,5*bS*,9*S*,13*S*,14*R*,16*aS*,16*bR*)-2-[(6-deoxy-2,3,4-tri-*O*-methyl- $\alpha$ -L-mannopyranosyl)oxy]-13-[[[(2*R*,5*S*,6*R*)-5-(dimethylamino)tetrahydro-6-methyl-2*H*-pyran-2-yl]oxy]-9-ethyl-2,3,3*a*,5*a*,5*b*,6,9,10,11,12,13,14,16*a*,16*b*-tetradeca-hydro-14-methyl-1*H*-as-indaceno[3,2-*d*]oxacyclododecin-7,15-dione mixture with

(2*S*,3*aR*,5*aS*,5*bS*,9*S*,13*S*,14*R*,16*aS*,16*bS*)-2-[(6-deoxy-2,3,4-tri-*O*-methyl- $\alpha$ -L-mannopyranosyl)oxy]-13-[[[(2*R*,5*S*,6*R*)-5-(dimethylamino)tetrahydro-6-methyl-2*H*-pyran-2-yl]oxy]-9-ethyl-2,3,3*a*,5*a*,5*b*,6,9,10,11,12,13,14,16*a*,16*b*-tetradeca-hydro-4,14-dimethyl-1*H*-as-indaceno[3,2-*d*]oxacyclododecin-7,15-dione

CAS No.: Spinosyn A: 131929-60-7

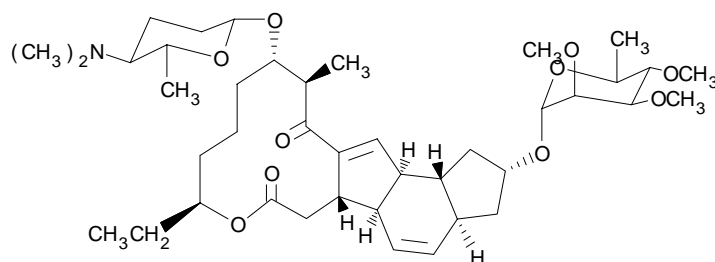
Spinosyn D: 131929-63-0

Synonyms DE-105; XDE-105; DE-105 Factors A and D

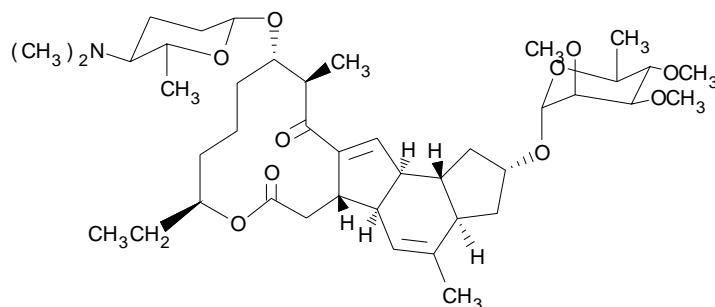
Trade names: Success, Tracer, Spintor, Spinoace, Boomerang, Laser, Extinosad

Structural formula:

Spinosyn A



Spinosyn D



Molecular formula: Spinosyn A:  $C_{41}H_{65}NO_{10}$   
 Spinosyn D:  $C_{42}H_{67}NO_{10}$

Molecular weight: Spinosyn A: 731.98  
 Spinosyn D: 745.99

### Physical and chemical properties

#### Pure active ingredient

	<u>Spinosyn A</u>	<u>Spinosyn D</u>	<u>Reference</u>
Appearance	Light grey-white solid		
Vapour pressure, mPa (25°C)	$3.0 \times 10^{-5}$	$2.0 \times 10^{-5}$	Chakrabarti, 1991a,b
Melting point	84 to 99.5°C (98.3% purity)	161.5 to 170°C (98% purity)	Jones-Jefferson, 1994a
Octanol/water partition coefficient			Jones-Jefferson, 1994a Morrissey, 1994a,b
Log $P_{ow}$ at 23°C (water)	3.9	4.4	
Log $P_{ow}$ at 23°C (pH 5)	2.8	3.2	
Log $P_{ow}$ at 23°C (pH 7)	4.0	4.5	
Log $P_{ow}$ at 23°C (pH 9)	5.2	5.2	
Water solubility			Jones-Jefferson, 1994a,b Heimerl, 1994
water, mg/l at 20°C	89	0.50	
pH 5, mg/l at 20°C	290	29	
pH 7, mg/l at 20°C	235	0.33	
pH 9, mg/l at 20°C	16	0.053	Heimerl, 1993
Solvent solubility			Jones-Jefferson, 1994a,b
dichloromethane, g/l at 20°C	525	448	
methanol, g/l at 20°C	190	2.5	
acetone, g/l at 20°C	168	10	
acetonitrile, g/l at 20°C	134	2.6	
amyl acetate, g/l at 20°C	37	23	
hexane, g/l at 20°C	4.5	0.74	
1-octanol, g/l at 20°C	9.3	1.3	
toluene, g/l at 20°C	457	152	
isopropyl alcohol, g/l at 20°C	40	1.3	
ethyl acetate, g/l at 20°C	194	19	Richardson and Comb, 1999a,b
n-heptane, g/l at 20°C	12.4	0.3	
xylene g/l at 20°C	>250	64	

	<u>Spinosyn A</u>	<u>Spinosyn D</u>	<u>Reference</u>
Hydrolysis at 25°C, sterile buffer, dark			Saunders <i>et al.</i> , 1994a
pH 5	not observed in 30-day test	not observed in 30-day test	
pH 7	not observed in 30-day test	not observed in 30-day test	
pH 9 half life	approx. 100-300 days	approx. 100-300 days	
Photolysis - half-life for degradation in dilute aqueous sterile buffer at pH 7, June-July, Indiana USA, 39.9°N.	22.3 h	19.7 h	Saunders and Powers, 1994b
Dissociation constant (determined by capillary electrophoresis)			
pKa (20°C)	8.10	7.87	Gluck, 1994a,b
Equivalent Ka	$7.94 \times 10^{-9}$	$1.35 \times 10^{-8}$	

Chakrabarti (1991a) measured the vapour pressure of spinosyn A (99.9% pure) over the range 33.1°C to 49.1°C by the Knudsen-effusion weight loss method. The vapour pressure at 25°C ( $2.4 \times 10^{-10}$  mm Hg or  $3.0 \times 10^{-5}$  mPa) was obtained by extrapolation using the Clausius-Clapeyron equation. Chakrabarti (1991b) measured the vapour pressure of spinosyn B (99+%) in the same way over the range 38.9°C to 55.1°C and obtained a value of  $1.6 \times 10^{-10}$  mm Hg or  $2.0 \times 10^{-5}$  mPa at 25°C.

Saunders *et al.* (1994) measured the hydrolysis rates of [ $^{14}\text{C}$ ]spinosyns A and D in sterile buffers of pH 5, 7 and 9 held at 25°C in the dark for 30 days. Spinosyns A and D were dissolved in 0.01M buffers at 2 mg/ml each, with 0.5% acetonitrile to maintain solubility, and solutions were analysed by HPLC at intervals, but hydrolysis was insufficient to be observed. It did occur at pH 9, but the estimation of accurate half-lives was difficult because the results were variable and measurements were made for only 30 days. Estimated half-lives were 100-300 days. Hydrolysis products, shown to result from loss of the amino sugar and water, were minor and not fully identified.

#### Technical material

Minimum purity: minimum 85% (w/w), mixture of spinosyn A and spinosyn D in a ratio between 95:5 and 50:50.

Melting point range: melting point minimum 112°C, maximum 123°C, determined as the endothermic peak by differential scanning calorimetry for 88% purity (spinosyn A + spinosyn B). Exothermic decomposition temperature 173°C. (Jones-Jefferson, 2000).

#### Formulations

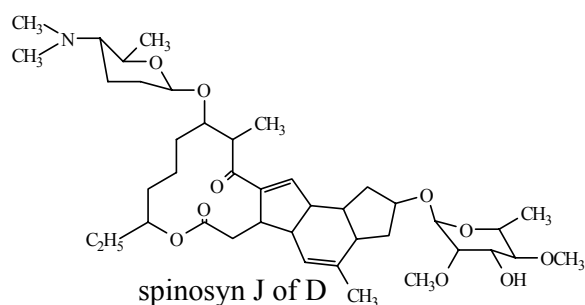
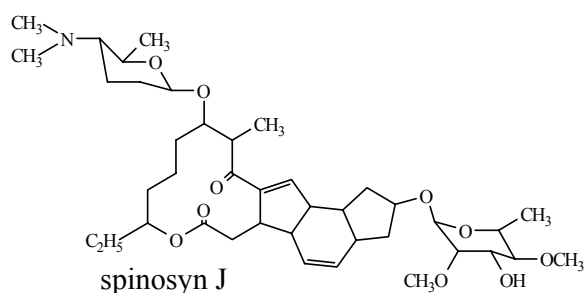
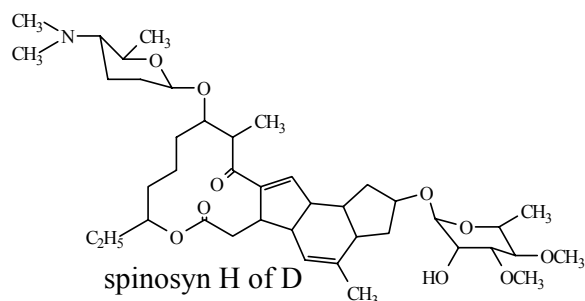
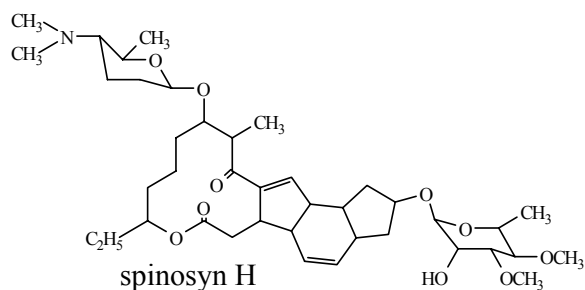
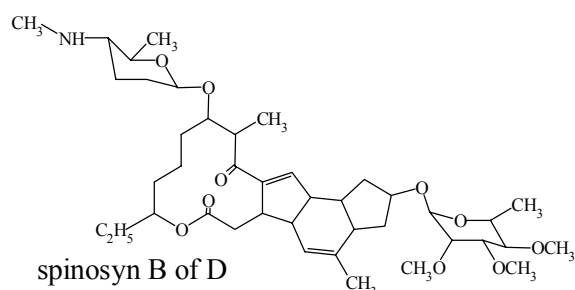
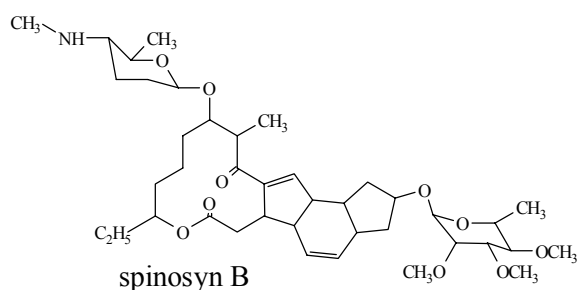
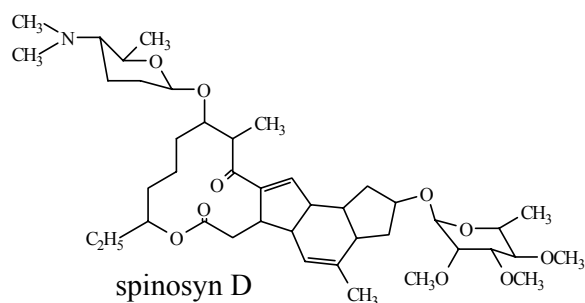
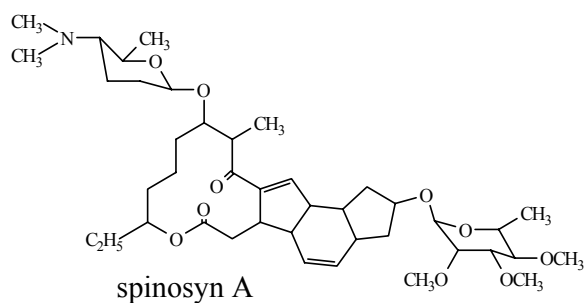
Spinosad is available as SC, WG and fly bait formulations:

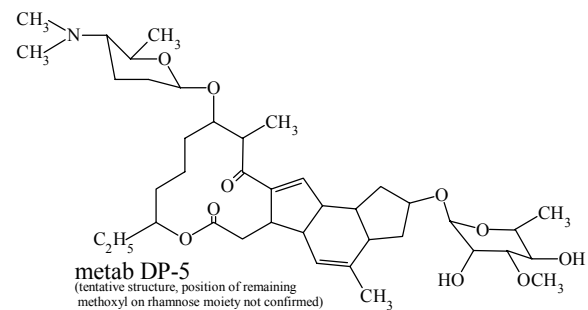
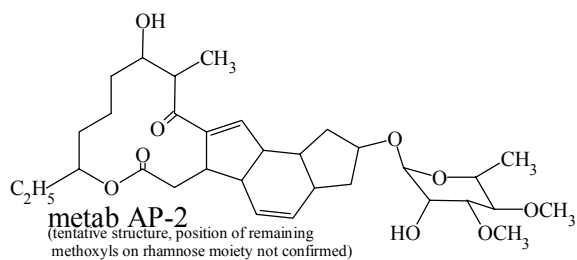
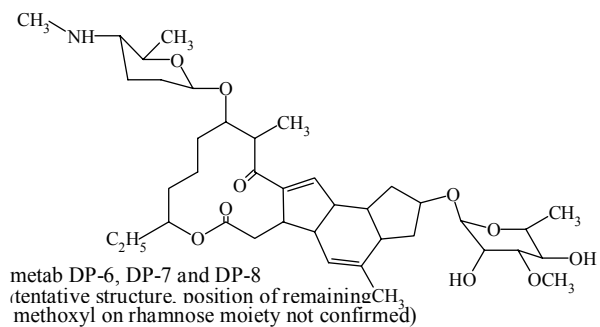
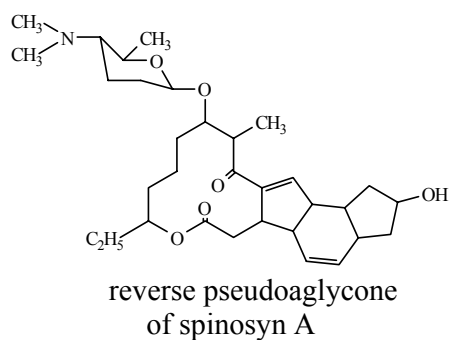
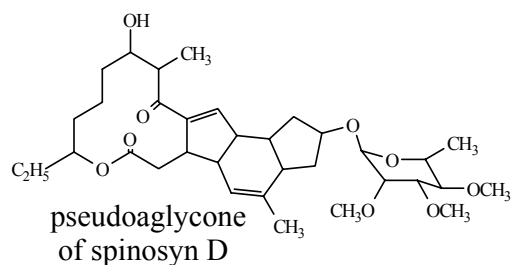
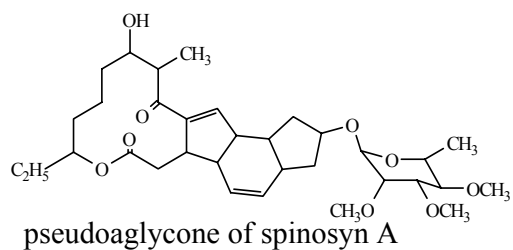
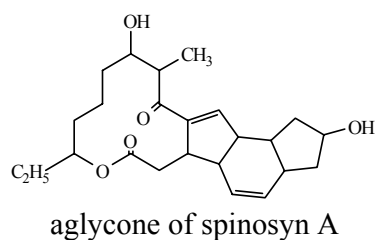
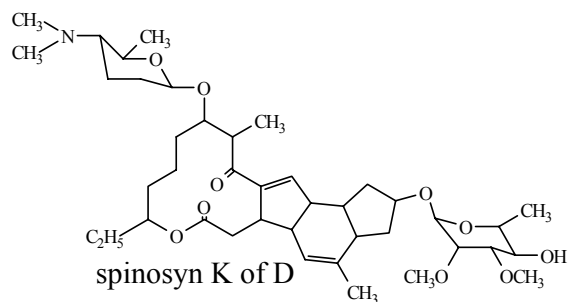
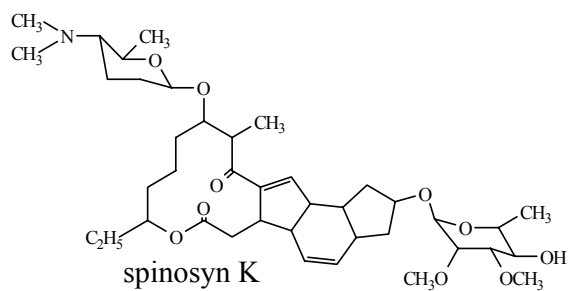
- suspension concentrate (SC) formulations containing 25, 120, 240 or 480 g/l.
- water dispersible granule (WG) formulations containing 250, 780, or 800 g/kg.
- fly bait containing 0.008% spinosad.

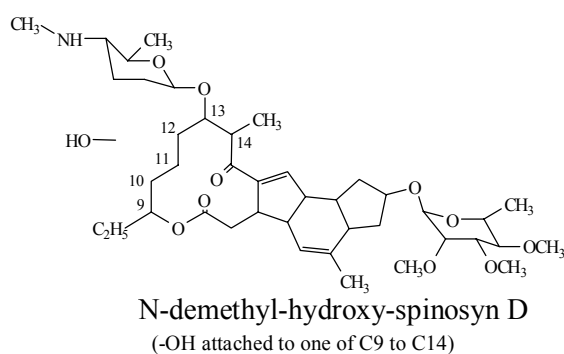
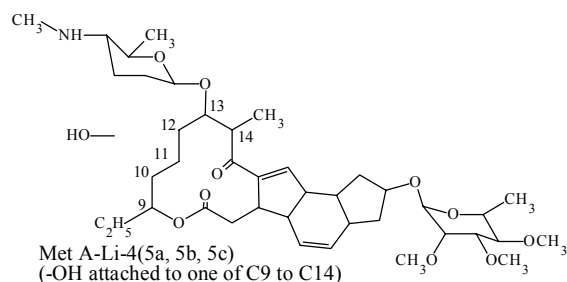
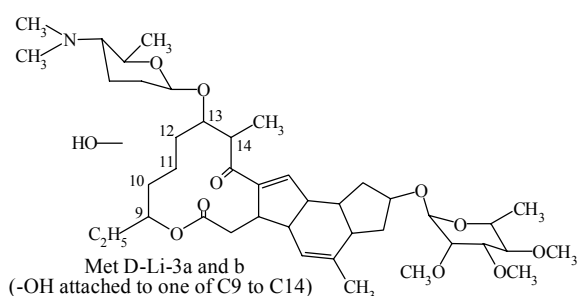
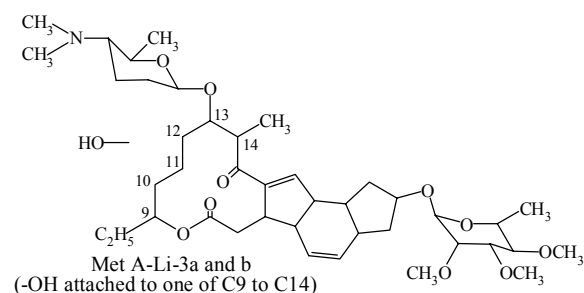
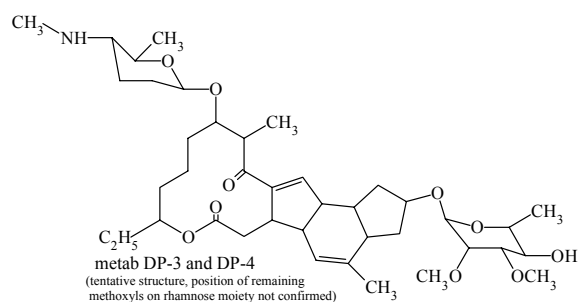
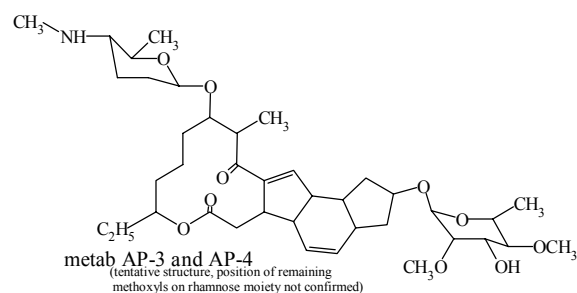
## METABOLISM AND ENVIRONMENTAL FATE

Radiolabelled spinosyns A and D were produced by fermentation using  $[1,2-^{14}\text{C}]$ acetate as a carbon source, which resulted in material reasonably uniformly labelled at 23 carbons in the aglycone ring system. The amino and rhamnose sugars do not contain the  $^{14}\text{C}$  label.

### Structures of compounds appearing during metabolism







## Animal metabolism

The Meeting received information on the results of studies on lactating goats and laying hens given oral doses of spinosyns and on lactating goats treated dermally with spinosyns.

**Goats.** The tissues, milk and excreta of two lactating dairy goats, one dosed with spinosyn A, the other with spinosyn D, body weights 47 and 43 kg respectively were analysed for residues (Rainey, 1994a). The goats were dosed orally for 3 days by capsule with a nominal 25 mg/day [ $^{14}$ C]spinosyn equivalent to 10 ppm in the feed, The feed intake was 2.5-3.0 kg/animal/day, and the milk averages 0.98 and 1.36 kg per day. The milk and excreta were collected throughout the study and the goats slaughtered within 24 hours of the last dose. Tissue samples (composites of equal amounts of longissimus dorsi, semimembranosus and triceps muscles, and of omental and perirenal fat, and kidneys and liver) were analysed. The results are shown in Table 2.

A considerable proportion of the residues, 45% for spinosyn A and 20% for spinosyn D, was found in the tissues and milk - predominantly the fat (Table 1), and excretion of the  $^{14}$ C was mainly via the faeces, 61% from A and 76% from D were found in excreta and intestine.

Spinosyns A and D were the main components of the residue in the tissues and milk, especially in the fat, and some of the metabolites were characterized without being fully identified. Metabolites Met A-Li-3, Met A-Li-4 and Met D-Li-3 were shown to be hydroxylated in the macrolide

ring between C9 and C14. Met A-Li-4 had also been *N*-demethylated. Other metabolites were investigated but insufficient information was available to propose structures.

Table 1. Disposition of  $^{14}\text{C}$  in goats dosed with a nominal 25 mg/day [ $^{14}\text{C}$ ]spinosyn (spinosyn A in one goat, spinosyn D in the other) for 3 days (Rainey, 1994a).

Sample	% of administered $^{14}\text{C}$	
	spinosyn A dose	spinosyn D dose
Milk	1.8	0.69
Faeces	38	67
Urine	1.1	0.21
Composite fat	33	17
Composite muscle	8.7	2.7
Kidney	0.27	0.07
Liver	3.2	0.67
Rumen and contents	8.0	6.3
Intestine with contents	22	9.4
TOTAL	116	104

Table 2. Distribution of metabolites in the tissues and milk of lactating goats dosed orally for 3 days by capsule at a rate equivalent to 10 ppm spinosyn A or D in the feed (Rainey, 1994a). TRR is the total radioactive residue in the tissue or milk.

Compound or fraction	Fat		Muscle		Kidney		Liver		Milk	
	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg
<b>SPINOSYN A</b>										
Spinosyn A	86	3.1	50	0.15	35	0.34	30	0.47	71	0.45
Spinosyn B	0.7	0.026	8.3	0.025	10	0.099	2.9	0.046	1.9	0.012
Met A-Li-3a	2.7	0.095	8.3	0.025	10	0.10	7.7	0.12	6.3	0.040
Met A-Li-3b	1.3	0.046	4.0	0.012	6.1	0.059	5.0	0.079	3.8	0.024
Met A-Li-4(5a)			13	0.040	16	0.15	3.0	0.047	1.6	0.010
Met A-Li-4(5b)			<sup>1</sup>	<sup>1</sup>	<sup>1</sup>	<sup>1</sup>	4.1	0.065	1.7	0.011
Met A-Li-4(5c)							4.1	0.064	1.4	0.009
Aq sol + extr tissue + misc	9.1	0.33	16	0.046	22	0.20	40	0.63	11	0.07
Total measured $^{14}\text{C}$		3.6		0.30		0.97		1.6		0.63
<b>SPINOSYN D</b>										
Spinosyn D	85	1.5	57	0.063	40	0.12	20	0.10	81	0.13
Spinosyn B of D	1.1	0.020	12	0.013	15	0.046	4.4	0.022	2.5	0.004
Met D-Li-1	2.5	0.046	2.7	0.003			5.6	0.028		
Met D-Li-3a					3.0	0.009	2.2	0.011		
Met D-Li-3b	1.6	0.030	7.3	0.008	13	0.038	6.4	0.032	5.6	0.009
Aq sol + extr tissue + misc	11	0.19	24	0.026	29	0.087	57	0.28	9.4	0.015
Total measured $^{14}\text{C}$		1.8		0.11		0.30		0.50		0.16

<sup>1</sup> Metabolite A-Li-4 (5b) in muscle and kidney is included with Met A-Li-4 (5a)

In a dermal application metabolism study Burnett *et al.* (1999) treated a lactating goat with [ $^{14}\text{C}$ ]spinosyn A in a solution of isopropyl myristate and oleic acid at 18 mg ai/kg bw, and a second goat with [ $^{14}\text{C}$ ]spinosyn D at 4.1 mg ai/kg bw. The dose was applied by pouring the weighed solution down the midline of the back from withers to tail. Milk and excreta were collected during the treatment period and for 4 days until the animals were slaughtered. Teats were cleaned before milking to prevent contamination of milk with spinosyns remaining on the animals' backs. The goats weighed 50 and 74 kg respectively. The distribution of  $^{14}\text{C}$  among the samples is shown in Table 3 and among the identified compounds in Table 4.

Residues were higher in the liver and fat than in other tissues, and the parent compound was dominant, particularly in the fat and milk. Metabolites were produced by *N*-demethylation and hydroxylation of the macrolide ring, a process already identified in the oral dosing study.

Table 3. Levels of  $^{14}\text{C}$  in the tissues and milk and percentages of the applied dose excreted by goats treated dermally with [ $^{14}\text{C}$ ]spinosyn A or D at 18 or 4.1 mg ai/kg bw respectively and slaughtered 4 days later (Burnett *et al.*, 1999).

Sample	spinosyn A		spinosyn D	
	mg/kg	% of dose	mg/kg	% of dose
Liver	1.7		0.39	
Kidneys	0.86		0.17	
Muscle, loin	0.30		0.041	
Muscle, rump	0.27		0.043	
Muscle, shoulder	0.25		0.050	
Fat, abdominal	0.81		0.23	
Fat, perirenal	1.1		0.21	
Bile	5.5		1.9	
Faeces		2.3		2.6
Urine (3rd day only)		<0.1		<0.1
Milk total		0.05		0.05
Milk, 7 h	0.017		0.021	
Milk, 19 h	0.079		0.061	
Milk, 31 h	0.13		0.077	
Milk, 43 h	0.20		0.093	
Milk, 55 h	0.32		0.092	
Milk, 67 h	0.51		0.074	
Milk, 79 h	0.54		0.076	
Milk, 92 h	0.52		0.085	

mg/kg:  $^{14}\text{C}$  expressed as spinosyn A or D

Table 4. Distribution of metabolites in the tissues and milk of goats treated dermally with [ $^{14}\text{C}$ ]spinosyn A or D at 18 or 4.1 mg ai/kg bw respectively and slaughtered 4 days later (Burnett *et al.*, 1999).

Compound	Fat		Muscle		Kidney		Liver		Milk	92-h
	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg
SPINOSYN A										
Spinosyn A	77	0.74	57	0.16	35	0.30	46	0.78	68	0.35
Spinosyn B	5.0	0.047	8.7	0.024	14	0.12	9.4	0.16	5.6	0.029
hydroxy-spinosyn A <sup>1</sup>	9.5	0.090	8.7	0.024	14	0.12	10	0.17	7.3	0.038
N-demethyl-hydroxy-spinosyn A <sup>2</sup>	1.2	0.012	9.7	0.026	20	0.18	13	0.22	6.9	0.036
unidentified	7		16		18		21		13	
SPINOSYN D										
Spinosyn D	73	0.16	49	0.022	32	0.055	27	0.10	66	0.056
Spinosyn B of D	4.2	0.009	13	0.006	21	0.035	17	0.064	7.7	0.007
Hydroxy-spinosyn D <sup>3</sup>	7.9	0.017	9.6	0.004	13	0.022	12	0.046	6.8	0.006
N-demethyl hydroxy-spinosyn D	0.8	0.002	4.8	0.002	9.6	0.016	7.5	0.029	1.7	0.001
unidentified	13		22		23		36		16	

<sup>1</sup> Hydroxy-spinosyn A: same as Met A-Li-3a,b

<sup>2</sup> N-demethyl-hydroxy-spinosyn A : same as Met A-Li-4(5a,5b,5c)

<sup>3</sup> Hydroxy-spinosyn D: same as Met D-Li-3a,b

Metabolic pathways of spinosyn A in goats are shown in Figure 1.



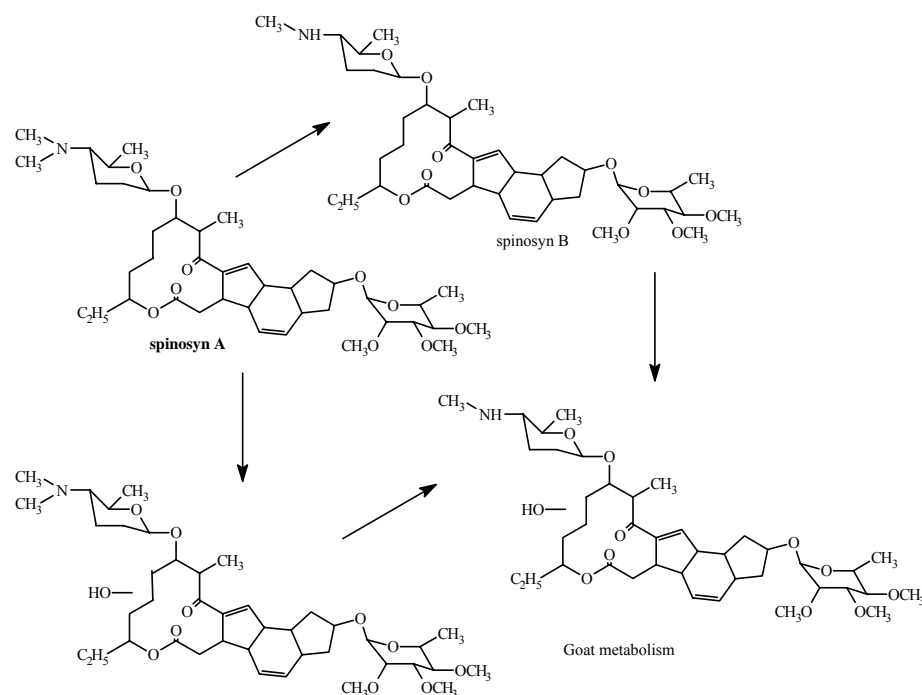


Figure 1. Metabolism of spinosyn A by goats. Spinosyn D metabolism parallels that of spinosyn A.

**Hens.** Residues were measured in the tissues, eggs and excreta of 10 Leghorn laying hens, each weighing 1.4 kg, dosed orally by capsule with 0.94 mg [ $^{14}\text{C}$ ]spinosyn A, equivalent to 10 ppm spinosyn A in the feed, daily for 5 days (Magnussen and Castetter, 1994). The feed intake was 94 g/bird/day. Eggs and excreta were collected throughout, and the birds were slaughtered within 24 hours of the last dose. Another 10 laying hens were dosed with [ $^{14}\text{C}$ ]spinosyn D in a parallel study.

For spinosyn A approximately 73% of the applied  $^{14}\text{C}$  was accounted for (69% in the excreta, 0.77% in eggs, 1.4% in fat, 0.68% in liver, 0.53% in muscle and 0.12% in kidney), and for spinosyn D approximately 82% (78% in the excreta, 0.6% in eggs, 0.8% in fat, 1.2% in liver, 0.5% in muscle and 0.2% in kidney).  $^{14}\text{C}$  residues in eggs were apparently still increasing at the end of the study, and from day 2 to 6 were 0.014, 0.082, 0.19, 0.32 and 0.38 mg/kg expressed as spinosyn A, and 0.019, 0.073, 0.14, 0.22 and 0.32 mg/kg as spinosyn D.

The highest residues occurred in the fat, most of which were accounted for by the parent compounds, and they were also major or important parts of the residue in muscle and eggs. Substantial metabolism occurred in the liver where metabolites were produced by *N*-demethylation, *O*-demethylation and loss of the furosamine sugar moiety (Table 5).

Table 5. Distribution of  $^{14}\text{C}$  residues in the tissues and eggs of laying hens dosed orally for 5 days by capsule at a rate equivalent to 10 ppm spinosyn A or D in the feed (Magnussen and Castetter, 1994).

	Fat		Liver		Muscle		Eggs (day 6)	
	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg
<b>SPINOSYN A</b>								
Spinosyn A	81	1.8	14	0.12	55	0.064	34	0.13
Spinosyn B	2.0	0.044	11	0.10	12	0.014	11	0.041
Spinosyn J	1.5	0.033	0.9	0.008	2.3	0.003	3.3	0.012
Spinosyns H and K	4.1	0.090	9.3	0.082	5.1	0.006	9.7	0.037
Pseudoaglycone			4.6	0.041				
Metab AP-1			7.0	0.062				
Metab AP-2			4.1	0.036				

	Fat		Liver		Muscle		Eggs (day 6)	
	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg
Metab AP-3			2.7	0.024	1.5	0.002	1.4	0.005
Metab AP-4			7.8	0.069	5.7	0.007	4.7	0.018
Metab AP-5			2.4	0.021			1.6	0.006
Metab AP-6			1.9	0.017			1.1	0.004
Aq sol + extr tissue + misc	12	0.26	34	0.30	20	0.024	33	0.12
Total measured <sup>14</sup> C		2.2		0.88		0.12		0.38
SPINOSYN D								
Spinosyn D	79	0.81	3.3	0.058	39	0.048	22	0.069
Spinosyn B of D	6.8	0.069	21	0.37	15	0.018	25	0.080
Spinosyn J of D	2.4	0.025						
Spinosyns H/K of D	6.0	0.061	12	0.21	6.1	0.007	8.0	0.026
Pseudoaglycone D			3.4	0.059	1.6	0.002		
Metab DP-1			5.2	0.091	2.7	0.003		
Metab DP-2			3.9	0.068				
Metab DP-3			6.7	0.12	1.5	0.002	5.4	0.017
Metab DP-4			17.7	0.31	6.0	0.007	12	0.037
Metab DP-5			2.2	0.038	2.0	0.002	2.6	0.008
Metab DP-6			2.4	0.042	1.2	0.001	1.7	0.005
Metab DP-7			2.5	0.044	0.9	0.001	1.5	0.005
Metab DP-8			2.5	0.044	0.8	0.001	1.4	0.004
Aq sol + extr tissue + misc			17	0.30	23	0.029	24	0.069
Total measured <sup>14</sup> C		1.0		1.7		0.12		0.32

mg/kg: <sup>14</sup>C expressed as spinosyn A or D

The metabolic pathways of spinosyn A in hens are shown in Figure 2.

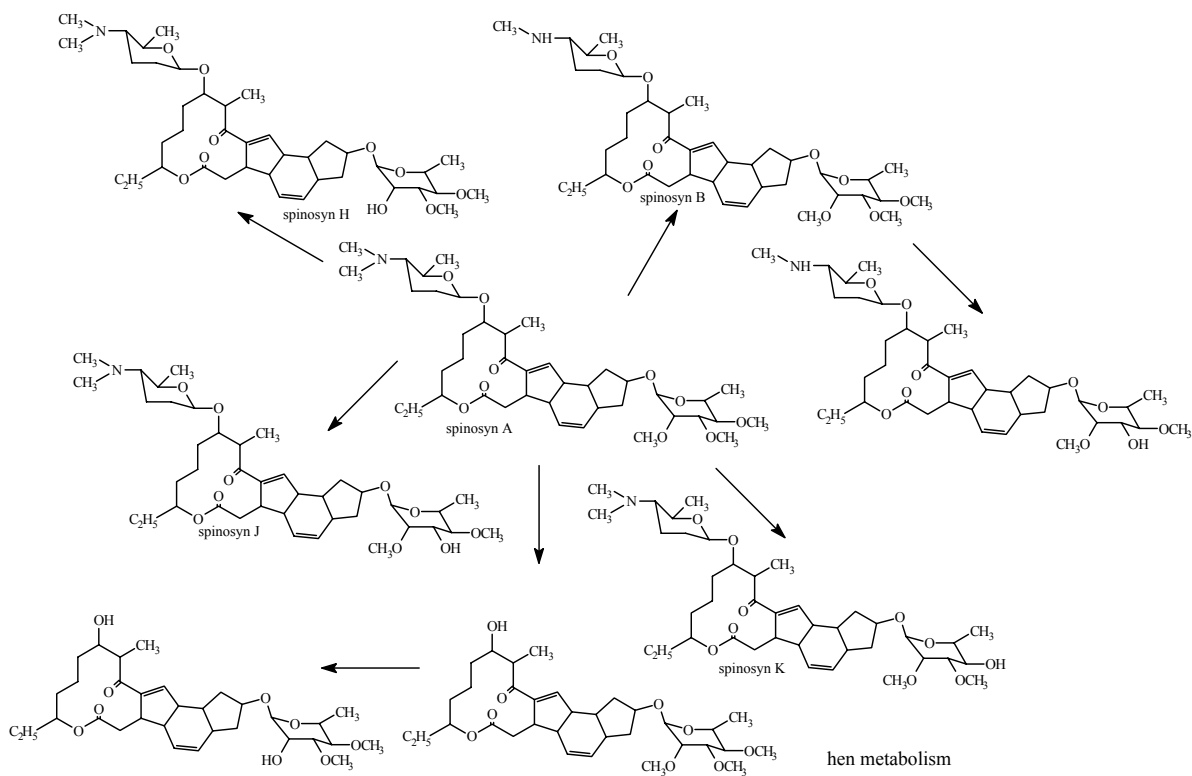


Figure 2. Metabolism of spinosyn A by hens. Spinosyn D metabolism parallels that of spinosyn A.

## Plant metabolism

The Meeting received information on the fate of spinosyns in apples, cabbage, tomatoes, turnips, grapes and cotton after foliar applications.

**Apples.** Dwarf Red Delicious apple trees were sprayed with [ $^{14}\text{C}$ ]spinosyn A at 0.089 kg ai/hl approximately one month before fruit maturity and fruit were sampled 0, 3, 7, 14, 28 and 42 days after treatment (Graper, 1996). Some apples were stored in the dark shortly after treatment to evaluate the influence of photolysis on the residue. Apples on one branch were protected from spray to assess translocation. Parallel experiments were run with [ $^{14}\text{C}$ ]spinosyn D applied at 0.035 kg ai/hl. The results are shown in Table 6.

The total  $^{14}\text{C}$  in the apples decreased by about 50% over the 42 days of sampling, probably owing to growth dilution. Solvent rinses removed much of the residue initially, but after 42 days 60% of the residue could still be rinsed from the surface. Some residue penetrated into the peel and a smaller amount into the pulp. About 10% of the remaining residue after 14 days was accounted for by parent spinosyn A which decreased quickly, and about 10% by spinosyn D after only 3 days. Spinosyns B and B of D were also identified. Spinosyns A and D were much more persistent on those apples kept from the light, demonstrating that photolysis is a dominant degradation process.  $^{14}\text{C}$  in the protected apples on day 42 was only 1.3% of that in apples sprayed directly, showing that translocation was very minor.

The  $^{14}\text{C}$  residues in the apples after day 0 were extensively analysed; they were mostly polar and multicomponent. Fractions from the day 14 spinosyn A apples had low sensitivity to the spinosyn immunoassay, suggesting they did not contain structures close to those for which the immunoassay is sensitive (spinosyns A, B, C, E, F, K and the pseudoaglycone of A).

Table 6. Distribution of  $^{14}\text{C}$  in apples treated with [ $^{14}\text{C}$ ]spinosyn A at 0.089 kg ai/hl or [ $^{14}\text{C}$ ]spinosyn D at 0.035 kg ai/hl approximately one month before maturity (Graper, 1996).

Apple sample	$^{14}\text{C}$ as spinosyn, mg/kg apple											
	spinosyn A						spinosyn D					
	day 0	day 3	day 7	day 14	day 28	day 42	day 0	day 3	day 7	day 14	day 28	day 42
total $^{14}\text{C}$	2.7	3.2	2.3	1.8	1.6	1.3	0.98	1.2	1.2	0.84	0.74	0.51
rinses $^{14}\text{C}$	2.6	2.8	1.9	1.5	0.85	0.80	0.91	0.88	0.89	0.59	0.44	0.30
peel $^{14}\text{C}$	0.12	0.43	0.40	0.32	0.40	0.33	0.031	9.25	0.19	0.22	0.25	0.17
pulp $^{14}\text{C}$	0.024	0.026	0.035	0.056	0.32	0.12	0.042	0.027	0.13	0.033	0.049	0.044
dark, total $^{14}\text{C}$		1.8	1.8					0.84	0.83			
dark, rinses $^{14}\text{C}$		1.7	1.7					0.81	0.75			
dark, peel $^{14}\text{C}$		0.079	0.17					0.027	0.070			
dark, pulp $^{14}\text{C}$		0.008	0.020					0.008	0.011			
protected, total $^{14}\text{C}$	0.002					0.017						
protected, rinses $^{14}\text{C}$	0.002					0.002						
protected, peel $^{14}\text{C}$	0					0.004						
protected, pulp $^{14}\text{C}$	0					0.011						
spinosyn A	2.3	1.1	0.39	0.19	0.080	0.025						
spinosyn B	0.064	0.24	0.14	0.088	0.014	0						
spinosyn D							0.77	0.12	0.062	0	0.019	0
spinosyn B of D							0.035	0.079	0.033	0.015	0.015	0
dark, spinosyn A		1.3	1.2									
dark, spinosyn B		0.11	0.11									
dark, spinosyn D							0.64	0.58				
dark, spinosyn B of D							0.05	0.06				

Berard and Satonin (1995) analysed the leaves from the apple trees treated by Graper (1996). Spinosyn A disappeared much more rapidly when exposed to sunlight, again suggesting that photolysis is the predominant mechanism of degradation (Table 7). Total  $^{14}\text{C}$  levels in leaves protected from direct spraying were much lower than those directly sprayed but continued to increase over 28 days suggesting some translocation. The translocated materials were probably highly polar.  $^{14}\text{C}$  was shown to be incorporated into structural carbohydrates in the treated leaves and the same pattern was found in the untreated leaves.

Table 7. Spinosyn A and D residues in leaves from apple trees treated with [ $^{14}\text{C}$ ]spinosyn A at 0.089 kg ai/hl or [ $^{14}\text{C}$ ]spinosyn D at 0.035 kg ai/hl approximately one month before maturity (Berard and Satonin, 1995).

Leaf sample	$^{14}\text{C}$ expressed as spinosyn, mg/kg leaf									
	spinosyn A					spinosyn D				
	day 0	day 3	day 7	day 10	day 28	day 0	day 3	day 7	day 10	day 28
total $^{14}\text{C}$	217	135	206	175	128	89	97	71	72	43
dark, total $^{14}\text{C}$	217 <sup>1</sup>	154	170			89 <sup>1</sup>	90	126		
protected, total $^{14}\text{C}$	0.023	0.13	0.31	0.56	0.85					
spinosyn A	183	21	20	8.6	0					
dark, spinosyn A	183 <sup>1</sup>	119	132							
spinosyn D						79	2.7	0	0	0
dark, spinosyn D						79 <sup>1</sup>	76	103		

<sup>1</sup> Apple leaves on day 0, same value for exposed and dark.

**Cabbages.** Wakamine variety plants were sprayed with [ $^{14}\text{C}$ ]spinosyn A or D formulated as ECs and diluted to concentrations of 0.14 kg ai/hl (Berard, 1995). Samples of leaf were collected after 2.5 hours and 3, 10, 19 and 34 days. Levels of  $^{14}\text{C}$  in the leaves decreased fairly rapidly, probably because of growth dilution and weathering. The parent compounds also disappeared quickly, probably mainly by photolysis, and accounted for only 10 and 13% of the residue after 3 days (Table 8). It was not possible to identify metabolites.

Table 8. Levels of  $^{14}\text{C}$  in cabbage foliage at intervals after treatment with [ $^{14}\text{C}$ ]spinosyn A or D at 0.14 kg ai/hl (Berard, 1995).

Days after treatment	Spinosyn A			Spinosyn D		
	$^{14}\text{C}$ , mg/kg as spinosyn		$^{14}\text{C}$ in spinosyn, % of total $^{14}\text{C}$	$^{14}\text{C}$ , mg/kg, as spinosyn		$^{14}\text{C}$ in spinosyn, % of total $^{14}\text{C}$
	Leaf rep 1	Leaf rep 2		Leaf rep 1	Leaf rep 2	
0	29	74	41	89	52	48
3	19	17	10	25	17	13
10	3.8	4.3	2.3	6.1	6.5	5.3
19	2.2	1.9	1.1	1.4	2.9	4.3
34	0.78	0.73	0.6	0.89	0.71	4.5

Copenhagen Market variety cabbages treated 4 times with [ $^{14}\text{C}$ ]spinosyn A in an EC formulation at the equivalent of 2 x 0.10 + 2 x 0.15 kg ai/ha were harvested 3 days after the last application and leaves were also sampled 3 days after the first application (Satonin and Collins, 1996). Leaf samples contained 2.4 and 5.6 mg/kg  $^{14}\text{C}$  (as spinosyn A) after the first and last applications respectively. Spinosyn A accounted for 15% and 12% of the  $^{14}\text{C}$  at the two samplings, and spinosyns B and K for 2.3% and 7.7% respectively after harvest. The remainder of the  $^{14}\text{C}$  was present either as numerous minor compounds in the extracts, as shown by TLC and HPLC, or very polar or incorporated into natural compounds. A likely pathway is an initial photodegradation to nonpolar

products such as spinosyns B and K followed by formation of polar products which are available for plant metabolism and incorporation.

**Tomatoes.** Early Girl hybrid tomatoes sprayed 4 times with [ $^{14}\text{C}$ ]spinosyn A as an EC formulation at a rate equivalent to  $2 \times 0.10 + 2 \times 0.15$  kg ai/ha were harvested 3 days after the first and 0 and 3 days after the final application (Satonin and Collins, 1996). The tomatoes contained 0.037, 0.13 and 0.080 mg/kg  $^{14}\text{C}$  (as spinosyn A) on days 0 after spray 1 and 0 and 3 after the last spray respectively. Spinosyn A accounted for 65% and 24% of the  $^{14}\text{C}$  0 and 3 days after the final spray. Numerous minor components of the residue were found by TLC and HPLC, with an increase in polar components at the longer interval after treatment. A portion of the tomatoes containing 0.080 mg/kg  $^{14}\text{C}$  as spinosyn A was processed to juice and a seeds + peel fraction. Residues in the juice (0.048 mg/kg  $^{14}\text{C}$ ) and seeds + peel (0.28 mg/kg  $^{14}\text{C}$ ) suggested that most of the  $^{14}\text{C}$  was on the surface.

**Turnips.** Seven Top variety plants were sprayed with [ $^{14}\text{C}$ ]spinosyn A or D as EC formulations at spray concentrations of 0.098 and 0.051 kg ai/hl respectively (Satonin and Berard, 1995). Samples of leaf and root were collected 0, 10, 24 and 48 days after treatment. Additional 0-day samples were collected immediately after treatment while still wet to minimize exposure of the compounds to sunlight. By day 10 spinosyns A and D were minor components of the  $^{14}\text{C}$  residue in the foliage. Residues of parent compounds that reached the root and were protected from sunlight were more persistent, and by day 24 were higher in the roots, and a higher proportion of the total  $^{14}\text{C}$ , than in the foliage. Spinosyns B, K and B of D, which are products of photolysis, appeared as components of the residue in leaf and root from day 0. Polar metabolites could not be fully characterized. A proportion of the  $^{14}\text{C}$  was shown to be incorporated into the cellulose of the leaves. The results are shown in Table 9.

Table 9. Levels of  $^{14}\text{C}$  in turnip foliage and roots after treatment with [ $^{14}\text{C}$ ]spinosyn A or D (Satonin and Berard, 1995).

Days after treatment	$^{14}\text{C}$ , mg/kg as spinosyn A						$^{14}\text{C}$ , mg/kg as spinosyn D					
	Leaf			Root			Leaf			Root		
	Total $^{14}\text{C}$	A	B/K	Total $^{14}\text{C}$	A	B/K	Total $^{14}\text{C}$	D	B of D	Total $^{14}\text{C}$	D	B of D
0	39	32	2.8	3.5	3.1	0.17	20	14	3.3	1.7	1.3	0.15
10	22	0.45	2.0	1.4	0.29	0.14	13	0.076	0.70	0.43	0.06	0.043
24	5.8	0.075	0.066	0.38	0.084	0.026	4.7	0.016	nd	0.21	0.036	0.010
48	0.33	0.001	0.003	0.18	0.047	0.013	0.30	0.001	nd	0.094	0.018	0.006

**Grapes.** Caley (1996) sprayed immature grapes at nominal concentrations of 500 mg ai/l with  $^{14}\text{C}$ -labelled spinosyns A and D separately when the grapes were about half-grown, and another group with [ $^{14}\text{C}$ ]spinosyn A which were immediately covered with black polythene to protect them from the light. The  $^{14}\text{C}$  label was in the macrolide ring.

Grapes harvested at intervals after treatment up to maturity 49 days later were solvent-washed and extracted, and the washings and extracts were examined for  $^{14}\text{C}$  content (Table 10) and the nature of the residue. A high percentage of the residue was always on the surface, even for aged residues. The percentage of the parent compound fell from 80-90% on day 0 to approximately 50% on day 21 for both spinosyns A and D. In the dark control it was still about 80-90% after 7 days. At mature harvest, spinosyn A accounted for approximately 35% of the residue and spinosyn D 22%.

Attempts to identify other components of the residue, which were polar and numerous, were unsuccessful. The decomposition products were probably products of photolysis.

Table 10. Distribution of  $^{14}\text{C}$  between surface and internal residues from grapes treated with [ $^{14}\text{C}$ ]spinosyns (Caley, 1996).

	$^{14}\text{C}$ expressed as spinosyn A or D, mg/kg							
	Day 0	Day 1	Day 3	Day 7	Day 10	Day 14	Day 21	Maturity 49 days
<b>Spinosyn A</b>								
Washings	6.1	5.5	4.2	3.6	3.2	3.4	2.0	2.2
Washed fruit	0.005	0.14	0.091	0.18	0.20	0.15	0.13	0.20
Total	6.1	5.7	4.3	3.8	3.4	3.5	2.1	2.4
<b>Spinosyn D</b>								
Washings	7.5		5.7	4.3		3.1	2.0	1.5
Washed fruit	0.006		0.35	0.44		0.45	0.36	0.31
Total	7.5		6.0	4.8		3.5	2.4	1.8
<b>Spinosyn A (dark control)</b>								
Washings		2.9	1.6	1.4				
Washed fruit		0.11	0.11	0.089				
Total		3.0	1.7	1.5				

Best *et al.* (1997) further investigated degradation products from Caley's 1996 metabolism study using HPLC and MS. They tentatively proposed hydroxy-spinosyn A and D as consistent with observed spectra.

**Cotton.** One plot of DPL-90 plants was treated 5 times at 6-8 day intervals with [ $^{14}\text{C}$ ]spinosyn A in an EC formulation at a rate equivalent to 0.39 kg ai/ha, and another with [ $^{14}\text{C}$ ]spinosyn D at a rate equivalent to 0.2 kg ai/ha (Magnussen, 1994). Leaves were sampled at various intervals before harvest, and seed and fibre 48 or 49 days after the last treatment before being ginned to separate them.

Levels of  $^{14}\text{C}$  expressed as spinosyn were 0.29 and 0.22 mg/kg for the spinosyn A treatment and 0.11 and 0.075 mg/kg for spinosyn D in seed and fibre respectively. Despite persistent attempts no spinosyn-related compounds were identified in the seed, but in the separated fractions some of the  $^{14}\text{C}$  was incorporated into natural compounds (Table 11). Other residues were multicomponent and highly polar. The  $^{14}\text{C}$  in the fibre was also shown to be incorporated into the cellulose. The leaves contained low levels of spinosyn A until just before harvest but none was translocated to the seed.

Table 11. Fate of spinosyn  $^{14}\text{C}$  in cotton seed fractions harvested from plants treated with [ $^{14}\text{C}$ ]spinosyn (Magnussen, 1994).

Fraction	spinosyn A		spinosyn D		Comments
	% of seed $^{14}\text{C}$	$^{14}\text{C}$ , mg/kg as spinosyn	% of seed $^{14}\text{C}$	$^{14}\text{C}$ , mg/kg as spinosyn	
Oil	32	0.091	37	0.041	incorporated into natural compounds
Water-soluble proteins	4.5	0.013	1.5	0.002	incorporation strongly suggested
Storage proteins	10.6	0.031	8.2	0.009	incorporation strongly suggested
Acid detergent fibre from extracted meal	8.6	0.025			incorporation strongly suggested
Extracted meal			21	0.024	possible incorporation, but spinosyn D fractions not extensively characterized
Various protein and hydrolysate fractions	40	0.12	30	0.033	no identifiable metabolite, but no proof that $^{14}\text{C}$ was incorporated into natural compounds

In another study DPL-90 plants were treated 5 times at 7-day intervals with [ $^{14}\text{C}$ ]spinosyn A as an EC formulation at a rate equivalent to 0.42 kg ai/ha (Magnussen and Castetter, 1995). Seed and fibre were collected from the plots 28 days after the last treatment and ginned to separate seed from fibre.

Levels of residue ( $^{14}\text{C}$  expressed as spinosyn A) were 0.25 and 0.20 mg/kg for meal and lint plus hulls respectively. The level in the meal was quite similar to that in the seed (0.29 mg/kg) in Magnussen's 1994 study (see above) as was the nature of the  $^{14}\text{C}$  residue in the seed which was shown to be incorporated into the fatty acids in the oil fraction, confirming the previous findings.

### Environmental fate in soil

The Meeting received information on the volatilization, photolysis, aerobic degradation, adsorption-desorption, leaching behaviour, field dissipation and crop rotation carry-over of spinosad applied to soil.

**Volatilization.** Knoch (2000) measured the volatilization rate of spinosad from soil and dwarf runner bean foliage in a wind tunnel with air at 20°C flowing at 1-1.5 m/s.  $^{14}\text{C}$ -labelled spinosad (labelled in spinosyn A or spinosyn D) as an SC formulation was applied to bean leaves or soil in petri dishes at a rate equivalent to 0.46 kg spinosyn A/ha or 0.081 kg spinosyn D/ha. Plant and soil samples were taken after 0, 1, 3, 6 and 24 hours for extraction and combustion analysis. Losses of spinosad by volatilization were too small to be observed (maxima of 1.6% from plant surface and 0.1% from soil).

**photolysis.** In a US study Saunders and Powers (1993) applied [ $^{14}\text{C}$ ]spinosyn A and D at a rate equivalent to 1.015 kg ai/ha to a 1 mm layer of a silt loam soil (pH 7.8, sand 22%, silt 55%, clay 23%, organic carbon 0.72%) which was exposed to sunlight (August-September, latitude 39.8°N) at 25°C to observe rates of disappearance and production of photoproducts. Soil samples were periodically examined for  $^{14}\text{C}$  and extracted for HPLC analysis.  $^{14}\text{C}$  balances ranged from 89 to 96% for both irradiated and dark control samples: only 1-2% of the  $^{14}\text{C}$  was volatilized. Little degradation occurred in the dark control samples. Both spinosyns were degraded quickly in the initial stages (initial half-lives of 17 and 7 days for A and D respectively), but subsequent degradation was slow with estimated half-lives exceeding 100 days, suggesting that residues become absorbed into the soil particles before UV exposure can take place. Spinosyn A produced 3 photoproducts accounting for 2-6.6% of the initial  $^{14}\text{C}$ ; the one in highest concentration was identified as spinosyn B. Spinosyn D produced 2 photoproducts accounting for 4-5.4% of the initial  $^{14}\text{C}$ , the smaller of which was identified as N-demethyl spinosad D (B of D).

Saunders *et al.* (1995a) similarly exposed [ $^{14}\text{C}$ ]spinosyn A applied at 1 kg ai/ha to the same silt loam soil and maintained the soil at 75% moisture capacity. Spinosyn A had a half-life of approximately 14 days. The photodegradation rate decreased during the 30 days; perhaps because part of the material was shaded or absorbed by the soil and therefore less available for photolysis. Spinosyn B was the primary photoproduct and the sum of spinosyns A and B disappeared with a half-life of approximately 20 days. In the dark control in 30 days 20% of spinosyn A disappeared and spinosyn B equivalent to 10% of the starting material was generated.

The photoproducts were examined by MS, but full characterization was not possible. Products A1 and A2 were shown to be parent compound with a hydroxyl attached to the macrolide ring, and product A0 was an N-demethyl derivative with a hydroxyl attached to the macrolide ring.

Table 12. Photolysis of [ $^{14}\text{C}$ ]spinosyn A applied at 1 kg ai/ha to a silt loam soil and exposed to sunlight for 30 days at 25°C (Saunders *et al.*, 1995a).

Days	Spinosyn A and photoproducts, % of initial spinosyn A dose				
	Spinosyn A	Spinosyn B	Product A0	Product A1	Product A2
0	93	1.3	0	0	0
2	77	4.9	1.3	1.3	1.7
4	67	6.7	1.9	1.9	1.9
7	57	9.6	2.4	2.1	2.1
11	47	12	2.9	2.6	2.0

Days	Spinosyn A and photoproducts, % of initial spinosyn A dose				
	Spinosyn A	Spinosyn B	Product A0	Product A1	Product A2
18	31	15	5	2.4	2.1
24	23	14	4.3	3.2	2.5
30	21	12	4.4	2.9	2.3

**Aerobic degradation.** Osborne *et al.* (1993) incubated [ $^{14}\text{C}$ ]spinosyn A at 1 mg/kg in a California sandy loam (59% sand, 35% silt, 6% clay, 1.4% organic matter, pH 6.8), a Mississippi sandy silt (23% sand, 65% silt, 12% clay, 1.1% organic matter, pH 7.7), a Georgia sandy loam (79% sand, 14% silt, 12% clay, 0.8% organic matter, pH 6.7) and an Oxfordshire clay loam (47% sand, 24% silt, 29% clay, 5.2% organic matter, pH 7.2) under aerobic conditions at 20°C for 237-359 days. Parallel experiments were run for spinosyn D at 1 mg/kg for 6 months. Mass balances for  $^{14}\text{C}$ , including volatiles, were 78-101% for spinosyn A (mostly exceeding 90%), and 89-101% for spinosyn D. The results are shown in Table 13.

Estimated half-lives for spinosyn A ranged from 40 to 75 days and half-lives for spinosyns A + B were 340, 140, 99 and 96 days for the sandy silt, clay loam and 2 sandy loams respectively. Estimated half-lives for spinosyn D were 65-85 days and for spinosyns D + B of D 650, 150, 250 and 81 days respectively. Mineralisation was variable in the different soils, ranging for spinosyn A from 5.8% in 1 year in the sandy silt to 26% in 6 months in a sandy loam, and for spinosyn D from 4.8%-25% in 8 months. Numerous metabolites were observed on HPLC and TLC examination of soil extracts, but could not be identified.

Table 13. Aerobic soil degradation of spinosyns A and D in four soils (Osborne *et al.*, 1993).

Days	Sandy silt			Clay loam			Sandy loam			Sandy loam		
	Residues, expressed as % of dose											
SPINOSYN A												
	spin A	spin B	volatiles	spin A	spin B	volatiles	spin A	spin B	volatiles	spin A	spin B	volatiles
0	94	1.2		85	3.2		90	1.4		93	0	
14	84	11										
28	68	23										
36	65	24										
56	35	57		21	44		37	41		16	39	
80										10	29	1.0
84	21	63		12	43	0.48	22	46	0.45			
182	5.7	67	2.2						26			
246				5.7	21	8.6	8.1	9.6		5.9	9.0	18
359	4.2	43	5.8									
SPINOSYN D												
	spin D	spin B of D	volatiles	spin D	spin B of D	volatiles	spin D	spin B of D	volatiles	spin D	spin B of D	volatiles
0	71	8.1		69	7.8		69	7.8		71	8.4	
7	71	7.1	1.3	50	12	1.1	50	12	0.65	42	16	1.5
14	62	10	0.56	41	14	0.93	41	14	0.63	35	18	2.4
28	57	18	1.1	27	18	1.9	27	18	1.3	18	15	2.3
56	35	37	2.5	18	27	3.9	18	27	1.6	12	27	4.3
84	31	38	3.8	15	34	1.3	15	34	4.4	11	28	8.7
237	6.6	53	4.8	4.4	17	14	4.4	17	11	3.9	4.0	25

Hale (1994) incubated [ $^{14}\text{C}$ ]spinosyn A in a sandy loam soil (56% sand, 34% silt, 10% clay, 1.0% organic matter, pH 7.5) and a silt loam (37% sand, 50% silt, 13% clay, 1.1% organic matter, pH 7.8) at 0.4 mg/kg under aerobic conditions at 25°C in the dark for 1 year. A parallel experiment was run for spinosyn D at 0.1 mg/kg in the silt loam for 6 months. Mass balances for  $^{14}\text{C}$ , including



volatiles, ranged from 90-106% for spinosyn A, with the exception of days 182 and 365 where they fell to 87 and 79% respectively, and for spinosyn D from 93-103%.

Levels of spinosyns A and D and degradation products are shown in Table 14. The initial process was *N*-demethylation to form spinosyn B or spinosyn B of D. Spinosyn A had initial half-lives of 9 and 16 days in the two soils, spinosyn D approximately 16 days in the silt loam, spinosyns A+B 98 and 92 days in the sandy loam and silt loam respectively, and spinosyns D + B of D 157 days in the silt loam. Spinosyn A was mineralised to the extent of 15% and 21% in the sandy loam and silt loam respectively after one year, but Spinosyn D only 3% after 6 months. Half-lives in sterilized soils were 130-240 days demonstrating that microbial action was mainly responsible for the degradation. The products were not fully characterized, but Metabolite 5 appeared to be a hydroxy-spinosyn A and Metabolite 2 a hydroxy-spinosyn B.

Table 14. Aerobic metabolism of [ $^{14}\text{C}$ ]spinosyns A and D in soil (Hale, 1994).

Incubation, days	Residues, expressed as % of $^{14}\text{C}$ dose							
	Spinosyn A	Spinosyn B	Met 2	Met 3	Met 4	Met 5	Met 6	Others
SANDY LOAM, SPINOSYN A								
0	85	2.2						
1	81	7.6						
3	67	22						
7	41	41	0.68				1.2	1.0
14	23	53	1.9					1.2
28	6.1	61	2.8		0.95		1.2	0.58
56	1.5	47	3.5	0.82	1.9		1.3	6.4
91	1.7	37	3.5	1.4	2.3	2.6	2.1	5.3
140	0.58	21	4.6	3.6	1.2	3.5	1.5	8.4
182	1.6	21	4.7	3.4	1.5	4.8	1.7	2.9
273	0.74	12	4.7	4.3	1.2	2.0	1.3	2.3
364	0.91	6.0	2.2	4.2	0.61	1.5	0.51	11
SILT LOAM, SPINOSYN A								
0	91	2.9						
1	91	4.4						
3	86	9.6						
7	75	18						
14	57	35					1.7	
28	23	51	0.91	0.41			1.2	5.9
56	8.9	56	2.1	0.92	0.61	0.92	2.1	4.4
91	2.7	36	6.4	2.9	2.8		2.0	3.8
140	2.5	34	5.8	2.3	0.51	1.5	3.3	6.9
182	1.9	17	8.1	2.6	1.4	1.2	2.5	2.8
273	1.6	22	6.3	2.8	1.2		2.8	8.4
364	1.6	2.8	3.4	3.2	1.6	0.64	0.54	2.7
SILT LOAM, SPINOSYN D								
	Spinosyn D	Spinosyn B of D						
0	92	3.1						
1	92	5.0						
7	70	26						
10	53	36						
14	45	48						
28	16	68						
56	7.2	60						
91	0	46						
182	0	49						

**Adsorption-desorption.** Saunders and Powers (1994c) measured the adsorption-desorption characteristics of [ $^{14}\text{C}$ ]spinosyn A on a sand, loamy sand, sandy loam, silt loam and clay loam in 0.01 M  $\text{CaCl}_2$  solutions of spinosyn A (initial concentrations 5.0, 1.0, 0.20 and 0.05 mg/l) equilibrated for 22 hours at 25°C in the dark (Table 15).  $K_{oc}$  values were not calculated because adsorption coefficients clearly were not correlated with organic matter content. The measured  $K_d$  values suggest that spinosyn A is unlikely to be leached in to ground water. Cohen *et al.* (1984) interpret  $K_d$  values below 1-5 as signifying a potential for leaching if other requirements, such as environmental persistence, are met.

Saunders and Powers (1994a) used the same procedure to measure the adsorption-desorption characteristics of [ $^{14}\text{C}$ ]spinosyn B on four of the same soils. Spinosyn B is a demethylation product of spinosyn A, ( $-\text{N}(\text{CH}_3)_2 \rightarrow -\text{NHCH}_3$ ). The results of both experiments are shown in Table 15.

Table 15. Soil properties and adsorption-desorption characteristics of spinosyns A and B (Saunders and Powers, 1994a,c).

Soil	Soil properties						Spinosyn A		Spinosyn B	
	sand, %	silt, %	clay, %	organic matter, %	pH	CEC, meq/100 g	Adsorp $K_d$	Desorp $K_d$	Adsorp $K_d$	Desorp $K_d$
Sand	89	5.6	5.2	0.5	7.7	3.5	8.3	8.8	6.2	6.4
Loamy sand	82	10	8	1.1	6.3	1.9	5.4	7.4	4.3	5.8
Sandy loam	56	34	10	1.0	7.5	6.9	25	29	17	20
Silt loam	22	55	23	0.4	7.8	12	320	320	180	180
Clay loam	24	44	32	2.0	6.9	21	280	290		

**Column leaching.** Magnussen and Meitl (1999) applied fresh [ $^{14}\text{C}$ ]spinosyn A at 0.46 mg/kg to a loamy sand (85% sand, 8.0% silt, 7.2% clay, 2.8% organic matter, pH 6.5). Leaching solution (0.01M  $\text{CaCl}_2$ ) was percolated through columns of the treated soil (5 cm i.d.  $\times$  30 cm) at 0.14 ml/min or 393 ml over 48 hours. Application rates were higher (1.4 mg/kg) for tests on microbially aged and photolytically aged residues. Parallel tests were run for spinosyn D. Recoveries of  $^{14}\text{C}$  were 80-100% for fresh, 92-96% for microbially aged and 86-89% for photolytically aged residues.

Fresh residues were not leached at all (Table 16). Some products of microbial metabolism and photolytic decomposition were leached from the column. The ageing processes produced quite polar compounds which probably represented degradation fragments of the spinosyns. The compounds could not be fully identified but were substantially modified from the spinosyns.

Table 16. Distribution of  $^{14}\text{C}$  after leaching tests on [ $^{14}\text{C}$ ]spinosyns A and D on a sandy loam soil (Magnussen and Meitl, 1999). Each result is the mean of results from duplicate columns.

Sample	% of applied $^{14}\text{C}$					
	spinosyn A residues			spinosyn D residues		
	fresh	microbial aged	photolysis aged	fresh	microbial aged	photolysis aged
0-5 cm segment	99.4	92.3	68.8	100	87.7	76.3
5-10 cm segment	0.6	2.2	7.7		6.5	6.8
10-15 segment		0.8	3.7		1.1	2.7
15-20 segment		0.7	3.4		0.6	1.8
20-25 segment		0.4	2.7		0.5	1.8
25-30 segment		0.3	1.7		0.3	1.6
0-24 h leachate		1.0	4.3		0.8	3.7
24-48 h leachate		0.9	7.1		1.0	4.4
48-68 h leachate						
NaOH traps		1.4			1.5	
exposure dish rinse			0.6			0.9

**Field dissipation.** Spinosyn A was applied to the soil surface at a nominal 0.5 kg ai/ha at 2 US sites, one in California (a sandy loam) and the other in Mississippi (a silty clay), after which soil samples down to 90 cm were taken at intervals up to 10 months later (Saunders *et al.*, 1995b). Spinosyn A disappeared very quickly (Table 17). Three products were formed at low levels and they too decreased within 2 months to undetectable levels. Very little of the residue penetrated below the top 15 cm layer of soil. The mineralization half-life was approximately 7 months at both sites.

Table 17. Field dissipation of spinosyn A (Saunders *et al.*, 1995b).

<sup>14</sup> C, as g/ha (nd = not detected at 0.003 mg/kg, equiv to 7 g/ha)									
Silty clay					Sandy loam				
Days	Spin A	Metab A0	Metab A1	Metab A2	Days	Spin A	Metab A0	Metab A1	Metab A2
0	501	nd	8	14	0	375	nd	nd	8
1	142	34	63	111	1	34	57	73	74
3	16	31	47	60	3	nd	38	51	59
4	11	50	49	52	5	7	48	55	53
8	7	31	30	25	8	nd	23	30	22
14	nd	19	19	16	14	nd	20	28	21
24	nd	15	20	10	23	nd	11	15	8
38	nd	10	18	nd	40	nd	nd	8	nd
64	nd	nd	nd	nd	72	nd	nd	nd	nd
93	nd	nd	nd	nd	98	nd	nd	nd	nd
126	nd	nd	nd	nd	205	nd	nd	nd	nd
196	nd	nd	nd	nd	247	nd	nd	nd	nd
247	nd	nd	nd	nd	286	nd	nd	nd	nd
308	nd	nd	nd	nd	316	nd	nd	nd	nd

**Residues in rotational crops.** Rainey (1994b) treated sandy loam soil plots with [<sup>14</sup>C]spinosyn A at a rate equivalent to 1.1 kg ai/ha and 30, 120 and 365 days after treatment sowed wheat, lettuce and radishes. Lettuce, radish root, radish foliage, wheat grain and wheat straw were harvested at maturity and analysed for <sup>14</sup>C content (Table 18). Immature wheat plants were also collected for analysis. The <sup>14</sup>C residues were characterized by HPLC and examination of natural products such as starch, lignin and protein. No spinosyns or closely related compounds were identified in the crops. At least some of the <sup>14</sup>C had been incorporated into natural compounds.

Table 18. Levels of <sup>14</sup>C in rotational crops sown in soil previously treated with [<sup>14</sup>C]spinosyn A at 1.1 kg ai/ha (Rainey, 1994b).

Sowing interval, days after treatment	Total <sup>14</sup> C as spinosyn A, mg/kg					
	Lettuce	Radish root	Radish foliage	Immature wheat plant	Wheat grain	Wheat straw
30	0.009	0.016	0.008	0.022	0.023	0.079
120	0.020	0.014	0.030	0.048	0.29	0.50
365	0.006	0.004	0.004	0.010	0.009	0.027

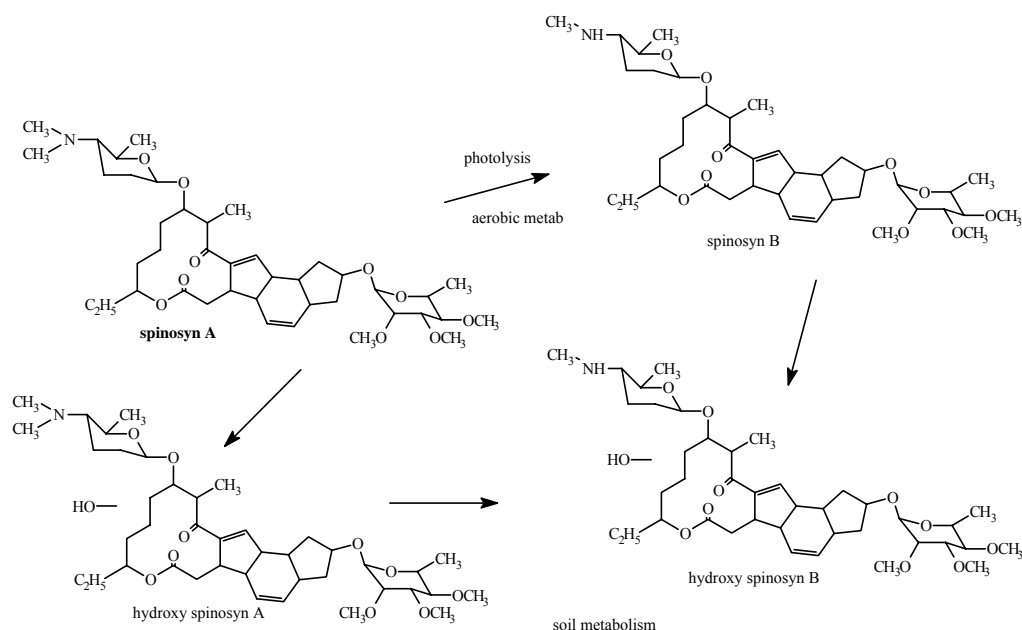


Figure 3. Degradation of spinosyn A in soil. Spinosyn D degradation parallels that of spinosyn A.

### Environmental fate in water/sediment systems

The Meeting received information on photolysis in solution, anaerobic degradation and dissipation in an aquatic environment.

**Photolysis.** In a study in the USA, Saunders and Powers (1994b) exposed [ $^{14}\text{C}$ ]spinosyns A and D dissolved in sterile pH 7 buffers at 2 mg/l in borosilicate glass tubes to natural sunlight at 25°C for 48 hours at 39.9°N latitude from June 26-28 and July 15-18, and samples were taken at various intervals for  $^{14}\text{C}$  measurement and analysis by HPLC. Recoveries of  $^{14}\text{C}$  ranged from 88.5-103%, and the half-lives of spinosyn A and D were 22.3 and 19.7 hours of sunlight respectively (Table 19).

Photoproduct A1 was identified by MS and NMR, after additional photolysis runs provided sufficient material, as the pseudoaglycone of spinosyn A with an additional 2 hydrogens saturating the double bond in the cyclopentane ring. Photoproduct A2 gave a molecular ion at  $m/z = 731$ , the same as spinosyn A, i.e. a rearrangement of spinosyn A, but the nature of the rearrangement was not determined. Photoproduct A3 gave a molecular ion at  $m/z = 749$ , equivalent to the addition of a water molecule to the parent. Photoproduct D1 had a molecular ion at  $m/z=606$  consistent with the pseudoaglycone of spinosyn D plus 2 hydrogens; it is probably the analogue of A1 with the extra methyl group. Photoproducts D2 and D3 did not produce useful mass spectra. Photoproduct D4 gave a molecular ion at  $m/z = 763$ , equivalent to spinosyn D plus water.

Table 19. Photolysis of [ $^{14}\text{C}$ ]spinosyns A and D dissolved in sterile pH 7 buffers at 2 mg/l in borosilicate glass tubes and exposed to natural sunlight for 48 hours (Saunders and Powers, 1994b).

	<sup>14</sup> C, % of initial concentration				
SPINOSYN A					
Irradiation time, h	spinosyn A	product A1	product A2	product A3	
0	98				
6.0	83	3.3	4.8	3.7	
10.8	68	7.5	7.0	4.7	
14.2	59	14	4.1	7.5	

	<sup>14</sup> C, % of initial concentration				
24.2	45	20	5.1	8.9	
31.7	37	24	6.0	8.3	
SPINOSYN D					
Irradiation time, h	spinosyn D	product D1	product D2	product D3	product D4
0	95				
5.5	76	5.8	4.1	2.7	3.3
11.0	61	12	5.3	4.1	3.4
19.0	47	18	7.1	5.7	3.7
28.3	33	16	3.5	6.0	3.0
39.8	24	19	3.1	5.2	3.4

Spinosyns A and D were subjected to photolysis in natural pond water at 25°C under sunlight for 2 days at 39.9°N latitude (USA) on 19-21 August to determine degradation rates (Yoder and Sanders, 1996). Spinosyn D has very low water solubility; the addition of 0.05% acetonitrile as a co-solvent raised the solubility to approximately 2.5 mg/l. Residual spinosyns in the solutions were measured periodically by an HPLC method. The half-lives for both were 4.3 hours (of sunlight). Concentrations in dark controls were substantially stable. Photodegradation products were identified as spinosyn B (from spinosyn A) and N-demethyl-spinosyn D or spinosyn B of D (from spinosyn D): their concentrations reached maximum levels about half way through the experiments and had begun to decrease by the end.

**Anaerobic degradation.** Reeves (1993) incubated pond water (100 ml) and sediment (50 g equiv dry weight) with [<sup>14</sup>C]spinosyn A -at 0.85 µg/ml water in the dark at 25°C under anaerobic conditions for 1 year. The water had an alkalinity of 390 mg/l, expressed as CaCO<sub>3</sub> and the sediment contained 2.8% organic matter. The procedures were repeated for [<sup>14</sup>C]spinosad D. Samples were taken at various intervals and sediment and water were separated by decanting and centrifuging. The sediment was then extracted with acetone-methanol and then acetone. <sup>14</sup>C balances for spinosyns A and D were 94-104% and 92-105% respectively. The distribution of <sup>14</sup>C in the spinosyns and degradation products in the water and sediment is shown in Table 20. The residue rapidly became attached to the sediment. Both compounds had initial half-lives of about 6 months, but were quite persistent in the second 6 months. Very small amounts (<2%) of volatile <sup>14</sup>C were produced. Major degradation products were identified as the N-demethyl compounds spinosyn B and spinosyn B of D. The others were not fully characterized.

Table 20. Anaerobic degradation of spinosyns A and D in pond water and sediment (Reeves, 1993). The distribution of the spinosyns and products are expressed as % of dose. Degradation products are identified by their retention times relative to the parent compound. Minor products (<5% of dose) are not included.

Days	<sup>14</sup> C expressed as % of dose							
	Total <sup>14</sup> C		Spinosyn A		Degradation products			
	Water	Sediment	water	sediment	Rt 0.86	Rt 0.82	Rt 0.72	Rt 0.64
0	58	41	52	38		9.3		
3	14	89	13	85		6.0		
7	8.5	87	7	81	3.0	4.3		
14	4.3	91	4	79	6.5	5.8		
28	3.4	93	3	79	9.1	2.3	3.8	
56	2.6	96	2	71	12	3.7	7.5	3.8
84	2.1	92	1	56	14	2.5	11	3.8
170	2.3	92	1	49	13	7.3	12	6.2
365	2.1	92	1	38	9.2	2.1	18	18
Days	Total <sup>14</sup> C		Spinosyn D		Degradation products			
	Water	Sediment	water	sediment	Rt 0.84-7	Rt 0.82-4	Rt 0.75-6	
0	50	52	43	45	4.6	5.6		

3	14	91	11	84	3.4	2.3	0.1	
7	6.0	89	4.7	80	3.4	3.6	1.3	
14	4.3	89	3.6	76	3.8	3.2	2.8	
28	3.1	91	2.8	77	6.9	2.3	3.3	
56	2.5	92	1.6	74	10	3.2	3.9	
84	2.2	91	2.2	71	11	2.7	4.6	
170	2.0	90	0.9	56	12	8.0	6.4	
365	1.9	92	0.7	59	14	2.4	9.1	

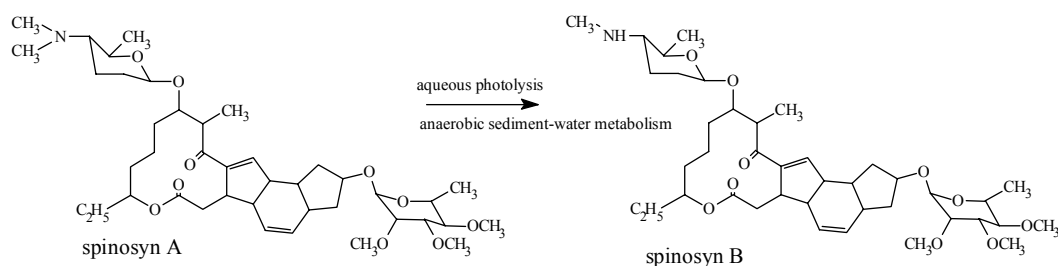
The fate of spinosad was investigated in an aquatic microcosm study using 3 open tanks 60 cm deep and 170 cm diameter holding 1100 l of pond water and a 6 cm layer of pond sediment exposed to sunlight and weather conditions (McGibbon *et al.*, 1995). Spinosad in a diluted SC formulation was applied to the surface at a nominal 0.10 kg ai/ha and samples were analysed by immunoassay and HPLC (Table 21). Spinosyn residues decreased rapidly in the water, with a half-life of about 1-2 days, and spinosyn A in the sediment generally accounted for only about 10-15% of that applied. The results suggest that spinosad dissipates principally by degradation (photolysis) then by adsorption to the sediment.

Table 21. Spinosyn residues in a water/sediment system treated with spinosad at 0.10 kg ai/ha (McGibbon *et al.*, 1995).

Hours	Spinosyn residues in water, mg/l (mean of 3 tanks)						Spinosyn residues in sediment, mg/kg (mean of 3 tanks)					
	A	D	B	B of D	Total		A	D	B	B of D	Total	
					HPLC	IA <sup>1</sup>					HPLC	IA <sup>1</sup>
0	0.028	0.0047	0.0011	0.0012	0.035	0.045						
1	0.018	0.0032	0.0014	0.0009	0.024	0.034						
2	0.015	0.0020	0.0011	0.0005	0.019	0.035						
4	0.014	0.0021	0.0017	0.0005	0.018	0.031						
8	0.011	0.0016	0.0022	0.0006	0.015	0.026						
24	0.0086	0.0008	0.0015	0.0004	0.011	0.020	0.01	<0.01	<0.01	<0.014	0.01	0.024
48	0.0049	0.0005	0.0015	<0.0001	0.0068	0.014	0.014	<0.01	<0.01	<0.014	0.014	0.029
96	0.0019	0.0008	0.0014	0.0005	0.0046	0.012	0.015	<0.01	<0.01	<0.014	0.019	0.031
192	0.0003	0.0001	0.0005	0.0005	0.0014	0.006	0.013	<0.01	<0.01	<0.014	0.018	0.041
360	<0.0001	0.0001	0.0002	<0.0001	0.0003	0.002	0.011	<0.01	<0.01	<0.014	0.019	0.039
840							0.014	<0.01	<0.01	<0.014	0.019	0.031

<sup>1</sup> IA: immunoassay

Figure 4. Water-sediment degradation of spinosyn A. Spinosyn D degradation parallels that of spinosyn A.



## METHODS OF RESIDUE ANALYSIS

### Analytical methods

Analytical methods for the determination of residues of the spinosyns fall into two main types after an extraction designed for the sample: HPLC and immunoassay. HPLC methods follow a reasonably standard clean-up with HPLC determination relying on UV or MS detection to measure the individual spinosyns, and in residue trials provide data on spinosyns A, D, K, B and B of D. Spinosyn A contributes most of the residue most of the time and some HPLC methods have concentrated on spinosyns A and D since national authorities have decided that spinosyns A and D should constitute the definition of the residue. Immunoassay methods may or may not require a clean-up before the final colorimetric determination. The method is specific and reports the sum of the spinosyns. The HPLC and immunoassay methods have been extensively validated with numerous recoveries on a wide range of substrates and when used side-by-side in trials agreement was usually good.

In an HPLC method for measuring residues in potatoes and tomatoes described by Balderrama Pinto and Matos (1996a) the residue is extracted from ground samples with an acetonitrile-water mixture which, after acidification with HCl, is washed with hexane. The aqueous phase is then made alkaline with NaOH and the residue extracted into hexane. The hexane extract is cleaned up on a solid-phase column and the eluate evaporated to dryness and taken up in acetonitrile-ammonium acetate solution for HPLC analysis with UV detection at 250 nm. Spinosyns A and D are measured separately as two peaks. Recoveries from potato samples were mean 99 and 101%, range 89-112%,  $n = 12$  for spiking at 0.01 and 0.1 mg/kg, and from tomato samples mean 92 and 91%, range 73-106%,  $n = 12$  at the same levels. The LOQ was 0.01 mg/kg for A and D.

In an HPLC method for measuring spinosad residues in almond nuts and hulls described by Duebelbeis *et al.* (1997) the residue is extracted with an acetonitrile-water mixture which, after filtration, is partitioned into 1-chlorobutane. The extract is cleaned up on silica and cyclohexyl solid-phase columns and analysed by reverse-phase HPLC with UV detection at 250 nm. Spinosyns A, D, B, K and N-demethyl-D are measured as 5 separate peaks. Identities are confirmed by reanalysis of the extracts by LC-MS. Recoveries from kernel samples were mean 85%, range 76-116%,  $n = 20$ , and from hulls mean 82%, range 72-94%,  $n=20$  for spiking at 0.01, 0.02, 0.05, 0.25 and 1.0 mg/kg. The LOQ was 0.01 mg/kg for each spinosyn.

Turner *et al.* (1996a,b, GRM 95.15, GRM 95.15.R1) used a similar HPLC method for eggs and poultry tissues. Extracting solvents were tailored to the substrates as follows: acetonitrile-methanol 1:1 for eggs, acetonitrile-water 4:1 for meat and liver, and hexane-dichloromethane 3:2 for fat. Turner and West (1999) described modifications needed to improve recoveries from spoiled poultry fat.

Atkin and Dixon-White (1995) examined the GLC behaviour of spinosyns A and D to determine whether spinosad could be analysed by an FDA multi-residue method. It was concluded that spinosyns A and D could not be chromatographed after tests on a DB-1-phase capillary column at various temperatures and with FID and MS detection; spinosad was not amenable to analysis in the multiresidue methods. Satonin (1996) reached the same conclusion for spinosyns B, K and B of D.

In an LC-MS method for spinosyns A, D, K, B and N-demethyl-spinosyn D in crop samples with a high water content described by Hastings *et al.* (2000a) residues are extracted with an acetonitrile-water mixture and an aliquot of the extract is cleaned up on a cation-exchange solid-phase cartridge and eluted with a methanol-water-acetonitrile mixture containing ammonium acetate. HPLC with detection and quantification by APCI MS (scanning for 718.6, 732.6 and 746.6 amu). The LOQ was 0.01 mg/kg. Recoveries were consistent from apricots, banana peel and pulp, whole banana, green beans, broccoli, Brussels sprouts, cabbages, cauliflowers, celery, courgettes, cucumbers, leeks, lettuce, melon peel and pulp, whole melon, nectarines, onions, peach flesh, peas, potatoes, watermelon peel and pulp and whole watermelon (Table 22).

Table 22. Analytical recoveries of spinosyns from crop samples with high water content by an LC-MS method (Hastings *et al.*, 2000a).

	Spinosyn				
	A	D	B	B of D	K
Fortification at 0.01 mg/kg each analyte					
mean, %	96	94	94	93	98
range, %	71-110	81-106	79-108	72-107	84-108
n	50	50	50	50	50
Fortification at 0.1 mg/kg each analyte					
mean, %	95	94	95	93	96
range, %	82-109	81-107	79-106	79-104	80-106
n	50	50	50	50	50
Fortification at 1 mg/kg each analyte					
mean, %	95	95	94	94	96
range, %	83-107	82-108	73-109	70-106	85-109
n	50	50	50	50	50

Hastings and Clements (2000, GRM 00.04) applied essentially the same LC-MS method to a range of crop substrates with a low water content (Table 24): chick peas and forage, lucerne forage and hay, mung beans and forage, oat grain, sorghum grain and forage, wheat grain and forage and chick pea, oat, sorghum and wheat straw. The LOQ for grain and forage was 0.01 mg/kg and for straw 0.02 mg/kg.

Hastings *et al.* (2000b) applied a very similar method to pome fruit and grapes and their processed commodities, kiwifruit and avocados. Wine was extracted with dichloromethane after the addition of aqueous sodium chloride and the extract evaporated to leave a residue for clean-up in the same way as for other samples. Analytical recoveries are shown in Table 23 for apple juice, apple pomace, apples, avocados, grape juice and pomace, grapes, kiwifruit, pear juice and pomace, pears and wine. The LOQ was 0.01 mg/kg.

Van Acker and Hastings (2000, GRM 00.01) extracted brandy with methyl *tert*-butyl ether after the addition of sodium bicarbonate. The extract was evaporated to dryness and taken up in acetonitrile-methanol for LC-MS-MS analysis. The LOQ was 0.01 mg/l.

West (1995a) extracted residues from water samples with dichloromethane. Clean-up, if required, was by silica solid-phase extraction. The spinosyns were determined by HPLC with UV detection. LOQs were 0.001 mg/l.

Boothroyd *et al.* (1999) extracted spinosad residues from water with methyl *tert*-butyl ether after the addition of a sodium hydroxide solution. The extract was evaporated and reconstituted in water-methanol for analysis by LC-MS. The method was tested on drinking, surface and ground waters. LOQs were 0.0001 mg/l.

The HPLC method was also applied to soil and sediment over the concentration range 0.01-1.0 mg/kg by West (1995b). Residues were extracted with alkaline methanol containing sodium chloride. The method was later improved by adding clean-up steps to reduce interferences from some soils and the introduction of glycerol as a keeper solvent to reduce loss of analytes during concentration (West and Turner, 1999).



Table 23. Analytical recoveries of spinosyns from pome fruit, grapes, processed commodities, kiwifruit and avocados by an LC-MS method (Hastings *et al.*, 2000b).

	Spinosyn				
	A	D	B	B of D	K
Fortification at 0.01 mg/kg each analyte					
mean, %	100	102	101	98	103
range, %	81-113	79-115	86-115	75-108	78-117
n	26	26	24	26	26
Fortification at 0.1 mg/kg each analyte					
mean, %	102	102	100	100	102
range, %	83-123	92-117	76-122	85-116	91-115
n	26	26	26	26	26
Fortification at 1 mg/kg each analyte					
mean, %	96	94	93	93	97
range, %	75-113	74-116	72-111	71-109	79-116
n	25	25	26	24	26

Table 24. Summary of extensive testing of HPLC methods with UV or MS detection.

Commodity	Spike concn., mg/kg	n	Spinosyns recovery %										Ref.
			A		D		K		B		B of D		
			mean	range	mean	range	mean	range	mean	range	mean	range	
alfalfa forage	0.01-0.1	3	94		94		73		74		77		GRM95.17.S1
alfalfa hay	0.01-5.0	14	92	79-104	88	74-109	96	84-115	87	58-105	88	57-103	GRM 97.06
apple	0.01-5.0	28	91	78-102	91	82-102	90	74-101	91	74-102	91	75-108	ERC 97.18
apple	0.01-1.0	20	93	86-101	92	85-95	92	88-95	90	84-95	88	82-93	GRM 95.05
apple juice	0.01-5.0	28	93	80-114	91	82-109	93	81-104	89	78-97	89	71-102	ERC 97.18
apple juice	0.01-1.0	20	93	91-98	93	89-90	93	89-98	88	82-95	88	81-94	GRM 95.05
apple pomace	0.01-5.0	28	98	73-110	96	70-113	98	74-110	95	74-108	97	74-112	ERC 97.18
apple purée	0.01-5.0	28	97	80-111	97	79-109	98	84-110	96	81-109	95	81-109	ERC 97.18
apple wet pomace	0.02-2.0	20	89	86-95	88	81-93	90	88-93	88	81-92	86	79-97	GRM 95.05
beef fat	0.01-10.0	11	98	81-108	95	84-107			94	85-102	82	56-93	GRM 95.03
beef kidney	0.01-1.0	11	84	76-97	84	76-99			114	98-136	97	86-107	GRM 95.03
beef liver	0.01-1.0	11	106	97-120	92	83-110			107	88-125	94	71-109	GRM 95.03
brandy	0.01-0.5	34	105	83-116	105	81-118	103	84-112	96	81-108	97	84-112	GRM 00.01
broccoli	0.01-2.0	23	94	90-98	92	85-98	91	86-96	74	68-80	74	64-84	GRM 94.22
cabbage	0.01-2.0	23	84	76-91	79	64-90	80	69-90	75	70-79	74	68-79	GRM 94.22
celery	0.01-5.0	20	93	89-95	93	87-97	84	75-92	86	81-90	85	80-88	GRM 95.17.R1
cereal grain, forage	0.01-0.1	26	91	76-100	88	70-104	81	68-91	79	66-90	76	62-86	GRM95.17.S1
chicken fat	0.02-2.0	11	114	102-124	112	104-121			102	84-114	99	86-105	GRM 95.15.R1
chicken liver	0.01-1.0	11	94	85-113	84	77-97			93	83-100	93	88-99	GRM 95.15.R1
chicken meat	0.01-1.0	11	92	84-101	88	92-94			100	97-109	95	84-103	GRM 95.15.R1
chicken meat/skin/fat	0.01-1.0	11	86	76-95	77	71-83			96	88-105	92	76-99	GRM 95.15.R1
chilli peppers	0.01-1.0	20	96	92-102	94	89-99	96	90-100	88	84-92	87	83-90	GRM 95.04
citrus fruit, whole	0.01-2.0	20	104	97-111	101	94-110	99	84-109	98	89-113	95	85-111	GRM 96.09.R1
corn stover	0.01-5.0	7	93		93		88		77		82		GRM 97.06
cotton seed	0.01-0.10	22	93	68-107	89	55-113							GRM 93.02.R2
cotton seed	0.01-0.10	18	99	74-119	95	82-117							GRM 94.02
cotton seed hulls	0.01-0.10	10	100	89-116	100	87-110							GRM 94.02
cotton seed meal	0.01-0.10	10	90	81-100	85	73-96							GRM 94.02

Commodity	Spike concn., mg/kg	n	Spinosyns recovery %										Ref.
			A		D		K		B		B of D		
			mean	range	mean	range	mean	range	mean	range	mean	range	
cotton seed oil, crude	0.01-0.10	18	96	86-113	93	85-99							GRM 94.02
cotton seed oil, ref	0.01-0.10	10	92	70-98	86	68-103							GRM 94.02
cotton seed soapstock	0.01-0.10	18	99	93-104	102	98-110							GRM 94.02
cream	0.01-10.0	11	103	96-114	99	87-115			107	96-116	106	97-113	GRM 95.03
cucurbits	0.01-0.1	12	99	88-117	96	82-116	98	84-115	85	66-113	86	72-111	GRM95.17.S1
eggs	0.01-1.0	20	88	73-100	87	69-97			102	94-110	97	87-109	GRM 95.15.R1
grain and forage	0.01	22	100	89-119	99	87-114	93	80-111	93	79-103	95	77-117	GRM 00.04
grain and forage	0.1	22	90	74-108	90	72-105	86	73-101	89	76-102	87	72-100	GRM 00.04
grain and forage	1.0	22	89	76-102	89	73-101	87	77-94	91	81-99	89	73-99	GRM 00.04
grape	0.01-3	11	90	75-100	93	73-105							BRC 99.1
grape must	0.01-5.0	44	97	80-117	98	78-118	96	78-109	87	76-109	86	71-115	ERC 97.18
grape pomace	0.01-5.0	28	87	75-101	87	74-102	85	72-100	94	77-119	90	76-111	ERC 97.18
grapes	0.01-5.0	28	90	79-111	89	79-112	88	81-111	90	78-108	88	71-112	ERC 97.18
green peppers	0.01-1.0	20	95	92-99	93	89-98	94	90-98	85	78-92	84	78-90	GRM 95.04
head lettuce	0.01-5.0	20	88	84-96	88	82-95	85	80-93	78	72-86	77	71-86	GRM 95.17.R1
leaf lettuce	0.01-5.0	20	93	86-98	91	85-95	88	83-90	83	74-90	81	71-89	GRM 95.17.R1
lean beef tissue	0.01-1.0	11	95	81-107	87	82-93			101	91-107	98	92-107	GRM 95.03
lemon	0.01-0.1	11	94	90-102	94	88-101							GRM00.6
lemon oil	0.01-0.05	11	83	68-114	90	71-120							GRM00.6
maize	0.01-1	18	80	54-125	89	58-137							GRM00.6
milk, whole	0.01-1.0	20	104	99-116	101	90-114			101	94-109	101	92-112	GRM 95.03
mustard greens	0.01-2.0	23	92	88-102	90	83-95	87	82-91	82	78-89	81	75-86	GRM 94.22
nectarine	0.01-1	11	96	81-110	100	69-113							BRC 99.1
orange	0.01-1.0	23	85	64-108	80	69-96							GRM00.6
orange juice	0.01-20.0	20	102	92-110	94	83-101	104	95-112	94	80-102	90	75-98	GRM 96.09.R1
orange juice	0.01-1	12	96	71-113	82	74-89							GRM00.6
orange oil	0.02-10.0	20	88	76-98	87	74-101	88	79-98	77	65-86	77	70-85	GRM 96.09.R1
orange peel	0.01-10.0	20	97	75-110	95	79-107	99	87-108	90	79-108	90	79-107	GRM 96.09.R1
orange pulp, dried	0.02-10.0	20	113	106-23	110	105-19	110	105-18	99	87-116	97	85-112	GRM 96.09.R1
orange, edible portion	0.01-2.0	20	102	92-108	99	88-106	98	86-106	92	79-106	91	78-105	GRM 96.09.R1
peas, beans, soya beans	0.01-0.1	9	99	98-109	95	84-108	95	80-109	85	70-94	83	68-92	GRM95.17.S1
peppers	0.01-5.0	28	92	83-114	95	82-129	94	81-119	89	77-110	88	73-104	ERC 97.18
potato	0.01-0.1	3	93		86		88		74		92		GRM95.17.S1
soil, sediment	0.01-1.0	35	82	71-89	83	71-95			78	64-87	76	61-85	GRM 94.20
soil	0.01-1.0	15	88	81-91	83	74-90			85	66-98	77	63-90	GRM 94.20.R1
sorghum fodder	0.01-5.0	7	88		91		82		72		72		GRM 97.06
spinach	0.01-5.0	20	91	87-102	91	88-98	87	78-96	84	78-94	84	75-94	GRM 95.17.R1
stone fruits	0.01-0.1	12	100	87-122	97	72-126	85	64-105	84	78-95	81	64-94	GRM95.17.S1
straw	0.02	8	97	90-106	94	84-108	97	91-108	90	82-105	91	81-104	GRM 00.04
straw	0.1	8	92	86-97	90	83-95	90	85-97	86	81-91	88	82-93	GRM 00.04
straw	1.0	8	88	77-93	88	79-92	90	84-95	88	81-94	87	79-91	GRM 00.04
strawberries	0.01-5.0	28	95	74-110	96	86-109	94	83-108	95	82-110	96	86-108	ERC 97.18
tangerine	0.01-1	13	91	82-106	99	87-108							GRM00.6
tomato dry pomace	0.04-4.0	20	95	87-98	84	84-98	95	92-98	90	87-93	89	86-93	GRM 95.04

Commodity	Spike concn., mg/kg	n	Spinosyns recovery %										Ref.
			A		D		K		B		B of D		
			mean	range	mean	range	mean	range	mean	range	mean	range	
tomato juice	0.01-1.0	7	96	92-98	95	90-97	95	92-97	89	88-91	88	86-90	GRM 95.04
tomato paste	0.01-1.0	7	90	87-93	90	84-100	87	80-92	87	86-90	87	85-90	GRM 95.04
tomato purée	0.01-1.0	7	95	93-97	94	89-95	94	90-96	91	90-91	89	86-90	GRM 95.04
tomato wet pomace	0.02-2.0	20	92	89-97	92	88-96	91	88-96	78	75-84	76	74-79	GRM 95.04
tomatoes	0.01-5.0	28	101	81-120	101	81-127	99	90-113	95	85-110	97	74-110	ERC 97.18
tomatoes	0.01-1.0	20	96	88-104	93	87-98	91	85-96	85	78-90	84	78-90	GRM 95.04
tomatoes juice	0.01-5.0	28	98	91-109	97	89-104	88	68-107	96	87-105	96	86-108	ERC 97.18
tomatoes purée	0.01-5.0	28	98	85-118	97	85-113	90	76-111	94	71-114	97	80-118	ERC 97.18
tomatoes, tinned	0.01-5.0	26	97	88-116	98	91-114	94	85-112	96	89-110	96	91-111	ERC 97.18
water (pond well tap)	0.001-0.10	35	93	77-106	90	73-104			87	71-96	90	71-111	GRM 94.12
water (pond) <sup>1</sup>	0.001-0.10	11	81	71-105	81	71-87			82	73-117	78	72-97	GRM 94.12
water, drinking	0.1-5 ug/l	32	107	99-115	105	87-118	108	100-115	109	91-122	107	87-121	ERC 98.23
water, ground	0.1-5 ug/l	32	95	73-101	96	64-109	98	88-108	100	80-108	99	73-108	ERC 98.23
water, surface	0.1-5 ug/l	32	98	80-111	99	87-117	100	91-114	102	89-118	101	88-119	ERC 98.23
wheat hay	0.01-5.0	7	90		86		87		70		78		GRM 97.06
wheat straw	0.01-5.0	7	93		93		87		72		69		GRM 97.06
wine	0.01-5.0	28	98	87-112	97	74-109	99	79-112	99	87-119	98	86-115	ERC 97.18

<sup>1</sup> Method includes silica SPE clean-up step

Pinheiro *et al.* (2000a, GRM 00.6) used an HPLC method for determining residues of spinosyns A and D in citrus fruits, juice and oil and maize in supervised and processing trials in Brazil and Argentina. The method is essentially the same as that of Balderrama Pinto and Matos (1996a) with the initial extraction step modified to suit the substrate. The method was extensively tested for recoveries. The LOQ was 0.01 mg/kg. The method was also tested on nectarines and grapes (Pinheiro *et al.*, 1999a).

Tidswell and Cowles (1998a) extracted milled cotton seed with a hexane-acetone mixture. The extract was evaporated and the residue taken up in acidic aqueous methanol which was washed with hexane. The spinosyns were partitioned into hexane after the addition of sodium hydroxide and further cleaned up on a cyclohexyl solid-phase extraction column. Spinosyns A and D were then determined by HPLC with UV detection. Recoveries from spiked cotton seed (0.01 and 0.1 mg/kg, n = 17) were spinosyn A mean 78%, range 66-93%; spinosyn D mean 69%, range 60-76%. Tidswell and Cowles (1998b) further tested the method on cotton bracts and trash with recoveries (0.01-5.9 mg/kg, n = 17) of spinosyn A mean 83%, range 68-107%, and for spinosyn D mean 82%, range 67-111%.

Khoshab and Marshall (1998) described an immunoassay analytical method for grapes, pomace, must and wine that relies on the Strategic Diagnostics "Spinosad Rapid Assay" test kit. The antibody is sensitive to several spinosyns and measures the total residue of spinosad and its metabolites. Residues are extracted from the substrate with an acetonitrile-water mixture, except wine which is extracted with dichloromethane, and a portion of the extract cleaned up by cyclohexyl solid-phase extraction.

A portion of the diluted sample is incubated with enzyme-conjugated spinosad and magnetic particles coated with antibodies specific to spinosad. Spinosad in the sample and enzyme-conjugated spinosad compete for antibody sites on the magnetic particles. When a magnetic field is applied to the particles at the end of the incubation period, the spinosad and enzyme-conjugated spinosad bound to

the antibodies on the particles are held in the sample tube by the magnetic field while the unbound reagents are decanted. A coloured product, produced by incubating the antibody-bound enzyme conjugate with hydrogen peroxide and 3,3',5,5'-tetramethylbenzidine, is measured by its absorbance at 450 nm.

The assay is sensitive to spinosyn analogues with little or no modification of the trimethylpyranosyl ring, but relatively insensitive to analogues or degradation products if the ring has been modified or is missing. The method was tested for interference by 30 pesticides, 16 inorganic compounds and 8 organic compounds (most naturally occurring). Carbendazim was the only compound generating a response, but at a low sensitivity. Recoveries were satisfactory over the spiking range 0.01-5 mg/kg spinosyn A (Table 25). The LOQ was 0.01 mg/kg.

The  $I_{50}$  concentration is the concentration that results in a 50% inhibition of conjugate binding to the available antibodies and is a measure of the sensitivity of the method. The  $I_{50}$  for spinosyn A is approximately 0.0003 µg/ml (Redstone, 1998). Compounds with high sensitivity ( $I_{50} < 0.002$  µg/ml) are spinosyns A, B, C, D, N-demethyl-D, E, F, K and A pseudoaglycone. Compounds with a low sensitivity ( $I_{50} > 0.05$  µg/ml) are spinosyns H, J, L, A reverse pseudoaglycone and A aglycone. The  $I_{50}$  for carbendazim was 5 µg/ml.

The immunoassay method was applied to sediment over the concentration range 0.05-0.35 mg/kg (Young and Mihaliak, 1995). Spinosad was extracted from the sediment with alkaline methanol containing sodium chloride. Recoveries are shown in Table 25.

The immunoassay method was also shown to be suitable for spinosad residues in bovine tissues and milk (Young and Mihaliak, 1996). The residues are extracted from tissues with an acetonitrile-water mixture, and from milk with acetonitrile. The method was validated in the range 0.01-0.5 mg/kg (Table 25).

Table 25. Analytical recoveries of spiked spinosyn A from various substrates by an immunoassay method.

Sample	Spike concn., mg/kg	n	Mean recov., %	Range, %	Ref.
apples	0.01-1.0	18	102	92-119	GRM 95.20
bovine kidney	0.01-0.50	13	78	68-84	GRM 95.14
bovine liver	0.01-0.50	14	77	64-86	GRM 95.14
bovine milk	0.01-0.50	32	84	67-100	GRM 95.14
bovine muscle	0.01-0.50	22	77	68-86	GRM 95.14
broccoli	0.01-1.0	17	105	81-125	GRM 95.20
broccoli	0.01-3.0	26	85	71-107	PA-RM-97-07
Brussels sprouts	0.01-3.0	21	100	75-120	PA-RM-97-04
cabbage	0.01-1.0	18	110	83-128	GRM 95.20
cabbage	0.01-3.0	33	98	81-120	PA-RM-97-04
capsicum	0.01-5.0	16	101	86-112	PA-RM-97-04
cauliflower	0.01-2.0	15	90	76-109	PA-RM-97-07
celery	0.01-1.0	19	105	91-123	GRM 96.10
cherries	0.01-0.20	12	98	70-123	GRM 96.11.S1
Chinese cabbage	0.01-3.0	16	103	78-117	PA-RM-97-04
cucumber	0.01-0.10	10	93	70-110	GRM 96.10.S1
grape pomace	0.01-5.0	28	101	80-120	ERC 97.09
grapefruit, whole	0.01-1.0	17	96	77-118	GRM 96.11
grapes	0.01-5.0	28	104	86-118	ERC 97.09
head lettuce	0.01-1.0	17	94	81-104	GRM 96.10
kiwifruit	0.01-6.0	17	105	87-131	PA-RM-97-01
leaf lettuce	0.01-1.0	19	102	92-113	GRM 96.10
lemon, whole	0.01-1.0	17	101	87-124	GRM 96.11
lucerne pasture	0.01-10	21	87	70-111	PA-RM-97-07
maize	0.01-0.10	7	103	88-126	GRM 96.10.S1

Sample	Spike concn., mg/kg	n	Mean recov., %	Range, %	Ref.
muskmelon	0.01-0.25	10	94	73-125	GRM 96.10.S1
must	0.01-5.0	28	103	88-120	ERC 97.09
mustard greens	0.01-1.0	17	101	85-112	GRM 95.20
navy bean hay	0.01-1.0	10	110	92-119	PA-RM-98-01
navy bean whole plant	0.01-1.0	4	102	87-114	PA-RM-98-01
navy bean, dry bean	0.01-1.0	6	93	74-110	PA-RM-98-01
orange peel	0.01-1.0	19	100	74-127	GRM 96.11
orange, edible portion	0.01-1.0	17	97	79-116	GRM 96.11
orange, whole	0.01-1.0	17	98	75-122	GRM 96.11
peach	0.01-0.20	6	87	72-101	GRM 96.11.S1
pear	0.01-0.10	9	96	70-115	GRM 96.11.S1
peppers	0.01-5.0	28	92	75-114	ERC 97.17
peppers, green	0.01-1.0	17	102	82-115	GRM 95.20
plum	0.01-0.10	9	80	60-102	GRM 96.11.S1
potatoes	0.01-1.0	16	103	76-121	GRM 96.10.S1
prunes, dried plums	0.01-0.10	6	95	67-111	GRM 96.11.S1
sediment	0.05-0.35	39	77	66-98	GRM 94.21
snap beans	0.01-0.50	12	109	89-126	GRM 96.10.S1
snow peas	0.01-0.50	7	94	72-111	GRM 96.10.S1
sorghum fodder	0.01-1.0	17	98	67-117	GRM 97.05
sorghum forage	0.01-2.5	15	92	73-112	GRM 97.05.S1
sorghum grain and grain dust	0.01-2.5	29	102	75-123	GRM 97.05.S1
soya beans	0.01-0.50	10	112	91-137	GRM 96.10.S1
spinach	0.01-1.0	17	104	84-129	GRM 96.10
squash	0.01-0.10	6	89	70-112	GRM 96.10.S1
sweet corn forage	0.01-1.0	13	100	71-116	GRM 96.10.S1
sweet corn grain	0.01-1.0	16	98	64-126	GRM 96.10.S1
sweet corn stover	0.01-1.0	13	94	60-124	GRM 96.10.S1
tomato haulm	0.01-30	28	98	78-123	PA-RM-97-06
tomato juice	0.01-5.0	28	97	74-114	ERC 97.17
tomato purée	0.01-5.0	28	108	80-129	ERC 97.17
tomatoes	0.01-5.0	28	98	79-117	ERC 97.17
tomatoes	0.01-1.0	18	106	73-124	GRM 95.20
tomatoes	0.01-1.0	12	98	71-120	PA-RM-97-07
tomatoes, canned	0.01-5.0	28	99	81-117	ERC 97.17
water	0.0001-0.02	31	101	71-117	GRM 94.10
wheat	0.01-0.20	15	102	84-121	GRM 96.10.S1
wheat forage, hay and straw	0.01-1.0	29	93	66-119	GRM 96.10.S1
wine	0.01-5.0 mg/l	28	98	81-119	ERC 97.09

### Stability of pesticide residues in stored analytical samples

The Meeting received information on the freezer storage stability of spinosyn residues in a range of commodities representing those in the supervised trials and feeding studies.

Fleeker *et al.* (1998) spiked blueberry homogenate with spinosad at 0.09 mg/kg and stored the samples at -20°C for 141 days. In the 4 separate tests 90%, 90%, 97% and 69% of the spiked spinosad remained.

Portions of untreated grape, pepper and strawberry samples in small polypropylene vessels were fortified with spinosyns A, D, K, B and N-demethyl-D at 0.1 mg/kg and stored at or below -18°C. Portions of wine in small glass vials were similarly fortified and stored (Khoshab, 2000a). Samples were analysed after various intervals up to approximately 18 months. On each occasion the method was tested with procedural recoveries but the results are unadjusted (Table 26). Residues were

generally stable. The calculated time for a 30% decrease in spinosyn D residues in wine was 12 months.

The rate of decrease and time for 30% loss of residue were calculated for each stability test by assuming a first-order rate (rate proportional to concentration). Where the calculated time for 30% decrease exceeded the duration of the test, the time is recorded as greater than the duration of the test, e.g. >18 months.

Table 26. Freezer storage stability of spinosyns in spiked grapes, peppers, strawberries and wine (Khoshab, 2000a).

Days	Spinosyn A		Spinosyn D		Spinosyn K		Spinosyn B		Spinosyn B of D	
	Conc, mg/kg <sup>1</sup>	Proc recov %	Conc, mg/kg	Proc recov %	Conc, mg/kg	Proc recov %	Conc, mg/kg	Proc recov %	Conc, mg/kg	Proc recov %
<b>GRAPES</b>										
0	0.087	93 92	0.084	92 81	0.086	92 82	0.104	97 92	0.103	95 89
98	0.096	88 87	0.090	91 85	0.098	86 88	0.099	83 84	0.097	82 82
202	0.095	87 88	0.087	87 85	0.092	85 85	0.093	88 86	0.092	85 85
571	0.085	86 82	0.060	85 83	0.085	85 81	0.082	92 91	0.070	95 94
Months for 30% decrease	>19		17		>19		>19		17	
<b>PEPPERS</b>										
0	0.094	94 93	0.094	94 94	0.095	94 94	0.096	99 97	0.095	96 96
101 <sup>2</sup>	0.12	93 159	0.12	89 156	0.12	98 156	0.12	103 162	0.12	97 159
205	0.099	90 93	0.094	89 94	0.094	87 89	0.092	86 89	0.092	85 87
574	0.092	88 91	0.069	85 88	0.095	94 93	0.085	87 86	0.082	86 85
Months for 30% decrease	>19		>19		>19		>19		>19	
<b>STRAWBERRIES</b>										
0	0.096	91 88	0.097	91 90	0.096	91 84	0.098	92 89	0.097	92 87
100	0.11	94 98	0.10	90 98	0.11	100 88	0.10	95 99	0.10	90 96
204	0.10	90 95	0.097	89 95	0.099	90 94	0.10	91 95	0.10	88 94
573	0.096	93 92	0.070	91 89	0.096	93 94	0.087	91 89	0.084	91 90
Months for 30% decrease	>19		>19		>19		>19		>19	
<b>WINE</b>										
0	0.11	107 103	0.11	107 107	0.11	106 104	0.11	111 110	0.11	109 108
99	0.14	115 107	0.14	114 106	0.15	117 110	0.15	119 107	0.15	115 108
203 <sup>3</sup>	0.12	106 115	0.11	101 115	0.11	100 114	0.028	27 37	0.029	27 38
572	0.097	98 98	0.072	94 92	0.10	100 99	0.10	104 103	0.10	105 103
Months for 30% decrease	>19		12		>19		>19		>19	

<sup>1</sup> The concentration at time 0 is the mean of 6 analyses and at later times the mean of 2.

<sup>2</sup> Results at 101 days for peppers are not included in the calculation because of an unacceptable procedural recovery.

<sup>3</sup> Results at 203 days for spinosyns B and B of D in wine are not included in the calculation because of unacceptable procedural recoveries.

Robb and Bormett (1996) tested the frozen storage stability of the spinosyns in spiked apples and juice stored at approximately -20°C. Spinosyns A and D were spiked together, and B, K and N-demethyl-D together. High-density polyethylene containers were used for apple samples and low density for the juice. Residues were stable for the periods tested. After 193 days apples were stored for one day at room temperature to simulate temperature conditions possible during shipping; no degradation of residues was observed. Robb and Rutherford (1996) tested the frozen storage stability of the residues in tomatoes using similar methods, and Phillips and Harris (1997) followed a similar procedure for almond kernels and hulls. Residues were stable for the periods tested. Phillips *et al.* (1996) tested stabilities in cabbages in the same way. Problems were experienced with low procedural recoveries on some occasions. The results of all the trials are shown in Table 27.

Table 27. Freezer storage stability of spinosyns in spiked apples, apple juice, tomatoes, almond kernels, almond hulls, cabbage and milk.

Days	Spinosyn A		Spinosyn D		Spinosyn K		Spinosyn B		Spinosyn B of D	
	Conc, mg/kg	Proc recov %	Conc, mg/kg	Proc recov %	Conc, mg/kg	Proc recov %	Conc, mg/kg	Proc recov %	Conc, mg/kg	Proc recov %
APPLES <sup>1</sup> Robb and Bormett, 1996										
0	0.091	90 93	0.089	89 90	0.098	86 88	0.080	85 78	0.080	82 78
28	0.085	90 87	0.084	89 86	0.096	90 87	0.076	87 87	0.073	84 84
90	0.087	87 88	0.086	92 93	0.096	92 85	0.079	80 84	0.075	78 82
194	0.091	90 92	0.087	86 88	0.097	92 93	0.086	77 82	0.083	78 83
Months for 30% decrease	>6		>6		>6		>6		>6	
APPLE JUICE <sup>1</sup> Robb and Bormett, 1996										
0	0.082	85 83	0.081	84 81	0.081	86 82	0.068	72 66	0.072	75 70
41	0.086	89 87	0.083	87 84	0.085	86 88	0.081	82 84	0.079	80 83
71	0.081	93 92	0.079	91 80	0.079	88 87	0.074	77 69	0.072	76 69
92	0.092	85 90	0.088	82 88	0.087	89 89	0.081	79 78	0.079	80 78
Months for 30% decrease	>3		>3		>3		>3		>3	
TOMATOES (HDPE containers, -20°C) <sup>2</sup> Robb and Rutherford, 1996										
0	0.092	93 91	0.093	95 91	0.087	84 91	0.081	87 82	0.081	85 82
34	0.092	92 95	0.091	91 94	0.076	82 82	0.081	79 84	0.080	80 85
99	0.088	90 90	0.087	90 88	0.082	80 79	0.066	73 70	0.068	73 73
183	0.092	108 109	0.088	106 106	0.092	92 94	0.075	78 75	0.074	77 74
336	0.098	105 105	0.091	104 104	0.094	101 100	0.087	81 86	0.081	80 83
Months for 30% decrease	>12		>12		>12		>12		>12	
ALMOND KERNELS (HDPE containers, -20°C) <sup>2</sup> Phillips and Harris, 1997										
0	0.088	87 94	0.084	83 90	0.088	86 87	0.083	83 78	0.082	82 77
31	0.084	95 95	0.080	92 91	0.089	97 93	0.084	91 84	0.083	89 82
64	0.089	97 97	0.084	93 94	0.087	98 98	0.079	90 79	0.079	86 85
93	0.094	97 98	0.088	104 104	0.090	100 109	0.084	95 98	0.082	91 94
178	0.089	101 104	0.086	109 113	0.091	104 102	0.083	92 91	0.082	89 88
Months for 30% decrease	>6		>6		>6		>6		>6	
ALMOND HULLS (HDPE containers, -20°C) <sup>2</sup> Phillips and Harris, 1997										
0 <sup>3</sup>	0.077	82 82	0.070	75 76	0.080	79 79	0.071	66 69	0.069	64 65
36 (16)	0.087	92 91	0.082	88 87	0.080	83 82	0.068	73 71	0.065	70 67
55 (35)	0.085	86 92	0.080	83 88	0.083	92 89	0.069	82 87	0.066	78 77
86 (66)	0.089	92 99	0.084	98 104	0.082	91 94	0.073	78 76	0.069	77 70
169 (149)	0.084	93 104	0.078	101 113	0.081	90 93	0.069	77 73	0.065	73 69
Months for 30% decrease	>6		>6		>5		>5		>5	
CABBAGE (HDPE containers, -20°C) <sup>2</sup> Phillips et al., 1996, RES94098. <sup>4</sup>										
0	0.081	89	0.082	89	(0.071)	65 67	(0.068)	68 59	(0.067)	68 59
35	0.081	95 88	0.082	94 88	0.090	94 90	0.076	80 73	0.074	79 72
93	0.067	76 66	(0.065)	74 64	0.067	70 82	0.064	68 72	(0.061)	66 70
133	0.090	98 97	0.086	92 94	0.084	95	0.075	82 85	0.073	81 81
186	0.091	94 91	0.086	87 86	0.087	88 82	0.082	78 79	0.078	76 74
367	0.071	69 78	0.070	64 76	0.070	79 91	(0.067)	65 60	(0.062)	58 56
Months for 30% decrease	>12		>12		>12		>6		>6	
MILK (HDPE containers, -20°C) <sup>2</sup> Rutherford and Robb, 1996b, RES95126.										
0	0.097	104 104	0.093	101 100			0.103	102 103	0.100	99 101
41	0.089	100 97	0.086	97 93			0.093	93 90	0.089	92 89
136	0.080	97 101	0.076	94 96			0.082	90 97	0.076	90 95
Months for 30% decrease	>5		>5				>5		>5	

<sup>1</sup> The concentration at each time is the mean of 2 analyses<sup>2</sup> The concentration at each time is the mean of 3 analyses

<sup>3</sup> Almond hulls: sampling times for spinosyns B, K and B of D are shown in parentheses

<sup>4</sup> Values in parentheses were not included in the estimate of storage stability because of poor procedural recoveries

Gardner and West (1994a) tested the storage stability of spinosyns A and D in spiked ground cotton seed at 0.1 mg/kg in polyethylene freezer cartons at -15°C to -20°C at intervals of 14-54 days, mainly about 50 days, in 17 separate tests without loss, and also showed that spinosyns A and D in cotton seed samples stored frozen in high-density polyethylene containers and in laminated cotton cloth bags were stable for the 9 months of the test (Table 28). Gardner *et al.* (1999) showed that the residues were still stable after 1 year.

Table 28. Freezer storage stability of spinosyns A and D in spiked cotton seed (Gardner and West, 1994a; Gardner *et al.*, 1999).

Days	Spinosyn A			Spinosyn D		
	HDPE <sup>1</sup>	PE-lam <sup>2</sup>		HDPE <sup>1</sup>	PE-lam <sup>2</sup>	
	Conc, mg/kg <sup>3</sup>	Conc, mg/kg <sup>3</sup>	Procedural recov %	Conc, mg/kg <sup>3</sup>	Conc, mg/kg <sup>3</sup>	Procedural recov %
0	0.075	0.073	77 85 80 76 74	0.072	0.064	78 80 71 72 72
36	0.075	0.079	82 79 90 90	0.066	0.073	70 67 81 83
98	0.074	0.079	91 89 94 86	0.065	0.077	81 81 86 78
212	0.094	0.081	97 97 88 89	0.085	0.075	88 92 82 84
283	0.096	0.092	102 98 96 96	0.098	0.095	105 99 97 97
366	0.092	0.091	98 98 93 97	0.088	0.088	91 94 88 94
Months for 30% decrease	>12	>12		>12	>12	

<sup>1</sup> HDPE (high-density polyethylene) freezer containers

<sup>2</sup> PE-laminated cotton cloth bags

<sup>3</sup> The concentration at each time is the mean of 2 analyses.

Phillips and Blakeslee (1997) fortified celery and spinach samples with a mixture of spinosyns A, D, B, K and B of D at a total spinosyn concentration of 0.5 mg/kg in HDPE bottles and stored them up to 202 days in a freezer at -20°C. Residues, measured by an immunoassay method, were stable (Table 29). Phillips *et al.* (1998a) fortified potato, and Robb *et al.* (1998) fortified maize grain and sweet corn forage and stover samples with 0.1 mg/kg spinosad (spinosyns A + D, 85 + 15) in HDPE containers for freezer storage at -20°C for 342 days. The residues, measured by an immunoassay method, were shown to be stable (Table 29).

Table 29. Freezer storage stability of spinosyns in potatoes, celery, spinach, maize grain, sweet corn forage and stover.

Days	Total spinosyns, mg/kg <sup>1</sup>	Proc recov %
POTATO, Phillips <i>et al.</i> , 1998a		
0	0.108	112 119
28	0.082	105 117
31	0.075	96 98
55	0.075	100 101
97	0.075	104 102
191	0.076	109 123
342	0.072	98 112
Months for 30% decrease	>12	
CELERY, Phillips and Blakeslee, 1997		
0	0.33	63 69
1	0.34	68 66
4	0.32	63 59
7	0.32	62 72
14	0.35	75 74



Days	Total spinosyns, mg/kg <sup>1</sup>	Proc recov %
22	0.32	70 71
32	0.35	74 74
55	0.32	79
90	0.34	78 80
146	0.30	67 68
202	0.30	74 75
Months for 30% decrease	>7	
SPINACH, Phillips and Blakeslee, 1997		
0	0.34	70 71
1	0.33	66 67
4	0.33	68 66
7	0.33	69 70
14	0.33	69 64
22	0.34	67 68
32	0.37	81 76
55	0.32	72
90	0.36	80 74
146	0.31	72 74
202	0.32	78 73
Months for 30% decrease	>7	
MAIZE GRAIN, Robb <i>et al.</i> , 1998		
0	0.103	108 106
28	0.099	102 100
97	0.090	107 99
191	0.094	101 110
342	0.091	99 95
Months for 30% decrease	>12	
SWEET CORN FORAGE, Robb <i>et al.</i> , 1998		
0	0.099	97 99
28	0.080	77 88
97	0.085	84 88
191	0.103	112 114
342	0.099	105 97
Months for 30% decrease	>12	
SWEET CORN STOVER, Robb <i>et al.</i> , 1998		
0	0.095	99 98
28	0.084 <sup>2</sup>	93 58
31	0.098	106 104
97	0.075	78 84
191	0.102	109 111
342	0.093	97 92
Months for 30% decrease	>12	

<sup>1</sup> The concentration at each time is the mean of 3 analyses, uncorrected for recoveries.

<sup>2</sup> This value disregarded because of problem with procedural recovery.

Spurlock-Brouwer *et al.* (2000) analysed 10 milk samples with incurred spinosad residues at approximately 0.1 mg/kg before and after freezer storage at intervals of 44 to 102 days and found an average loss of approximately 0.25% of the residue per day. They also tested 6 cream samples with incurred residues and found that spinosyn A residues were stable in freezer storage for the periods tested, 66 to 122 days.

Spurlock-Brouwer (1999) analysed samples of liver, kidney, muscle, subcutaneous and renal fat with incurred residues, then re-analysed them after storage at approximately -20°C for at least 6 months. The study was conducted with incurred residues because available fortification methods would denature the tissues. The residues were stable for the periods tested.

Table 30. Storage stability of incurred spinosad residues in animal tissues stored at -20°C (Spurlock-Brouwer, 1999). Each recorded value is the mean of 2 sample extractions.

Sample	Storage, months	Residue, mg/kg							
		spinosyn A		spinosyn D		spinosyn B		spinosyn B of D	
		initial	final	initial	final	initial	final	initial	final
Liver	7.5	0.455	0.442	0.066	0.068	0.099	0.099	0.030	0.030
Kidney	6.5	0.267	0.262	0.035	0.037	0.091	0.094	0.016	0.015
Muscle	6	0.082	0.095	0.013	0.015	<0.01	<0.01	<0.01	<0.01
SC fat	9	0.371	0.395	0.051	0.051	0.018	0.017	0.014	0.013
Renal fat	9	0.514	0.570	0.076	0.081	0.027	0.031	0.020	0.020

### Definition of the residue

Spinosad is a mixture of spinosyns A and D. After it is used on crops the closely related compounds spinosyn B, spinosyn K and spinosyn B of D are formed, principally by photolysis. HPLC methods measure all these compounds separately. An immunoassay analytical method measures these spinosyns and some other metabolites also. Spinosyn A constitutes approximately 85% of the residue initially and in practice constitutes the main part of the spinosyn residue; in 482 of 624 cases (77%) in the residue trials spinosyn A constituted 70% or more of the measured residue. Spinosyn A is well correlated with the total residue (as measured by HPLC): total spinosyn residue =  $1.206 \times \text{spinosyn A}$ ,  $r^2 = 0.997$ ,  $n = 624$ , range = 0.01 to 5 mg/kg. Spinosyn D levels are typically only 10-20% of the spinosyn A levels but spinosyn A and spinosyn D together generally constitute more than 90% of the total spinosyn residue.

Spinosyns A and D were the main identifiable components of the residue in fat, muscle, kidney, liver and milk of goats dosed orally or treated dermally with spinosyns A and D.

In some trials the residue was measured by the immunoassay method; the residue so measured may be considered sufficiently close to the sum of spinosyn A and spinosyn D for the purpose of estimating maximum residue levels or dietary intakes.

The log  $P_{ow}$  values of 4 and 4.5 (pH 7) and the animal metabolism studies suggest that spinosyns A and D should be described as fat-soluble in body fat, but spinosad is incompletely partitioned into milk fat. In trials of the direct treatment of dairy cows described later, the residues in cream were 4.2 times those in milk (mean of 119 observations), and were 3-5 times those in the milk in the feeding study.

The Meeting was aware that national governments had already adopted the sum of spinosyn A and spinosyn D as a definition of the spinosad residue.

The Meeting recommended that the definition of the residue for compliance with MRLs and for the estimation of dietary intake should be the sum of spinosyn A and spinosyn D.

The Meeting recommended that spinosad should be described as fat-soluble for the determination of residues in meat, but not for residues in milk.

### USE PATTERN

Information on registered uses made available to the Meeting is shown in Table 3131 and 32.

Table 31. Registered uses of spinosad on crops.

Crop	Country	Form	Application				PHI, days
			Method <sup>1</sup>	Rate, kg ai/ha	Spray conc. kg ai/hl	No.	
Alfalfa	Chile <sup>12</sup>	SC 480	foliar	0.060-0.072			7
Almonds	USA <sup>12</sup>	SC 240	foliar	0.070-0.18	0.0019-0.0047	<sup>4</sup>	14
Apple	Chile <sup>12</sup>	SC 480	foliar	0.060-0.072			14
Apple	Israel <sup>12</sup>	SC 480	foliar		0.0096		21
Apple	Japan <sup>12</sup>	SC 200	broadcast	0.60	0.010	3	3
Apple	USA <sup>12</sup>	SC 240	foliar	0.070-0.18	0.0025-0.0062	<sup>4</sup>	7
Avocado	Israel <sup>12</sup>	SC 480	foliar		0.0096		1
Avocado	Kenya <sup>12</sup>	SC 480	foliar		0.0096		21
Avocado	NZ <sup>12</sup>	SC 120	foliar		0.0048	4	3
Barley	USA <sup>12</sup>	SC 240	foliar	0.035-0.11		<sup>9</sup>	21 <sup>10</sup>
Barley	USA <sup>12</sup>	SC 480	foliar	0.050-0.11		<sup>9</sup>	21 <sup>10</sup>
Beans	Colombia <sup>12</sup>	SC 120	foliar	0.036-0.060			1
Beans	Kenya <sup>12</sup>	SC 480	foliar	0.12	0.012-0.024		1
Beans	Lebanon <sup>12</sup>	SC 480			0.036-0.048		1
Beans	Peru <sup>12</sup>	SC 120	foliar	0.012-0.024	0.003-0.006		1
Beans, dried	USA <sup>12</sup>	SC 240	foliar	0.053-0.11		<sup>6</sup>	28 <sup>5</sup>
Beans, succulent	USA <sup>12</sup>	SC 240	foliar	0.053-0.11		<sup>4</sup>	3 <sup>5</sup>
Brassica alboglabra	Malaysia	SC 25	broadcast	0.050	0.011	2	3
Brassica chinensis	Malaysia	SC 25	broadcast	0.025	0.055	2	3
Brassica oleracea	Malaysia	SC 25	broadcast	0.025	0.0055	2	3
Brassica rapa	Malaysia	SC 25	broadcast	0.025	0.055	2	3
Brassica vegetables	NZ <sup>12</sup>	SC 120	foliar	0.048			3
Brassicas	China	SC 25 ?	broadcast	0.025-0.13		2	1
Brassicas	Cyprus	SC 480	foliar	0.060-0.14	0.012-0.014		1
Broccoli	Australia <sup>12</sup>	SC 120	foliar (	0.048-0.096			3
Broccoli	Costa Rica <sup>12</sup>	SC 120	foliar	0.012-0.024		3	1
Broccoli	Guatemala <sup>12</sup>	SC 120	foliar	0.012-0.024		3	1
Broccoli	Honduras <sup>12</sup>	SC 120	foliar	0.012-0.024		3	1
Broccoli	Mexico	SC 480	foliar	0.012-0.024	0.0013-0.008	3	1
Broccoli	Paraguay <sup>12</sup>	SC 480	foliar	0.012-0.024			1
Brussels sprouts	Australia <sup>12</sup>	SC 120	foliar (	0.048-0.096			3
Buckwheat	USA <sup>12</sup>	SC 240	foliar	0.035-0.11		<sup>9</sup>	21 <sup>10</sup>
Buckwheat	USA <sup>12</sup>	SC 480	foliar	0.050-0.11		<sup>9</sup>	21 <sup>10</sup>
Cabbage	Australia <sup>12</sup>	SC 120	foliar (	0.048-0.096			3
Cabbage	Costa Rica <sup>12</sup>	SC 120	foliar	0.012-0.024		3	1
Cabbage	Guatemala <sup>12</sup>	SC 120	foliar	0.012-0.024		3	1
Cabbage	Honduras <sup>12</sup>	SC 120	foliar	0.012-0.024		3	1
Cabbage	Indonesia <sup>12</sup>	SC 25	broadcast	0.015-0.025	0.0014-0.0023		
Cabbage	Israel <sup>12</sup>	SC 480	foliar	0.072			7
Cabbage	Japan <sup>12</sup>	WG 250	broadcast	0.30	0.010	3	3
Cabbage	Kenya <sup>12</sup>	SC 480	foliar	0.12	0.012-0.024		3
Cabbage	Korea	SC 100	broadcast	0.038	0.0025	5	3
Cabbage	Peru	SC 240	foliar		0.006-0.0072	3	1

Crop	Country	Form	Application				PHI, days
			Method <sup>1</sup>	Rate, kg ai/ha	Spray conc. kg ai/hl	No.	
Cabbage	Philippines	SC 25	broadcast	0.025	0.004	4	7
Cabbage	Uruguay <sup>12</sup>	SC 240	foliar		0.006-0.0072		1
Cabbage	Vietnam	SC 25	broadcast	0.020-0.025	0.006-0.0078	2	1
Cauliflower	Australia <sup>12</sup>	SC 120	foliar (	0.048-0.096			3
Cauliflower	Peru <sup>12</sup>	SC 120	foliar	0.012	0.003		1
Chard	Peru	SC 240	foliar		0.006-0.0072	3	1
Chard	Uruguay <sup>12</sup>	SC 240	foliar		0.006-0.0072		1
Chinese cabbage	Australia <sup>12</sup>	SC 120	foliar (	0.048-0.096			3
Chinese cabbage	Japan <sup>12</sup>	WG 250	broadcast	0.30	0.010	3	3
Chinese cabbage	Korea	WG 100	broadcast	0.038	0.0025	5	7
Chinese kale	Thailand	SC 120	broadcast	0.12-0.24	0.012-0.024	2	3
Citrus	Cyprus	SC 480	foliar	0.29-0.70	0.0096-0.014		
Citrus	Kenya <sup>12</sup>	SC 480	foliar		0.0096		21
Citrus	Korea	WG 100	broadcast		0.005	5	14
Citrus	Lebanon <sup>12</sup>	SC 480			0.006		7
Citrus	Peru <sup>12</sup>	SC 120	foliar		0.003-0.006		1
Citrus	UAE	SC 480	foliar		0.0072		7
Citrus	USA <sup>12</sup>	SC 240	foliar	0.070-0.18		<sup>4</sup>	1
Cole crops	USA <sup>12</sup>	SC 240	foliar	0.026-0.18		<sup>4</sup>	1
Corm vegetables	USA <sup>12</sup>	SC 240	foliar	0.053-0.11		<sup>11</sup>	7
Corm vegetables	USA <sup>12</sup>	SC 240	foliar	0.053-0.11		<sup>11</sup>	7
Cotton	Argentina <sup>12</sup>	SC 480	foliar	0.072-0.086	0.090-0.11	1	
Cotton	Argentina <sup>12</sup>	SC 480	foliar	0.019	0.024	3	
Cotton	Australia <sup>12</sup>	EO? 125	foliar (	0.075-0.10		3	28 <sup>14</sup>
Cotton	Bolivia	SC 480	foliar	0.072-0.086	0.090-0.11	1	
Cotton	Bolivia	SC 480	foliar	0.019	0.024	3	
Cotton	Brazil <sup>12</sup>	SC 480	foliar	0.012-0.072			7
Cotton	Cent Amer <sup>12</sup>	SC 480	foliar	0.012-0.072			7
Cotton	Colombia <sup>12</sup>	SC 120	foliar	0.012-0.036			28
Cotton	Costa Rica <sup>12</sup>	SC 480	foliar	0.036-0.060			0
Cotton	Guatemala <sup>12</sup>	SC 480	foliar	0.036-0.060			0
Cotton	Honduras <sup>12</sup>	SC 480	foliar	0.036-0.060			0
Cotton	Mexico	SC 480	foliar	0.036-0.060	0.009-0.030		28
Cotton	Nicaragua <sup>12</sup>	SC 480	foliar	0.036-0.060			0
Cotton	Paraguay <sup>12</sup>	SC 480	foliar	0.036-0.060			28
Cotton	Peru <sup>12</sup>	SC 120	foliar	0.006-0.024	0.0015-0.006		28
Cotton	USA <sup>12</sup>	SC 480	foliar	0.050-0.10		<sup>4</sup>	28
Cucumber	Israel <sup>12</sup>	SC 480	foliar		0.0096	3	7
Cucumber	Korea	WG 100	broadcast <sup>3</sup>	0.075	0.005	5	2
Cucumber	Lebanon <sup>12</sup>	SC 480			0.036-0.048		1
Cucumber	Mexico	SC 480	foliar	0.024-0.048			3
Cucumber	USA <sup>12</sup>	SC 240	foliar	0.070-0.14		<sup>4</sup>	1
Cucurbits	Peru <sup>12</sup>	SC 120	foliar	0.012-0.024	0.003-0.006		1
Cucurbits, except cucumbers	USA <sup>12</sup>	SC 240	foliar	0.070-0.14		<sup>4</sup>	3
Egg plant	Japan <sup>12</sup>	WG 250	broadcast <sup>2</sup>	0.30	0.010	2	1
Fruit, tropical	USA <sup>12</sup>	SC 240	foliar	0.070-0.18		<sup>4</sup>	1
Fruiting vegetables	USA <sup>12</sup>	SC 240	foliar	0.026-0.14		<sup>4</sup>	1

Crop	Country	Form	Application				PHI, days
			Method <sup>1</sup>	Rate, kg ai/ha	Spray conc. kg ai/hl	No.	
Fruiting vegetables	USA <sup>12</sup>	SC 240	foliar	0.053-0.18		<sup>4</sup>	1
Grain Amaranth	USA <sup>12</sup>	SC 480	foliar	0.050-0.11		<sup>4</sup>	7 <sup>8</sup>
Grapes	Chile <sup>12</sup>	SC 480	foliar	0.060-0.072			14
Grapes	Cyprus	SC 480	foliar	0.036-0.072	0.0072		7
Grapes	Israel <sup>12</sup>	SC 480	foliar		0.0048-0.0096		7
Grapes	Lebanon <sup>12</sup>	SC 480			0.0036-0.0048		7
Grapes	UAE	SC 480	foliar		0.0048-0.0072		7
Japanese radish	Japan <sup>12</sup>	WG 250	broadcast	0.30	0.010	3	7
Kiwifruit	NZ <sup>12</sup>	SC 120	foliar		0.0048		120
Leafy vegetables	USA <sup>12</sup>	SC 240	foliar	0.026-0.18		<sup>4</sup>	1
Lettuce	Australia <sup>12</sup>	SC 120	foliar (	0.048-0.096			3
Lettuce	Japan <sup>12</sup>	WG 250	broadcast <sup>2</sup>		0.010	3	3
Lettuce	Kenya <sup>12</sup>	SC 480	foliar	0.12	0.012-0.024		3
Lettuce	Peru	SC 240	foliar		0.006-0.0072	3	1
Lettuce	Uruguay <sup>12</sup>	SC 240	foliar		0.006-0.0072		1
Maize	Bolivia	SC 480	foliar	0.029	0.036	1	
Maize	Brazil <sup>12</sup>	SC 480	foliar	0.018-0.048			7
Maize	Cent Amer <sup>12</sup>	SC 480	foliar	0.018-0.048			7
Maize	Israel <sup>12</sup>	SC 480	foliar	0.048-0.072			1
Maize	Paraguay	SC 480	foliar	0.029	0.036-0.048	2	
Maize	Peru <sup>12</sup>	SC 120	foliar	0.012	0.003		14
Maize	USA <sup>12</sup>	SC 240	foliar	0.050-0.11		<sup>6</sup>	28 <sup>7</sup>
Maize	USA <sup>12</sup>	SC 480	broadcast or directed spray	0.035-0.11		<sup>6</sup>	28 <sup>7</sup>
Maize	Venezuela <sup>12</sup>	SC 480		0.048-0.096			28
Mango	Kenya <sup>12</sup>	SC 480	foliar		0.0096		21
Melon	Costa Rica <sup>12</sup>	SC 480	foliar	0.031			1
Melon	Guatemala <sup>12</sup>	SC 480	foliar	0.031			1
Melon	Honduras <sup>12</sup>	SC 480	foliar	0.031			1
Melon	Israel <sup>12</sup>	SC 480	foliar	0.096			7
Melon	Mexico	SC 480	foliar	0.024-0.048			3
Millet, Pearl	USA <sup>12</sup>	SC 480	foliar	0.050-0.11		<sup>4</sup>	7 <sup>8</sup>
Millet, Proso	USA <sup>12</sup>	SC 480	foliar	0.050-0.11		<sup>4</sup>	7 <sup>8</sup>
Milo	USA <sup>12</sup>	SC 480	foliar	0.050-0.11		<sup>4</sup>	7 <sup>8</sup>
Nectarine	Chile <sup>12</sup>	SC 480	foliar	0.060-0.072	0.0072-0.0096		14
Nectarine	Israel <sup>12</sup>	SC 480	foliar		0.0072-0.0096		7
Oats	USA <sup>12</sup>	SC 240	foliar	0.035-0.11		<sup>9</sup>	21 <sup>10</sup>
Oats	USA <sup>12</sup>	SC 480	foliar	0.050-0.11		<sup>9</sup>	21 <sup>10</sup>
Onion	Israel <sup>12</sup>	SC 480	foliar	0.072-0.096			
Onion	Kenya <sup>12</sup>	SC 480	foliar	0.12	0.012-0.024		1
Passion fruit	Kenya <sup>12</sup>	SC 480	foliar	0.14	0.014		1
Peach	Chile <sup>12</sup>	SC 480	foliar	0.060-0.072	0.0072-0.0096		14
Peach	Japan <sup>12</sup>	SC 200	broadcast		0.010	3	3
Pear	Israel <sup>12</sup>	SC 480	foliar		0.0096		21
Peas	Kenya <sup>12</sup>	SC 480	foliar	0.12	0.012-0.024		1
Peas, dried	USA <sup>12</sup>	SC 240	foliar	0.053-0.11		<sup>6</sup>	28 <sup>5</sup>
Peas, succulent	USA <sup>12</sup>	SC 240	foliar	0.053-0.11		<sup>4</sup>	3 <sup>5</sup>
Peppers	Australia <sup>12</sup>	SC 120	foliar (	0.048-0.096			1
Peppers	Costa Rica <sup>12</sup>	SC 120	foliar	0.036-0.060			1

Crop	Country	Form	Application				PHI, days
			Method <sup>1</sup>	Rate, kg ai/ha	Spray conc. kg ai/hl	No.	
Peppers	Guatemala <sup>12</sup>	SC 120	foliar	0.036-0.060			1
Peppers	Honduras <sup>12</sup>	SC 120	foliar	0.036-0.060			1
Peppers	Israel <sup>12</sup>	SC 480	foliar		0.0096		1
Peppers	Japan <sup>12</sup>	WG 250	broadcast <sup>2</sup>		0.010	2	1
Peppers	Kenya <sup>12</sup>	SC 480	foliar	0.19-0.24	0.019-0.048		3
Peppers	Mexico	SC 480	foliar	0.036-0.060			1
Peppers	Paraguay <sup>12</sup>	SC 480	foliar	0.036-0.060			1
Peppers	Peru	SC 240	foliar	0.012-0.024	0.0072	3	1
Peppers	Uruguay <sup>12</sup>	SC 240	foliar		0.0072		1
Pistachios	USA <sup>12</sup>	SC 240	foliar	0.070-0.18	0.0019-0.0047	<sup>4</sup>	14
Plums	Israel <sup>12</sup>	SC 480	foliar		0.0072-0.0096		7
Pome fruit	NZ <sup>12</sup>	SC 120	foliar		0.0048		3
Popcorn	USA <sup>12</sup>	SC 240	foliar	0.050-0.11		<sup>6</sup>	28 <sup>7</sup>
Popcorn	USA <sup>12</sup>	SC 480	broadcast or directed spray	0.035-0.11		<sup>6</sup>	28 <sup>7</sup>
Potato	Brazil <sup>12</sup>	SC 480	foliar	0.16-0.20			3
Potato	Cent Amer <sup>12</sup>	SC 480	foliar	0.16-0.20			3
Potato	Chile <sup>12</sup>	SC 480	foliar	0.048-0.072			7
Potato	Colombia <sup>12</sup>	SC 120	foliar	0.036-0.060			1
Potato	Cyprus	SC 480	foliar	0.12-0.36	0.024-0.036		0
Potato	Israel <sup>12</sup>	SC 480	foliar	0.048-0.096			14
Potato	Korea	WG 100	broadcast		0.005	5	7
Potato	Lebanon <sup>12</sup>	SC 480		0.24-0.34			0
Potato	Turkey <sup>12</sup>	SC 480	foliar		0.0048		1
Potato	UAE	SC 480	foliar		0.0048-0.024		1
Rye	USA <sup>12</sup>	SC 240	foliar	0.035-0.11		<sup>9</sup>	21 <sup>10</sup>
Rye	USA <sup>12</sup>	SC 480	foliar	0.050-0.11		<sup>9</sup>	21 <sup>10</sup>
Snowpeas	Kenya <sup>12</sup>	SC 480	foliar	0.12	0.012-0.024		3
Sorghum	USA <sup>12</sup>	SC 480	foliar	0.050-0.11		<sup>4</sup>	7 <sup>8</sup>
Soya beans	Argentina <sup>12</sup>	SC 480	foliar	0.024	0.030	1	
Soya beans	Bolivia	SC 480	foliar	0.024	0.030	1	
Soya beans	Brazil <sup>12</sup>	SC 480	foliar	0.006-0.024			9
Soya beans	Cent Amer <sup>12</sup>	SC 480	foliar	0.006-0.024			9
Soya beans	Paraguay	SC 480	foliar	0.036	0.045-0.060	2	
Soya beans	USA <sup>12</sup>	SC 480	foliar	0.035-0.070		<sup>6</sup>	28 <sup>5</sup>
Spinach	Australia <sup>12</sup>	SC 120	foliar (	0.048-0.096			3
Spinach	Peru	SC 240	foliar		0.006-0.0072	3	1
Spinach	Uruguay <sup>12</sup>	SC 240	foliar		0.006-0.0072		1
Stone fruit <sup>13</sup>	USA <sup>12</sup>	SC 240	foliar	0.070-0.14	0.0019-0.0037	<sup>4</sup>	7, 14
Strawberries	Kenya <sup>12</sup>	SC 480	foliar	0.072-0.14	0.0096-0.014		2
Strawberry	Israel <sup>12</sup>	SC 480	foliar	0.096			1
Strawberry	UAE	SC 480	foliar		0.014-0.019		1-2
Sweet corn	Australia <sup>12</sup>	SC 120	foliar (	0.048-0.096			
Sweet corn	USA <sup>12</sup>	SC 240	foliar	0.050-0.11		<sup>4</sup>	1
Tea	Japan <sup>12</sup>	SC 200	broadcast		0.010	2	7
Teosinte	USA <sup>12</sup>	SC 480	broadcast or directed spray	0.035-0.11		<sup>6</sup>	28 <sup>7</sup>
Tomato	Argentina <sup>12</sup>	SC 480	foliar	0.072-0.11	0.0072	3	3
Tomato	Australia <sup>12</sup>	SC 120	foliar (	0.048-0.096			1
Tomato	Brazil <sup>12</sup>	SC 480	foliar	0.048-0.060			3

Crop	Country	Form	Application				PHI, days
			Method <sup>1</sup>	Rate, kg ai/ha	Spray conc. kg ai/hl	No.	
Tomato	Cent Amer <sup>12</sup>	SC 480	foliar	0.048-0.060			3
Tomato	Chile <sup>12</sup>	SC 480	foliar	0.058-0.072	0.0048-0.0072		1
Tomato	Costa Rica <sup>12</sup>	SC 120	foliar	0.036-0.060			1
Tomato	Cyprus	SC 480	foliar	0.060-0.17	0.012-0.017		1
Tomato	Guatemala <sup>12</sup>	SC 120	foliar	0.036-0.060			1
Tomato	Honduras <sup>12</sup>	SC 120	foliar	0.036-0.060			1
Tomato	Israel <sup>12</sup>	SC 480	foliar	0.096			3
Tomato	Japan <sup>12</sup>	WG 250	broadcast <sup>2</sup>		0.010	2	1
Tomato	Kenya <sup>12</sup>	SC 480	foliar	0.19-0.24	0.019-0.048		3
Tomato	Korea	WG 100	broadcast <sup>3</sup>	0.075	0.005	5	2
Tomato	Lebanon <sup>12</sup>	SC 480			0.036-0.048		1
Tomato	Mexico	SC 480	foliar	0.036-0.060			1
Tomato	NZ <sup>12</sup>	SC 120	foliar	0.048			3
Tomato	Paraguay <sup>12</sup>	SC 480	foliar	0.036-0.060			1
Tomato	Peru	SC 240	foliar	0.012-0.024	0.0072	3	1
Tomato	Peru <sup>12</sup>	SC 120	foliar	0.012-0.024	0.003-0.006		1
Tomato	UAE	SC 480	foliar		0.036-0.048		1
Tomato	Uruguay <sup>12</sup>	SC 240	foliar		0.0072		1
Triticale	USA <sup>12</sup>	SC 240	foliar	0.035-0.11		<sup>9</sup>	21 <sup>10</sup>
Triticale	USA <sup>12</sup>	SC 480	foliar	0.050-0.11		<sup>9</sup>	21 <sup>10</sup>
Tuber vegetables	USA <sup>12</sup>	SC 240	foliar	0.053-0.11		<sup>11</sup>	7
Vegetables	UAE	SC 480	foliar		0.012-0.036		1
Vegetables, leafy	Cyprus	SC 480	foliar	0.060-0.14	0.012-0.014		1
Watermelon	Korea	WG 100	broadcast		0.005	5	14
Watermelon	Mexico	SC 480	foliar	0.024-0.048			3
Wheat	USA <sup>12</sup>	SC 240	foliar	0.035-0.11		<sup>9</sup>	21 <sup>10</sup>
Wheat	USA <sup>12</sup>	SC 480	foliar	0.050-0.11		<sup>9</sup>	21 <sup>10</sup>
Zucchini	Mexico	SC 480	foliar	0.024-0.048			3

<sup>1</sup> ( aerial application

<sup>2</sup> field or glasshouse use

<sup>3</sup> glasshouse use

<sup>4</sup> max total application 0.50 kg ai/ha

<sup>5</sup> do not feed forage or hay to meat or dairy animals

<sup>6</sup> max total application 0.21 kg ai/ha

<sup>7</sup> PHI 28 days for grain or fodder, 7 days for forage

<sup>8</sup> PHI 7 days for grain or fodder, 14 days for forage

<sup>9</sup> max total application 0.31 kg ai/ha

<sup>10</sup> PHI 21 days for grain or straw, 14 days for forage or hay

<sup>11</sup> Max. total application 0.37 kg ai/ha

<sup>12</sup> Label or label copy provided

<sup>13</sup> Stone fruit, USA. PHI 7 days for cherries, plums and prunes. PHI 14 days for peaches, nectarines and apricots.

<sup>14</sup> Cotton, Australia. Do not allow livestock to graze treated cotton crop, stubble or gin trash.

US labels for spinosad list the crops included in crop groups as follows.

Citrus: including but not limited to: grapefruit, lemons, limes, oranges and tangerines.

Cole crops: including but not limited to: broccoli, broccoli raab, Brussels sprouts, cabbage, cauliflower, cavalo, Chinese broccoli, Chinese cabbage (bok choy), Chinese cabbage (napa), Chinese mustard cabbage (gai choy), collards, kale, kohlrabi, mizuna, mustard greens, mustard spinach, rape greens and turnip greens.

Cucurbit crops: including but not limited to: cucumber, edible gourds, muskmelons (cantaloupe, honeydew, etc), pumpkin, summer squash, watermelon and winter squash.

Fruiting vegetable crops: egg plant, ground cherry, pepino, pepper, tomatillo and tomato.

Leafy vegetables: including but not limited to: arugula, celery, chervil, cilantro, corn salad, cress, dandelion, dock, edible chrysanthemum, endive, fennel, garden purslane, head lettuce, leaf lettuce, parsley, radicchio, rhubarb, spinach, Swiss chard, turnip greens and water cress.

Tuber and corm vegetables: including but not limited to: cassava, chayote root, Chinese artichoke, ginger, Jerusalem artichoke, potatoes, sweet potatoes, turmeric and yams.

Stone fruit: including but not limited to: apricots, cherries, nectarines, peaches, plums and prunes.

Succulent and dried beans and peas including but not limited to: Adzuki bean, blackeyed pea, chickpea, cowpea, crowder pea, edible-pod pea, English pea, fava bean, field bean, field pea, Garbanzo bean, garden pea, green pea, kidney bean, lentil, lima bean, lupins, mung bean, navy bean, pigeon pea, pinto bean, runner bean, snap bean, snow pea, sugar snap pea, tepary bean, wax bean and yardlong bean.

Tropical fruit: acerola, atemoya, avocado, biriba, black sapote, canistel, cherimoya, custard apple, feijoa, guava, ilama, jaboticaba, longan, lychee, mamey sapote, mango, papaya, passionfruit, pulasan, rambutan, sapodilla, soursop, Spanish lime, star apple, starfruit, sugar apple, wax jambu and white sapote.

Table 32. Registered uses of spinosad for external treatment of animals.

Animal	Country	Form	Application			whp slaughter, days	whp milk, days
			Method	Rate	Conc.		
Beef cattle	USA <sup>1</sup>	SC 25	pour-on	2 mg ai/kg bw	25 g ai/l		
Dairy cattle, non-lactating	USA <sup>1</sup>	SC 25	pour-on	2 mg ai/kg bw	25 g ai/l		
Dairy cattle, lactating	USA <sup>1</sup>	SC 25	pour-on	2 mg ai/kg bw	25 g ai/l		
Cattle, beef and dairy	USA <sup>1</sup>	SC 25	spray	0.38-0.76 g ai/animal	400 mg ai/l		
Sheep	Australia <sup>1</sup>	25	jetting	0.5 l fluid per month of wool growth	25 mg ai/l	0	<sup>2</sup>
Sheep	Australia <sup>1</sup>	25	wound dressing	Apply 1-2 l on wound	25 mg ai/l	0	<sup>2</sup>

whp: withholding period

<sup>1</sup> Label or label copy provided.

<sup>2</sup> Do not use on female sheep producing milk.

## RESIDUES FROM SUPERVISED TRIALS

The Meeting received information on supervised field trials of spinosad on the following crops.

Fruits	Table 34	citrus, Japan, USA
	Table 35	citrus, Argentina
	Table 36	apples, France, Italy, Japan, Spain, USA
	Table 37	stone fruit, USA
	Table 38	stone fruit, Chile, Japan
	Table 39	grapes, France, Italy, Spain
	Table 40	grapes, Chile
	Table 41	strawberries, Belgium, Greece, Italy, Spain, UK



Vegetables	Table 42	blueberries, USA
	Table 43	kiwifruit, New Zealand
	Table 44	Brassica vegetables, Australia, New Zealand
	Table 45	cabbage and Chinese cabbage, Japan
	Table 46	Brassica vegetables, USA
	Table 47	cucurbits, USA
	Table 48	tomatoes, Australia, New Zealand
	Table 49	tomatoes, Argentina, Brazil
	Table 50	tomatoes, Greece, Italy, Spain, UK
	Table 51	tomatoes, Mexico, USA
	Table 52	peppers, USA
	Table 53	sweet peppers, Australia, Greece, Italy, Mexico, Spain, UK
	Table 54	sweet corn, USA
Cereals	Table 55	egg plant, Japan
	Table 55	lettuce, Australia
	Table 57	leafy vegetables, USA
	Table 58	leafy vegetables, celery, USA
	Table 59	legume vegetables, USA
	Table 60	navy beans, Australia
	Table 61	soya beans, USA
	Table 62	soya beans, Argentina, Brazil
	Table 63	potatoes, Brazil, USA
	Table 64	Japanese radish, Japan
Tree nuts	Table 65	maize, sorghum, wheat, USA
	Table 66	maize, Argentina, Brazil
Oilseeds	Table 67	almonds, USA
Animal feeds	Table 68	cotton seed, Australia, USA
	Table 69	cereal forage and fodder, USA
	Table 70	cotton trash, Australia
	Table 71	navy bean forage, Australia
	Table 72	almond hulls, USA

Trials were generally well documented with full laboratory and field reports. Laboratory reports included method validation, with batch recoveries at spiking levels similar to those occurring in samples from the supervised trials. Dates of analyses were also provided. Field reports provided data on the sprayers used and their calibration, plot size, residue sample size and sampling dates.

Where residues were not detected, they are recorded in the Tables as below the limit of quantification (LOQ), e.g. <0.01 mg/kg. Residue data, application rates and spray concentrations have generally been rounded to 2 significant figures or, for residues near the LOQ, to 1 significant figure. Although trials included control plots, no control data are recorded except where residues in control samples exceeded the LOQ; these residues are prefixed with a "c". Residues are recorded uncorrected for percentage recoveries in most cases. In some trials, for example on peppers in Spain in 1997-98, there is no clear statement whether results are adjusted for recoveries or not.

The conditions of the supervised residue trials are shown in Table 33. Most trials were not replicated. US trials were generally on single plots with 2 independent random field samples per sampling, and European and Australian trials generally on single plots with a single random sample per sampling. South American trials were generally on 3 replicate plots with a sample from each plot per sampling.

Periods of freezer storage between sampling and analysis were recorded for all trials and were within the acceptable proven stability period of 1 year for many commodities except in a few cases. Excessive freezer storage periods are noted.

Supervised trials on the direct treatment of animals were reported from Australia and the USA. Trials on sheep and dairy cattle were well described with trial conditions comparable to GAP or proposed GAP.

Feeding studies were provided for dairy cows and laying hens.

Table 33. Sprayers, plot sizes and sample sizes in the spinosad supervised trials on crops.

Crop	Country	Year	Sprayer	Plot size	Sample size
almonds	USA	1996	airblast	220-390 m <sup>2</sup>	3-10 kg
apple	France	1999	backpack, airblast	32-120 m <sup>2</sup>	24 fruits
apple	Italy	1999	backpack, spraygun	68-94 m <sup>2</sup>	24 fruits
apple	Spain	1998-9	knapsack, motorpump backpack	40-50 m <sup>2</sup>	16-24 fruits
apple	USA	1995	tractor airblast, backpack mistblower	71-595 m <sup>2</sup>	24-32 fruits
blueberries	USA	1998			1-1.5 kg
broccoli	Australia	1997	precision gas sprayer	12-48 m <sup>2</sup>	
broccoli	USA	1995	backpack with boom, tractor mounted boom	45-204 m <sup>2</sup>	12 heads
Brussels sprouts	Australia	1997	precision gas sprayer	50 m <sup>2</sup>	
cabbage	USA	1995	backpack with boom, tractor mounted boom	45-124 m <sup>2</sup>	12 heads
cauliflower	Australia and NZ	1996-7	precision gas sprayer	12-15 m <sup>2</sup>	
celery	USA	1996	backpack	75-100 m <sup>2</sup>	12 plants
cherries	USA	1997	airblast	160-270 m <sup>2</sup>	1-2 kg
cotton	Argentina	1996-7	backpack	100-130 m <sup>2</sup>	1-2 kg
cotton	Australia	1994,96	tractor mounted boom, precision sprayer, spinning disc	90-135 m <sup>2</sup>	
cotton	Brazil	1994	CO <sub>2</sub> backpack	21-180 m <sup>2</sup>	
cotton	USA	1993	tractor mounted compressed air, knapsack compressed air	70-680 m <sup>2</sup>	1-6 kg
cucumber	USA	1997	backpack with boom, tractor mounted boom	80-340 m <sup>2</sup>	0.6-5 kg
grapefruit	USA	1996	airblast	110-840 m <sup>2</sup>	5-10 kg
grapes	France	1997-8	mistblower	80-230 m <sup>2</sup>	0.6-1 kg
grapes	Italy	1997-8	motorpump backpack, plot sprayer	90-150 m <sup>2</sup>	1-2 kg
grapes	Spain	1997-8	motorpump backpack	50-110 m <sup>2</sup>	1 kg
head lettuce	USA	1996	backpack with boom	85-93 m <sup>2</sup>	12 plants
kiwifruit	NZ	1996-9	airblast, handgun	15 m <sup>2</sup>	
leaf lettuce	USA	1996	backpack sprayer with boom	75-100 m <sup>2</sup>	12 plants
lemon	USA	1996-7	airblast	350-540 m <sup>2</sup>	3-5 kg
lettuce	Australia	1998	precision sprayer	45-70 m <sup>2</sup>	12 lettuce
lettuce	USA	1996	CO <sub>2</sub> backpack sprayer	74-93 m <sup>2</sup>	12 heads
maize	Argentina	1999	CO <sub>2</sub> backpack sprayer	24 m <sup>2</sup>	2 kg
maize	Brazil	1995	CO <sub>2</sub> backpack sprayer	72-108 m <sup>2</sup>	
maize	USA	1997	backpack with boom, tractor mounted boom	90-150 m <sup>2</sup>	12 areas
muskmelon	USA	1997	backpack with boom, tractor mounted boom	84-335 m <sup>2</sup>	3-30 kg
mustard greens	USA	1995	backpack with boom	45-289 m <sup>2</sup>	20-60 plants, 1.5-3 kg
navy beans	Australia	1995		22-30 m <sup>2</sup>	
nectarine	Chile	1999	airblast	360-810 m <sup>2</sup>	2 kg
orange	USA	1996-7	airblast	220-700 m <sup>2</sup>	3-8 kg
peach	USA	1997	airblast	140-230 m <sup>2</sup>	24 fruit
peppers	Australia	1997	precision gas sprayer	18-30 m <sup>2</sup>	
peppers	Italy	1997-8	plot sprayer	16-40 m <sup>2</sup>	1 kg, 12 fruit
peppers	Spain	1997-8	motorpump backpack	8-40 m <sup>2</sup>	8-12 fruit
peppers	UK	1997-8	plot sprayer	13 m <sup>2</sup>	12 fruit
peppers	USA	1995	backpack with boom, tractor mounted boom	41-120 m <sup>2</sup>	1-3 kg
plum	USA	1997	airblast	140-250 m <sup>2</sup>	24 fruit min
potato	Brazil	1995-6	CO <sub>2</sub> backpack	36-40 m <sup>2</sup>	2 kg
potato	USA	1997	CO <sub>2</sub> backpack, tractor mounted CO <sub>2</sub> sprayer, tractor powered pump	30-124 m <sup>2</sup>	2-5-3.5 kg

Crop	Country	Year	Sprayer	Plot size	Sample size
prune	USA	1997	airblast	160-210 m <sup>2</sup>	24 fruit min
snap beans	USA	1997	backpack, tractor mounted	56-120 m <sup>2</sup>	1.3 kg
snow peas	USA	1997	backpack, tractor mounted	90-120 m <sup>2</sup>	1.3 kg
sorghum	USA	1997	backpack with boom, tractor mounted boom	93-1300 m <sup>2</sup>	12 plants
soya beans	Argentina	1995-6	CO <sub>2</sub> backpack	16-42 m <sup>2</sup>	1-2 kg
soya beans	Brazil	1994-5	CO <sub>2</sub> backpack	28-72 m <sup>2</sup>	
soya beans	USA	1997	tractor mounted, backpack	48-93 m <sup>2</sup>	1.3 kg
spinach	USA	1996	backpack with boom, tractor mounted boom, CO <sub>2</sub> backpack	56-120 m <sup>2</sup>	12-24 plants
strawberry	Belgium	1998	boom spray	24 m <sup>2</sup>	1.2 kg
strawberry	Italy	1999	spray gun, motorpump backpack, knapsack	20-63 m <sup>2</sup>	1-2 kg
strawberry	Spain	1998-9	compressed air sprayer	10-22 m <sup>2</sup>	0.5-1.5 kg
strawberry	UK	1999	compressed air sprayer	10 m <sup>2</sup>	1 kg
summer squash	USA	1997	backpack with boom, tractor mounted boom	84-120 m <sup>2</sup>	1.5-5 kg
sweet corn	USA	1997	tractor mounted boom , backpack with boom	75-250 m <sup>2</sup>	12 ears, 12 stalks
tomato	Argentina	1996-7	CO <sub>2</sub> backpack	7-640 m <sup>2</sup>	2 kg
tomato	Australia	1996-7	precision gas sprayer	10-60 m <sup>2</sup>	20 fruit
tomato	Brazil	1995-6	CO <sub>2</sub> backpack	6-14 m <sup>2</sup>	2 kg
tomato	Greece	1998	precision gas sprayer	14-16 m <sup>2</sup>	2 kg
tomato	Italy	1997-8	spray gun	13-46 m <sup>2</sup>	1 kg, 24 fruit
tomato	Spain	1997-8	motorpump backpack	8-44 m <sup>2</sup>	1 kg, 12 fruit
tomato	UK	1997-8	plot sprayer	13-21 m <sup>2</sup>	1.4 kg, 12 fruit
tomato	USA	1995-6	tractor mounted boom, backpack with boom, CO <sub>2</sub> backpack	41-200 m <sup>2</sup>	12-24 fruit
wheat	USA	1997	tractor mounted boom	140-230 m <sup>2</sup>	12 areas

Table 34. Spinosad residues in citrus fruits from supervised trials in Japan and the USA. Double-underlined residues are from treatments according to GAP and are valid for the estimation of maximum residue levels.

Country, year (variety)	Application					PHI, days	Spinosyn residues, mg/kg				Ref.
	Form	kg ai/ha	kg ai/hl	water, l/ha	no.		A HPLC	D HPLC	Total <sup>1</sup> HPLC	IA <sup>2</sup>	
Japan, 1995 (Mandarin)	WG 200	0.60	0.01	6000	g 2 5	21 28	p 0.09 p 0.05	p <0.01 p <0.01			GHF-P-1683
						21 28	f <0.01 f <0.01	f <0.01 f <0.01			
Japan, 1996 (Mandarin)	WG 200	0.60	0.01	6000	2	14 21 28	p <0.01 p <0.01 p <0.01	p <0.01 p <0.01 p <0.01			GHF-P-1682
						14 21 28	f <0.01 f <0.01 f <0.01	f <0.01 f <0.01 f <0.01			
USA (AZ), 1996 (Marsh Ruby grapefruit)	SC 480	0.070 +0.10 +0.15 +0.18		500	4	1 4				<u>0.021</u> <u>0.018</u>	RES96023 AZ22

Country, year (variety)	Application					PHI, days	Spinosyn residues, mg/kg				Ref.
	Form	kg ai/ha	kg ai/hl	water, l/ha	no.		A HPLC	D HPLC	Total <sup>1</sup> HPLC	IA <sup>2</sup>	
USA (CA), 1996 (Ruby Red grapefruit)	SC 480	0.070 +0.10 +0.15 +0.18		3700	4	1 4 7 14				<u>0.013</u> 0.012 <0.01 <0.01	RES96023 CA19
USA (FL), 1996 (Flame grapefruit)	SC 480	0.067 +0.10 +0.15 +0.18		3600 +3700 +3800 +4200	4	1 4				<u>0.086</u> 0.036	RES96023 FL11
USA (FL), 1996 (Ruby Red grapefruit)	SC 480	0.070 +0.10 +0.15 +0.18		190	4	1 4	<u>0.16</u> 0.072	<u>0.025</u> 0.011	0.19 0.091	0.13 0.094	RES96023 FL09
USA (FL), 1996 (White Marsh grapefruit)	SC 480	0.073 +0.10 +0.16 +0.19		490	4	1 4	<u>0.051</u>	<u>0.01</u>	0.061	0.063 0.044	RES96023 FL10
USA (TX), 1996 (Henderson Rio Red grapefruit)	SC 480	0.070 +0.10 +0.15 +0.18		3600	4	1 4				<u>0.030</u> 0.020	RES96023 TX13
USA (AZ), 1996 (Limonera lemons)	SC 480	0.14 +0.21 +0.30 +0.36		7400 +7500 +7200 +7200	4	1 4 7 14				0.048 0.01 0.01 <0.01	RES96023 AZ24
USA (AZ), 1996 (Lisbon lemons)	SC 480	0.070 +0.10 +0.15 +0.18		190	4	1 4				<u>0.14</u> 0.12	RES96023 AZ23
USA (CA), 1996 (Lisbon lemons)	SC 480	0.070 +0.10 +0.15 +0.18		460	4	1 4	<u>0.021</u>	<0.01	0.026	0.033 0.023	RES96023 CA20
USA (CA), 1996 (Lisbon lemons)	SC 480	0.070 +0.10 +0.15 +0.18		3800	4	1 4	<u>0.037</u> 0.023	<0.01 <0.01	0.051 0.029	0.056 0.046	RES96023 CA21
USA (FL), 1996 (Eureka lemons)	SC 480	0.075 +0.10 +0.15 +0.19		3900	4	1 4				<u>0.056</u> 0.035	RES96023 FL12

Country, year (variety)	Application					PHI, days	Spinosyn residues, mg/kg				Ref.
	Form	kg ai/ha	kg ai/hl	water, l/ha	no.		A HPLC	D HPLC	Total <sup>1</sup> HPLC	IA <sup>2</sup>	
USA (CA), 1996 (Attwood oranges)	SC 480	0.070 +0.10 +0.15 +0.18		470	4	1 4 o <sup>3</sup> 1 4 p <sup>4</sup> 1 4				<u>0.046</u> 0.022  0.01 0.01  0.10 0.052	RES96023 CA17
USA (CA), 1996 (Cutter Valencia oranges)	SC 480	0.070 +0.10 +0.15 +0.18		3700	4	1 4 7 14				<u>0.01</u> 0.01 0.01 0.01	RES96023 CA16
USA (CA), 1996 (Navel oranges)	SC 480	0.070 +0.10 +0.15 +0.18		3700	4	1 4 o <sup>3</sup> 1 4 p <sup>4</sup> 1 4				<u>0.017</u> 0.01  0.01 0.01  0.021 0.035	RES96023 CA18
USA (CA), 1996 (Washington Navel oranges)	SC 480	0.070 +0.10 +0.15 +0.18		190	4	1 4				<u>0.11</u> 0.060	RES96023 CA15
USA (FL), 1996 (Ambersweet oranges)	SC 480	0.070 +0.10 +0.15 +0.18		190	4	1 4				<u>0.044</u> 0.033	RES96023 FL02
USA (FL), 1996 (Hamlin oranges)	SC 480	0.070 +0.10 +0.15 +0.18		480	4	1 4 o <sup>3</sup> 1 4 p <sup>4</sup> 1 4	<u>0.11</u> 0.05  0.01 0.01  0.35 0.18	<u>0.016</u> 0.01 0.01 0.01  0.050 0.020	0.14 0.06 0.01 0.01  0.42 0.23	0.14 0.06 0.01 0.01  0.64 0.30	RES96023 FL01
USA (FL), 1996 (Hamlin oranges)	SC 480	0.070 +0.10 +0.15 +0.18		3670 +3800 +4060 +3700	4	1 4 o <sup>3</sup> 1 4 p <sup>4</sup> 1 4	<u>0.12</u> 0.034	<u>0.019</u> 0.01	0.18 0.050	0.20 0.072  0.01 0.01  0.78 0.32	RES96023 FL03
USA (FL), 1996 (Hamlin oranges)	SC 480	0.075 +0.10 +0.15 +0.18		3800	4	1 4				<u>0.15</u> 0.10	RES96023 FL04

Country, year (variety)	Application					PHI, days	Spinosyn residues, mg/kg				Ref.
	Form	kg ai/ha	kg ai/hl	water, l/ha	no.		A HPLC	D HPLC	Total <sup>1</sup> HPLC	IA <sup>2</sup>	
USA (FL), 1996 (Hamlin oranges)	SC 480	0.070 +0.10 +0.14 +0.17		450	4	1 4				<u>0.070</u> 0.032	RES96023 FL07
USA (FL), 1996 (Hamlin oranges)	SC 480	0.070 +0.10 +0.15 +0.19		3900	4	1 4	<u>0.047</u> 0.025	<u>0.006</u> 0.003	0.066 0.035	0.081 0.066	RES96023 FL08
USA (FL), 1996 (Navel oranges)	SC 480	0.070 +0.10 +0.15 +0.18		190	4	1 4 o <sup>3</sup> 1 4 p <sup>4</sup> 1 4				<u>0.053</u> 0.011  <0.01 <0.01  0.080 0.054	RES96023 FL06
USA (FL), 1996 (Pineapple oranges)	SC 480	0.076 +0.10 +0.15 +0.18		3800	4	1 4				<u>0.14</u> 0.084	RES96023 FL05
USA (TX), 1996 (Navel oranges)	SC 480	0.070 +0.10 +0.15 +0.18		200	4	1 4 o <sup>3</sup> 1 4 p <sup>4</sup> 1 4				<u>0.031</u> 0.016  <0.01 <0.01  0.046 0.11	RES96023 TX14

<sup>1</sup> Total includes spinosyns A, D, B, N-demethyl-D and K

<sup>2</sup> Immunoassay

<sup>3</sup> peeled orange

<sup>4</sup> orange peel

<sup>5</sup> g: glasshouse, p: peel, f: flesh.

Table 35. Spinosad residues in citrus from supervised trials in Argentina. Replicate residues represent samples from split or replicate plots.

Location, year (variety)	Application					PHI, days	Residues, mg/kg				Ref.
	Form	kg ai/ha	kg ai/hl	water, l/ha	no.		spinosyn A		spinosyn D		
Buenos Aires, 1999 (Frost Navel orange)	SC 480		0.0072	2060	2	0 3 7 10 15	0.05 0.11 0.06 0.01 0.01 0.01 0.02 0.01 0.01 <0.01 (3) <0.01 0.01 <0.01	<0.01 0.02 0.01 <0.01 (3) <0.01 (3) <0.01 (3) <0.01 (3)	GHB-P 449		
Buenos Aires, 1999 (Frost Navel orange)	SC 480		0.014	2060	2	0 3 7 10 15	0.11 0.14 0.10 0.11 0.03 0.02 0.02 0.06 0.04 <0.01 0.02 0.02 <0.01 <0.01 0.01	0.03 0.03 0.02 0.02 <0.01 <0.01 <0.01 (3) <0.01 (3) <0.01 (3)	GHB-P 449		

Table 36. Spinosad residues in apples from supervised trials in France, Italy, Japan, Spain and the USA. US residues are the mean of 2 independent composite samples. Double-underlined residues are from treatments according to GAP and are valid for the estimation of maximum residue levels.

Country, year, (variety)	Application						PHI, days	Spinosyn residues, mg/kg						Ref.
	Form	kg ai/ha	kg ai/hl	water, l/ha	no.	A		D	K	B	B of D	Total		
France, 1998 (Golden)	SC 480	0.29	0.030	960	3 4	40 0 1 3 7 14	<0.01 0.18 0.14 0.09 0.09 0.02	<0.01 0.04 0.04 0.02 0.02 0.01	<0.01 0.01 0.01 0.01 0.01 0.01	<0.01 0.02 0.02 0.01 0.01 0.01	<0.01 0.25 0.21 0.13 0.12 0.03	GHE-P-8252		
France, 1999 (Gloster)	SC 480	0.30 +0.27 +0.30 +0.35	0.029	1000 +920 +1000 +1200	4	7 14	0.03 0.03	<0.01 0.01	<0.01 0.01	<0.01 0.01	<0.01 0.01	0.04 0.04	GHE-P-8527 R99-001C	
France, 1999 (Golden)	SC 480	0.31	0.029	1100	3 4	38 0 1 3 7 14	<0.01 0.16 0.18 0.06 0.03 0.01	<0.01 0.03 0.03 0.01 0.01 0.01	<0.01 0.01 0.01 0.01 0.01 0.01	<0.01 0.01 0.01 0.01 0.01 0.01	<0.01 0.20 0.21 0.08 0.04 0.01	GHE-P-8527 R99-001B		
France, 1999 (Golden)	SC 480	0.30	0.030	1000	3 4	49 0 1 3 7 14	<0.01 0.25 0.19 0.03 0.02 0.01	<0.01 0.05 0.04 0.01 0.01 0.01	<0.01 0.01 0.01 0.01 0.01 0.01	<0.01 0.01 0.01 0.01 0.01 0.01	<0.01 0.30 0.23 0.04 0.02 0.01	GHE-P-8528 002A		
France, 1999 (Jonagold)	SC 480	0.29	0.029	1000	3 4	33 0 1 3 7 14	<0.01 0.20 0.16 0.05 0.01 0.01	<0.01 0.04 0.03 0.01 0.01 0.01	<0.01 0.01 0.01 0.01 0.01 0.01	<0.01 0.01 0.01 0.01 0.01 0.01	<0.01 0.24 0.20 0.07 0.01 0.01	GHE-P-8527 R99-001A		
France, 1999 (Red Chief)	SC 480	0.29	0.040	730	4	7 14	0.01 0.01	<0.01 0.01	<0.01 0.01	<0.01 0.01	<0.01 0.01	0.01 0.01	GHE-P-8528 002B	
France, 1999 (Royal Gala)	SC 480	0.29 +0.30 +0.32 +0.31	0.029	1000 +1050 +1100 +1080	4	7 14	0.03 0.03	<0.01 0.01	<0.01 0.01	<0.01 0.01	<0.01 0.01	0.03 0.03	GHE-P-8527 R99-001D	

Country, year, (variety)	Application					PHI, days	Spinosyn residues, mg/kg						Ref.
	Form	kg ai/ha	kg ai/hl	water, l/ha	no.		A	D	K	B	B of D	Total	
Italy, 1999 (Golden Delicious)	SC 480	0.29	0.029 +0.019 +0.019 +0.019	980 +1500 +1500 +1500	4	7 14	0.05 0.01	<0.01 0.01	<0.01 0.01	<0.01 0.01	<0.01 0.01	0.05 0.01	GHE-P- 8531
Italy, 1999 (Red Delicious)	SC 480	0.29	0.024	1200	3 4	49 0 1 3 7 14	<0.01 0.13 0.11 0.09 0.04 0.01	<0.01 0.02 0.02 0.02 0.01 0.01	<0.01 0.01 0.01 0.01 0.01 0.01	<0.01 0.01 0.01 0.01 0.01 0.01	<0.01 0.16 0.13 0.11 0.05 0.01	GHE-P- 8529 003A	
Italy, 1999 (Royal Gala)	SC 480	0.29	0.029	1000	3 4	30 0 1 3 7 14	0.03 0.23 0.18 0.15 0.11 0.02	<0.01 0.04 0.03 0.04 0.02 0.01	<0.01 0.01 0.01 0.01 0.01 0.01	<0.01 0.01 0.01 0.01 0.01 0.01	0.04 0.28 0.22 0.20 0.14 0.03	GHE-P- 8529 003B	
Japan, 1995 laboratory IET	SC 200		0.010	6000	3	3 7 14 21	0.01 0.01 0.01 0.01	<0.01 0.01 0.01 0.01	<0.01 0.01 0.01 0.01	<0.01 0.01 0.01 0.01	<0.01 0.01 0.01 0.01	0.01 0.01 0.01 0.01	GHF-P- 1489 Nagano
Japan, 1995 laboratory JCAC						3 7 14 21	<u>0.03</u> 0.01 0.01 0.01	<0.01 0.01 0.01 0.01	<0.01 0.01 0.01 0.01	<0.01 0.01 0.01 0.01	<0.01 0.01 0.01 0.01	0.03 0.01 0.01 0.01	GHF-P- 1489 Nagano
Japan, 1995 laboratory IET <sup>1</sup>	SC 200		0.010	6000	3	3 7 14 21	0.12 0.05 0.02 0.01	0.02 0.01 0.01 0.01	<0.01 0.01 0.01 0.01	<0.01 0.01 0.01 0.01	<0.01 0.01 0.01 0.01	0.14 0.06 0.02 0.01	GHF-P- 1489 Miyagi
Japan, 1995 laboratory JCAC <sup>2</sup>						3 7 14 21	<u>0.15</u> 0.08 0.03 0.01	<u>0.02</u> 0.01 0.01 0.01	<0.01 0.01 0.01 0.01	<0.01 0.01 0.01 0.01	<0.01 0.01 0.01 0.01	0.17 0.08 0.03 0.01	GHF-P- 1489 Miyagi
Spain, 1998 (Golden 972)	SC 480	0.29	0.029	1010	4	7 14	0.08 0.11	0.06 0.10	<0.01 0.01	<0.01 0.01	<0.01 0.01	0.15 0.21	GHE-P- 8250
Spain, 1998 (Golden)	SC 480	0.29	0.025	1200	3 4	48 0 1 3 7 14	<0.01 0.07 0.05 0.08 0.08 0.01	<0.01 0.05 0.04 0.06 0.06 0.01	<0.01 0.01 0.01 0.01 0.01 0.01	<0.01 0.01 0.01 0.01 0.01 0.01	<0.01 0.12 0.09 0.14 0.14 0.02	GHE-P- 8251	
Spain, 1999 (Golden Smothee)	SC 480	0.33 +0.34 +0.28 +0.29	0.030	1080 +1100 +930 +950	4	7 14	0.05 0.01	0.01 0.01	<0.01 0.01	<0.01 0.01	<0.01 0.01	0.07 0.02	GHE-P- 8530



Country, year, (variety)	Application					PHI, days	Spinosyn residues, mg/kg						Ref.
	Form	kg ai/ha	kg ai/hl	water, l/ha	no.		A	D	K	B	B of D	Total	
USA (CA), 1995 (Gala)	WG 820	0.050 +0.070 +2x0.10 +0.18		470	5	0 1 7 14	0.054 0.042 <u>&lt;0.01</u> 0.01	<0.01 <0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01	0.059 0.046 <0.01 <0.01	RES950 14	
USA (CA), 1995 (Gala)	WG 820	0.050 +0.070 +2x0.10 +0.18		1870	5	0 1 7 14	0.092 0.064 <u>0.024</u> 0.01	0.01 0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01	0.10 0.072 0.026 0.01	RES950 14	
USA (CA), 1995 (Granny Smith)	WG 820	0.050 +0.070 +2x0.10 +0.18		470	5	0 7 14	0.15 <u>0.045</u> 0.055	0.017 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	0.18 0.051 0.062	RES950 14	
USA (CA), 1995 (Granny Smith)	WG 820	0.050 +0.070 +2x0.10 +0.18		1870	5	0 7 14	0.071 <u>0.041</u> 0.035	0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	0.081 0.046 0.040	RES950 14	
USA (ID), 1995 (Red Delicious)	WG 820	0.050 +0.070 +2x0.10 +0.18		470	5	0 7	0.13 <u>0.079</u>	0.01 <u>0.01</u>	<0.01 <0.01	<0.01 <0.01	0.15 0.092	RES950 14	
USA (ID), 1995 (Red Delicious)	WG 820	0.050 +0.070 +2x0.10 +0.18		1870	5	0 7	0.12 <u>0.077</u>	0.014 <u>0.01</u>	<0.01 <0.01	<0.01 <0.01	0.13 0.090	RES950 14	
USA (IL), 1995 (Jonathan)	WG 820	0.050 +0.070 +2x0.10 +0.18		470	5	0 7	0.19 <u>0.024</u>	0.026 <0.01	<0.01 <0.01	<0.01 <0.01	0.22 0.024	RES950 14	
USA (IL), 1995 (Jonathan)	WG 820	0.050 +0.070 +2x0.10 +0.18		1870	5	0 7	0.10 <u>0.016</u>	0.018 <0.01	<0.01 <0.01	<0.01 <0.01	0.12 0.016	RES950 14	
USA (IN), 1995 (Golden Delicious)	WG 820	0.050 +0.070 +2x0.10 +0.18		470	5	0 7	0.058 <u>&lt;0.01</u>	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	0.064 <0.01	RES950 14	
USA (IN), 1995 (Golden Delicious)	WG 820	0.050 +0.070 +2x0.10 +0.18		1870	5	0 7	0.074 <u>&lt;0.01</u>	0.01 <0.01	<0.01 <0.01	<0.01 <0.01	0.086 <0.01	RES950 14	
USA (MI), 1995 (Empire)	WG 820	0.050 +0.070 +2x0.10 +0.18		470	5	0 7	0.10 <u>&lt;0.01</u>	0.01 <0.01	<0.01 <0.01	<0.01 <0.01	0.12 <0.01	RES950 14	

Country, year, (variety)	Application					PHI, days	Spinosyn residues, mg/kg						Ref.
	Form	kg ai/ha	kg ai/hl	water, l/ha	no.		A	D	K	B	B of D	Total	
USA (MI), 1995 (Empire)	WG 820	0.050 +0.070 +2x0.10 +0.18		1870	5	0 7	0.068 <u>0.015</u>	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	0.078 0.015	RES950 14
USA (MI), 1995 (Golden Delicious)	WG 820	0.050 +0.070 +2x0.10 +0.18		470	5	0 7	0.15 <u>0.01</u>	0.019 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	0.17 0.01	RES950 14
USA (MI), 1995 (Golden Delicious)	WG 820	0.050 +0.070 +2x0.10 +0.18		1870	5	0 7	0.21 <u>0.024</u>	0.026 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	0.24 0.029	RES950 14
USA (NC), 1995 (Winesap)	WG 820	0.050 +0.070 +2x0.10 +0.18		470	5	0 7 14	0.13 <u>0.017</u> 0.016	0.014 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	0.15 0.017 0.016	RES950 14
USA (NC), 1995 (Winesap)	WG 820	0.050 +0.070 +2x0.10 +0.18		1870	5	0 7 14	0.079 <u>0.032</u> 0.014	0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	0.096 0.037 0.014	RES950 14
USA (NY), 1995 (Golden Delicious)	WG 820	0.050 +0.070 +2x0.10 +0.18		470	5	0 3 7 10 14	0.10 0.019 <u>0.014</u> <0.01 <0.01	0.013 <0.01 <0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01 <0.01	0.11 0.020 0.014 <0.01 <0.01	RES950 14
USA (NY), 1995 (Golden Delicious)	WG 820	0.050 +0.070 +2x0.10 +0.18		1870	5	0 3 7 10 14	0.079 0.023 <u>0.01</u> <0.01 <0.01	0.01 <0.01 <0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01 <0.01	0.095 0.025 0.01 <0.01 <0.01	RES950 14
USA (NY), 1995 (McIntosh)	WG 820	0.050 +0.070 +2x0.10 +0.18		470	5	0 7	0.041 <u>&lt;0.01</u>	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	0.046 <0.01	RES950 14
USA (NY), 1995 (McIntosh)	WG 820	0.050 +0.070 +2x0.10 +0.18		1870	5	0 7	0.050 <u>&lt;0.01</u>	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	0.056 <0.01	RES950 14
USA (OR), 1995 (Red Delicious)	WG 820	0.050 +0.070 +2x0.10 +0.18		470	5	0 7	0.061 <u>0.025</u>	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	0.068 0.029	RES950 14
USA (OR), 1995 (Red Delicious)	WG 820	0.050 +0.070 +2x0.10 +0.18		1870	5	0 7	0.090 <u>0.028</u>	0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	0.10 0.033	RES950 14

Country, year, (variety)	Application					PHI, days	Spinosyn residues, mg/kg						Ref.
	Form	kg ai/ha	kg ai/hl	water, l/ha	no.		A	D	K	B	B of D	Total	
USA (OR), 1995 (Red Delicious)	WG 820	0.050 +0.070 +2x0.10 +0.18		470	5	0 7	0.052 <u>0.015</u>	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	0.059 0.015	RES950 14
USA (OR), 1995 (Red Delicious)	WG 820	0.050 +0.070 +2x0.10 +0.18		1870	5	0 7	0.064 <u>0.020</u>	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	0.072 0.020	RES950 14
USA (PA), 1995 (Red Delicious)	WG 820	0.050 +0.070 +2x0.10 +0.18		470	5	0 7	0.063 <u>&lt;0.01</u>	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	0.071 <0.01	RES950 14
USA (PA), 1995 (Red Delicious)	WG 820	0.050 +0.070 +2x0.10 +0.18		1870	5	0 7	0.095 <u>&lt;0.01</u>	0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	0.11 <0.01	RES950 14
USA (VA), 1995 (Red Yorking)	WG 820	0.050 +0.070 +2x0.10 +0.18		470	5	0 7	0.049 <u>0.01</u>	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	0.055 0.01	RES950 14
USA (VA), 1995 (Red Yorking)	WG 820	0.050 +0.070 +2x0.10 +0.18		1870	5	0 7	0.075 <u>0.01</u>	0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	0.084 0.01	RES950 14
USA (WA), 1995 (Basin Beauty)	WG 820	0.050 +0.070 +2x0.10 +0.18		470	5	0 3 7 10 14	0.087 0.058 <u>0.033</u> 0.032 0.032	0.01 <0.01 <0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01 <0.01	0.098 0.064 0.040 0.037 0.036	RES950 14
USA (WA), 1995 (Basin Beauty)	WG 820	0.050 +0.070 +2x0.10 +0.18		1870	5	0 3 7 10 14	0.077 0.069 <u>0.036</u> 0.012 0.018	0.01 0.01 <0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01 <0.01	0.087 0.077 0.040 0.012 0.020	RES950 14
USA (WA), 1995 (Red Delicious)	WG 820	0.050 +0.070 +2x0.10 +0.18		470	5	0 7	0.046 <u>0.041</u>	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	0.051 0.045	RES950 14
USA (WA), 1995 (Red Delicious)	WG 820	0.050 +0.070 +2x0.10 +0.18		1870	5	0 7	0.063 <u>0.033</u>	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	0.071 0.037	RES950 14

<sup>1</sup> IET: Institute of Environmental Toxicology<sup>2</sup> JCAC: Japan Chemical Analysis Consultant

Table 37. Spinosad residues in stone fruit from supervised trials at 19 sites in the USA. Double-underlined residues are from treatments according to GAP and are valid for the estimation of maximum residue levels.

Crop, location, year	Application					PHI, days	Residues, mg/kg spinosad	Ref.
	Form	kg ai/ha	kg ai/hl	water, l/ha	no.			
Cherry, CA, 1997	SC 240	0.07 +0.13 2×0.15		1900	4	7	<u>0.083</u>	RES97004
Cherry, CA, 1997	SC 240	0.07 +0.13 2×0.15		470	4	7	<u>0.03</u>	RES97004
Cherry, CA, 1997	SC 240	0.07 +0.13 2×0.15		1900	4	7	<u>0.060</u>	RES97004
Cherry, MI, 1997	SC 240	0.07 +0.13 2×0.15		1900	4	7	<u>0.040</u>	RES97004
Cherry, NY, 1997	SC 240	0.07 +0.13 2×0.15		1900	4	7	<u>0.023</u>	RES97004
Cherry, UT, 1997	SC 240	0.07 +0.13 2×0.15		1900	4	7	<u>0.11</u>	RES97004
Cherry, WA, 1997	SC 240	0.07 +0.13 2×0.15		1900	4	7	<u>0.11</u>	RES97004
Cherry, WI, 1997	SC 240	0.07 +0.13 2×0.15		1900	4	7	< <u>0.02</u>	RES97004
Peach, CA, 1997	SC 240	0.07 +0.13 2×0.15		1900	4	14	<u>0.055</u>	RES97004
Peach, CA, 1997	SC 240	0.07 +0.13 2×0.15		470	4	14	<u>0.055</u>	RES97004
Peach, CA, 1997	SC 240	0.07 +0.13 2×0.15		1900	4	14	<u>0.050</u>	RES97004
Peach, MI, 1997	SC 240	0.07 +0.13 2×0.15		1900	4	14	< <u>0.02</u>	RES97004
Peach, NC, 1997	SC 240	0.07 +0.13 2×0.15		1900	4	14	< <u>0.02</u>	RES97004

Crop, location, year	Application					PHI, days	Residues, mg/kg spinosad	Ref.
	Form	kg ai/ha	kg ai/hl	water, l/ha	no.			
Peach, PA, 1997	SC 240	0.07 +0.13 2×0.15		1900	4	14	< <u>0.02</u>	RES97004
Peach, SC, 1997	SC 240	0.07 +0.13 2×0.15		1900	4	14	<u>0.03</u>	RES97004
Plum, CA, 1997	SC 240	0.07 +0.13 2×0.15		1900	4	7	< <u>0.02</u>	RES97004
Plum, CA, 1997	SC 240	0.07 +0.13 2×0.15		470	4	7	< <u>0.02</u>	RES97004
Plum, CA, 1997	SC 240	0.07 +0.13 2×0.15		1900	4	7	< <u>0.02</u>	RES97004
Plum, CA, 1997	SC 240	0.07 +0.13 2×0.15		1900	4	7	< <u>0.02</u>	RES97004
Plum, MI, 1997	SC 240	0.07 +0.13 2×0.15		1900	4	7	< <u>0.02</u>	RES97004
Prune, dried, CA, 1997	SC 240	0.25 +0.46 2×0.53		1900	4	7	0.055	RES97004
Prune, dried, CA, 1997	SC 240	0.25 +0.46 2×0.53		1900	4	7	0.04	RES97004
Prune, fresh, CA, 1997	SC 240	0.25 +0.46 2×0.53		1900	4	7	0.065	RES97004
Prune, fresh, CA, 1997	SC 240	0.25 +0.46 2×0.53		1900	4	7	0.060	RES97004

Table 38. Spinosad residues in stone fruit from supervised trials in Chile and Japan.

Country, year (variety)	Application					PHI, days	Residues, mg/kg <sup>1, 2</sup>		Ref.
	Form	kg ai/ha	kg ai/hl	water, l/ha	no.		spinosyn A	spinosyn D	
Chile, 1999 nectarine (August red)	SC 480	0.17		2300	1	0	0.07 0.04 0.06	0.02 0.01 0.02	GHB-P 425
						1	<0.01 (2) 0.10	<0.01 (2) 0.02	
						4	0.03 0.04 0.04	<0.01(3)	
						7	<0.01(3)	<0.01 (2) 0.01	
						11	0.03 0.02 0.02	0.01 0.01 0.01	
						14	0.02 0.02 0.01	0.01 0.01 <0.01	
						1	c <0.01(3)	c 0.02 0.02 0.02	
						11	c <0.01 (2) 0.02	c <0.01(3)	
						14	c <0.01(3)	c <0.01 (2) 0.02	
Chile, 1999 nectarine (August red)	SC 480	0.33		2300	1	0	0.10 0.12 0.09	0.03 0.04 0.04	GHB-P 425
						1	0.23 0.14 0.11	0.06 0.04 0.04	
						4	<0.01 0.01 0.03	<0.01(3)	
						7	0.01 0.02 0.02	<0.01(3)	
						11	0.09 0.06 0.07	0.02 0.02 0.02	
						14	0.02 0.02 0.02	<0.01(3)	
						1	c <0.01(3)	c 0.02 0.02 0.02	
						11	c <0.01 (2) 0.02	c <0.01(3)	
						14	c <0.01(3)	c <0.01 (2) 0.02	
Chile, 1999 nectarine (September red)	SC 480	0.14		2000	1	0	0.05 0.02 0.03	0.01 <0.01 0.01	GHB-P 425
						6	0.20 0.03 0.10	0.06 <0.01 0.02	
						10	0.05 0.13 0.04	0.01 0.04 0.01	
						15	0.02 0.01 0.04	<0.01(3)	
						21	0.01 0.02 0.08	<0.01 (2) 0.01	
						21	c <0.01 (2) 0.04	c <0.01 (2) 0.02	
Chile, 1999 nectarine (September red)	SC 480	0.29		2000	1	0	0.49 0.28 0.37	0.12 0.07 0.09	GHB-P 425
						6	0.08 0.16 0.27	0.02 0.04 0.06	
						10	0.12 0.10 0.08	0.04 0.02 0.02	
						15	0.02 0.03 <0.01	<0.01 (2) 0.02	
						21	0.01 <0.01 (2)	0.01 <0.01 (2)	
						21	c <0.01 (2) 0.04	c <0.01 (2) 0.02	
Chile, 1999 nectarine (Sunrice late)	SC 480	0.14		2200	1	0	0.07 0.04	<0.01 (2)	GHB-P 425
						1	<0.01 0.01 0.01	<0.01(3)	
						3	0.03 0.04 <0.01	<0.01(3)	
						7	<0.01 0.02 0.04	<0.01(3)	
						11	<0.01(3)	<0.01(3)	
						14	<0.01(3)	<0.01(3)	
						7	c 0.02 <0.01 (2)	c <0.01(3)	
						14	c <0.01(3)	c 0.02 <0.01 (2)	
Chile, 1999 nectarine (Sunrice late)	SC 480	0.29		2200	1	0	0.05 0.04 0.05	<0.01(3)	GHB-P 425
						1	0.03 0.02 0.03	<0.01(3)	
						3	0.05 <0.01 0.05	<0.01(3)	
						7	0.01 <0.01 (2)	<0.01(3)	
						11	0.02 0.03 <0.01	<0.01(3)	
						14	0.02 <0.01 (2)	<0.01(3)	
						7	c 0.02 <0.01 (2)	c <0.01(3)	
						14	c <0.01(3)	c 0.02 <0.01 (2)	

Country, year (variety)	Application					PHI, days	Residues, mg/kg <sup>1, 2</sup>		Ref.
	Form	kg ai/ha	kg ai/hl	water, l/ha	no.		spinosyn A	spinosyn D	
Japan, 1995 peach	SC 200	0.50	0.01	5000	2	3	p 0.38 f <0.01	p 0.04 f <0.01	GHB P-331
						7	p 0.08 f <0.01	p 0.01 f <0.01	
						14	p 0.03 f <0.01	p <0.01 f <0.01	
						21	p 0.02 f <0.01	p <0.01 f <0.01	
					3	3	p 0.06 f <0.01	p <0.01 f <0.01	

<sup>1</sup> p: peel, f: flesh<sup>2</sup> c: samples from control plot

Table 39. Spinosad residues in grapes from supervised trials in France, Italy and Spain.

Country, year (variety)	Application <sup>3</sup>					PHI, days	Spinosyn residues, mg/kg				Ref.
	Form	kg ai/ha	kg ai/hl	water, l/ha	no.		A HPLC	D HPLC	Total <sup>1</sup> HPLC	IA <sup>2</sup>	
France, 1997 (Chenin, Riparia Gloire)	SC 480	0.060	0.029	210	4 5	16	<0.01	<0.01	<0.01	<0.01	GHE-P-7575
						0	0.03	<0.01	0.05	0.08	
						5	0.01	<0.01	0.01	0.02	
						10	<0.01	<0.01	<0.01	<0.01	
						14	<0.01	<0.01	<0.01	<0.01	
France, 1997 (Gamay)	SC 480	0.060	0.032	190	4 5	17	<0.01	<0.01	<0.01	<0.01	GHE-P-7575
						0	0.08	0.02	0.10	0.09	
						5	0.02	<0.01	0.03	0.04	
						10	0.02	<0.01	0.03	0.02	
						15	0.01	<0.01	0.02	0.02	
France, 1997 (Negrette)	SC 480	0.060	0.024	260	5	15 <sup>6</sup>	<0.01	<0.01	0.01	<0.01	GHE-P-7577
France, 1998 (Cabernet Franc)	SC 480	2×0.096 +3×0.048	2×0.036 +3×0.019	260	5	15	<0.01	<0.01	0.02		GHE-P-7853
France, 1998 (Chenin)	SC 480	2×0.096 +3×0.048	2×0.036 +3×0.019	260	5	15	<0.01	<0.01	<0.01		GHE-P-7850
						0	0.03	0.02	0.05		
						5	<0.01	<0.01	0.02		
						10	<0.01	<0.01	0.02		
						16	<0.01	<0.01	<0.01		
France, 1998 (Gamay)	SC 480	2×0.096 +3×0.048	2×0.042 +3×0.021	230	4 5	15	0.02	0.02	0.04		GHE-P-7851
						0	0.04	0.03	0.07		
						6	0.03	0.02	0.05		
						10	0.03	0.02	0.05		
						15	0.01	<0.01	0.02		
France, 1998 (Red wine grape, Cot)	SC 480	0.096 +0.096 +0.048 +0.048 +0.048	0.042 +0.042 +0.021 +0.021 +0.021	230	5	15	0.02	0.01	0.03		GHE-P-7856

Country, year (variety)	Application <sup>3</sup>					PHI, days	Spinosyn residues, mg/kg				Ref.
	Form	kg ai/ha	kg ai/hl	water, l/ha	no.		A HPLC	D HPLC	Total <sup>1</sup> HPLC	IA <sup>2</sup>	
Italy, 1997 (Tocai Verde)	SC 480	0.060	0.015	400	4 5	22	0.01	<0.01	0.02	0.02	GHE-P-7579
						0	0.18	0.04	0.24	0.45	
						5	0.07	0.02	0.09	0.15	
						10	0.04	0.01	0.06	0.08	
						16	0.03	0.01	0.04 <sup>4</sup>	0.05 <sup>4</sup>	
Italy, 1998 (Italia)	SC 480	0.099 +0.095 +0.041 +0.049 +0.048	0.014 +0.014 +0.0060 +0.0060 +0.0060	720 +690 +590 +710 +700	4 5	18	0.02	0.03	0.05		GHE-P-7852
						0	0.18	0.14	0.32		
						5	0.10	0.08	0.18		
						9	0.06	0.05	0.11		
						14	0.03	0.03	0.06		
Italy, 1998 (Trebiano)	SC 480	0.098 +0.091 +0.049 +0.046 +0.050	0.016 +0.016 +0.005 +0.005 +0.005	610 +570 +1010 +950 +1030	5	15	0.01	0.01	0.03		GHE-P-7855
Spain, 1997 (Italia Moscatel)	SC 480	0.060	2×0.0075 +3×0.0060	2×800 +3×995	5	15 <sup>7</sup>	0.11	0.03	0.15	0.17	GHE-P-7576
Spain, 1997 (Italia)	SC 480	0.060	0.0075 +0.0075 +0.0060 +0.0060 +0.0060	780 +770 +960 +950 +1010	4 5	17	0.08	0.02	0.11	0.12	GHE-P-7578
						0	0.23	0.06	0.30	0.40	
						5	0.14	0.05	0.19	0.24	
						10	0.12	0.03	0.16	0.17	
						15	0.07	0.02	0.09 <sup>4</sup>	0.12 <sup>4</sup>	
Spain, 1998 (Cencibel)	SC 480	2×0.096 +3×0.048	0.019 +0.016 +0.0069 +2×0.006	500 +590 +700 +790 +795	5	15	0.04 c 0.01 <sup>5</sup>	0.03 c <0.01	0.07 c 0.02		GHE-P-7854

<sup>1</sup> Total includes spinosyns A, D, B, N-demethyl-D and K

<sup>2</sup> Immunoassay

<sup>3</sup> Application rate, spray concentration and volume per ha were not identical for each application (variation generally within 20%). Single values are those for the last application.

<sup>4</sup> Samples analysed by IA method Nov-Dec 97 and by HPLC method Jan-Feb 99

<sup>5</sup> c: sample from control plot

<sup>6</sup> Samples in freezer storage approx. 17 months.

<sup>7</sup> Samples in freezer storage approx. 18 months.

Table 40. Spinosad residues in grapes from supervised trials in Chile.

Year (variety)	Application					PHI, days	Residues, mg/kg			Ref.			
	Form	kg ai/ha	kg ai/hl	water, l/ha	no.		spinosyn A		spinosyn D				
1998 (Red Globe) <sup>1</sup>	SC 480	0.13		1800	2	0	1.4	1.5	1.5	0.50	0.61	0.61	GHB-P 419
						18	0.02	0.02	0.01	<0.01	0.01	<0.01	
						38	<0.01(3)		<0.01(3)				
						66	<0.01(3)		<0.01(3)				
						86	<0.01(3)		<0.01(3)				



Year (variety)	Application					PHI, days	Residues, mg/kg				Ref.		
	Form	kg ai/ha	kg ai/hl	water, l/ha	no.		spinosyn A		spinosyn D				
1998 (Red Globe) <sup>1</sup>	SC 480	0.26		1800	2	0	3.2	3.1	3.9	1.3	1.2	1.4	GHB-P 419
						18	0.06	0.02	0.04	0.03	<0.01	0.02	
						38	<0.01	(3)		<0.01	(3)		
						66	<0.01	(3)		<0.01	(3)		
						86	<0.01	(3)		<0.01	(3)		

<sup>1</sup> Applications made when flowering commenced and 7 days later at full bloom. Grapes sampled shortly after flowering are unrelated to proper harvest.

Table 41. Spinosad residues in strawberries from supervised trials in Belgium, Greece, Italy, Spain and the UK.

Country, year, (variety)	Application					PHI, days	Spinosyn residues, mg/kg						Ref.
	Form	kg ai/ha	kg ai/hl	water, l/ha	no. <sup>3</sup>		A	D	K	B	B of D	Total	
Belgium, 1998 (Elsanta) <sup>1</sup>	SC 480	0.074 +0.076 +0.081		760 +720 +800	g 2 g 3	14	0.01	<0.01	<0.01	<0.01	<0.01	0.02	GHE-P- 8056 Ittre
						0	0.05	0.04	<0.01	<0.01	<0.01	0.09	
						1	0.04	0.03	<0.01	<0.01	<0.01	0.07	
						2	0.03	0.02	<0.01	<0.01	<0.01	0.04	
Belgium, 1998 (Elsanta) <sup>1</sup>	SC 480	0.077		800 +790 +860	g 2 g 3	14	0.01	<0.01	<0.01	<0.01	<0.01	0.02	GHE-P- 8056 Wepion
						0	0.05	0.04	<0.01	<0.01	<0.01	0.09	
						1	0.04	0.03	<0.01	<0.01	<0.01	0.07	
						2	0.03	0.03	<0.01	<0.01	<0.01	0.05	
Greece, 1998 (Tulda)	SC 480	0.081	0.010	800	2 3	8	0.01	<0.01	<0.01	<0.01	<0.01	0.02	GHE-P- 8053
						0	0.03	0.02	<0.01	<0.01	<0.01	0.05	
						1	0.02	0.02	<0.01	<0.01	<0.01	0.04	
						2	0.02	0.02	<0.01	<0.01	<0.01	0.04	
Italy, 1999 (Camarosa)	SC 480	0.080 +0.079 +0.081	0.013	590 +590 +600	2 3	7	0.01	<0.01	<0.01	<0.01	<0.01	0.01	GHE-P- 8095 Catania
						0	0.09	0.02	<0.01	<0.01	<0.01	0.11	
						1	0.07	0.01	<0.01	<0.01	<0.01	0.09	
						2	0.07	0.01	<0.01	<0.01	<0.01	0.09	
Italy, 1999 (Camarosa)	SC 480	0.079 +0.078 +0.079	0.011	700 +680 +700	3	1	0.06	0.01	<0.01	<0.01	<0.01	0.08	GHE-P- 8097
						2	0.07	0.01	<0.01	<0.01	<0.01	0.09	
Italy, 1999 (Camarosa)	SC 480	0.081	0.013	600	3	1	0.12	0.03	<0.01	<0.01	<0.01	0.15	GHE-P- 8098
						2	0.09	0.02	<0.01	<0.01	<0.01	0.10	
Italy, 1999 (Eddie)	SC 480	0.57 +0.079 +0.077	0.071 +0.010 +0.010	810	3	1	0.03	0.02	<0.01	<0.01	<0.01	0.05	GHE-P- 8054
						2	0.02	0.02	<0.01	<0.01	<0.01	0.04	
Italy, 1999 (Marmolada)	SC 480	0.081 +0.076 +0.078	0.013	630 +600 +610	2 3	16	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	GHE-P- 8095 Forli
						0	0.10	0.02	<0.01	<0.01	<0.01	0.12	
						1	0.04	0.01	<0.01	<0.01	<0.01	0.05	
						2	0.01	<0.01	<0.01	<0.01	<0.01	0.02	
Italy, 1999 (Pajaro)	SC 480	0.077 +0.079 +0.079	0.011	670 +700 +700	g 3	1	0.19	0.04	<0.01	<0.01	<0.01	0.24	GHE-P- 7617
						2	0.12	0.02	<0.01	<0.01	<0.01	0.14	

Country, year, (variety)	Application					PHI, days	Spinosyn residues, mg/kg						Ref.
	Form	kg ai/ha	kg ai/hl	water, l/ha	no. <sup>3</sup>		A	D	K	B	B of D	Total	
Spain, 1998 (Camarosa) <sup>2</sup>	SC 480	0.54	0.054	1000	3	3 6	1.4 0.66	0.34 0.16	0.02 0.01	0.04 0.02	<0.01 <0.01	1.8 0.86	GHE-P- 8055
Spain, 1998 (Camarosa) <sup>2</sup>	SC 480	0.54 +0.54 +0.52	0.054	1000 +1000 +960	g 3	3 6	1.0 0.72	0.25 0.18	0.02 0.01	0.03 0.02	<0.01 <0.01	1.4 0.94	GHE-P- 8057
Spain, 1999 (Camarosa)	SC 480	0.069 +0.075 +0.075	0.0093	740 +800 +810	pt 2 pt 3	12	0.03	<0.01	<0.01	<0.01	<0.01	0.04	GHE-P- 6561
						0	0.10	0.03	<0.01	<0.01	<0.01	0.12	
						1	0.09	0.02	<0.01	<0.01	<0.01	0.11	
						2	0.09	0.02	<0.01	<0.01	<0.01	0.10	
Spain, 1999 (Camarosa)	SC 480	0.076	0.0093	760 +810 +820	g 3	1 2	0.07 0.05	0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	0.09 0.06	GHE-P- 6582
Spain, 1999 (Camarosa)	SC 480	0.073 +0.075 +0.076	0.0093	790 +810 +820	2 3	12	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	GHE-P- 7787
						0	0.09	0.02	<0.01	<0.01	<0.01	0.12	
						1	0.04	<0.01	<0.01	<0.01	<0.01	0.05	
						2	0.03	<0.01	<0.01	<0.01	<0.01	0.03	
Spain, 1999 (Camarosa)	SC 480	0.076	0.0093	760 +820 +810	3	1 2	0.09 0.02	0.02 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	0.11 0.03	GHE-P- 8096
UK, 1999 (Elsanta)	SC 480	0.080	0.013	590	g 2 g 3	11	0.02	<0.01	<0.01	<0.01	<0.01	0.03	GHE-P- 6562 11A
						0	0.06	0.01	<0.01	<0.01	<0.01	0.07	
						1	0.06	0.01	<0.01	<0.01	<0.01	0.07	
						2	0.07	0.01	<0.01	<0.01	<0.01	0.08	
UK, 1999 (Symphony)	SC 480	0.081 +0.080 +0.083	0.013	600 +600 +620	g 3	1	0.06	0.02	<0.01	<0.01	<0.01	0.07	GHE-P- 6562 11B
						2	0.06	0.01	<0.01	<0.01	<0.01	0.07	

<sup>1</sup> Samples stored in a freezer for approx. 16 months<sup>2</sup> Samples stored in a freezer for approx. 18 months<sup>3</sup> g glasshouse. pt: plastic tunnel

Table 42. Spinosad residues in blueberries from supervised trials in the USA, 1998.

Location, year	Application, kg ai/ha	PHI, days	Spinosad, mg/kg	Ref.
ME	1.0	1	0.066 0.015	06850.98-ME02
MI	1.0	1	0.19 0.10	06850.98-MI32
MI	1.0	1	0.17 0.15	06850.98-MI33
MI	1.0	1	0.032 0.037	06850.98-MI34
NC	1.0	1	0.085 0.082	06850.98-NC15
NC	1.0	1	0.075 0.065	06850.98-NC16
OR	1.0	1	0.18 0.17	06850.98-OR14

Location, year	Application, kg ai/ha	PHI, days	Spinosad, mg/kg	Ref.
PA	1.0	1	0.13 0.084	06850.98-PA04

Table 43. Spinosad residues in kiwifruit from supervised trials in New Zealand. Double-underlined residues are from treatments according to GAP and are valid for the estimation of maximum residue levels.

Year (variety)	Application					PHI, days	Spinosyn residues, mg/kg				Ref.
	Form	kg ai/ha	kg ai/hl	water, l/ha	no.		A HPLC	D HPLC	Total HPLC	IA	
1996	SC480	0.038		1500	8	0 3 7 14 45				0.22 0.13 0.08 0.09 0.03	GHF-P 1551
1996	SC480	0.075		1500	8	0 3 7 14 45				0.54 0.32 0.15 0.17 0.12	GHF-P 1551
1996	SC480	0.15		1500	8	0 3 7 14 45				0.78 0.54 0.27 0.36 0.34	GHF-P 1551
1996	SC480	0.30		1500	8	0 3 7 14 45				1.2 0.66 0.30 0.30 0.16	GHF-P 1551
1998 (Hayward)	SC120		0.0048		3	0 7 14 28				0.14 <0.05 0.06 <0.05	GHF-P 1798
1998 (Hayward)	SC120		0.0096		3	0 7 14 28				0.20 0.14 0.10 <0.05	GHF-P 1798
1998 (Hayward)	SC120		0.0048		3	134 156				<0.05 (2) <0.05 (2)	GHF-P 1798
1998 (Hayward)	SC120		0.0096		3	134 156				<0.05 0.11 0.07 0.08	GHF-P 1798
1998 (Hayward)	SC120		0.0048	2000	2	142				<0.05	GHF-P 1799
1998 (Hayward)	SC120		0.0096	2000	2	142				<0.05	GHF-P 1799

Year (variety)	Application					PHI, days	Spinosyn residues, mg/kg				Ref.
	Form	kg ai/ha	kg ai/hl	water, l/ha	no.		A HPLC	D HPLC	Total HPLC	IA	
1999 (Hayward)	SC120		0.0048	2000	2	142	<0.01	<0.01	<0.01		GHF-P 1958 trial 98493-01
						143	<0.01	<0.01	<0.01		
						145	<0.01	<0.01	<0.01		
						149	<0.01	<0.01	<0.01		
						156	<0.01	<0.01	<0.01		
						170	<0.01	<0.01	<0.01		
					3	0	0.30	0.06	0.37		
						1	0.13	0.03	0.17		
						3	0.11	0.02	0.14		
						7	0.09	0.02	0.12		
						14	0.09	0.01	0.11		
						28	0.07	0.01	0.08		
1999 (Hayward)	SC120		0.0096	2000	2	142	<0.01	<0.01	<0.01		GHF-P 1958 trial 98493-01
						143	<0.01	<0.01	<0.01		
						145	<0.01	<0.01	<0.01		
						149	<0.01	<0.01	<0.01		
						156	<0.01	<0.01	<0.01		
						170	<0.01	<0.01	<0.01		
					3	0	0.46	0.09	0.57		
						1	0.20	0.04	0.26		
						3	0.15	0.03	0.19		
						7	0.19	0.03	0.24		
						14	0.11	0.02	0.14		
						28	0.07	0.01	0.09		
1999 (Hayward)	SC120		0.0048	2000	2	142	<0.01	<0.01	<0.01		GHF-P 1958 trial 98493-02
						143	<0.01	<0.01	<0.01		
						145	<0.01	<0.01	<0.01		
						149	<0.01	<0.01	<0.01		
						156	<0.01	<0.01	<0.01		
						170	<0.01	<0.01	<0.01		
					3	0	0.23	0.04	0.28		
						1	0.08	0.02	0.11		
						3	0.04	<0.01	0.05		
						7	0.04	<0.01	0.05		
						14	0.04	<0.01	0.05		
						28	0.02	<0.01	0.02		
1999 (Hayward)	SC120		0.0096	2000	2	142	<0.01	<0.01	<0.01		GHF-P 1958 trial 98493-02
						143	<0.01	<0.01	<0.01		
						145	<0.01	<0.01	<0.01		
						149	<0.01	<0.01	<0.01		
						156	<0.01	<0.01	<0.01		
						170	<0.01	<0.01	<0.01		
					3	0	0.45	0.09	0.56		
						1	0.16	0.03	0.21		
						3	0.11	0.02	0.14		
						7	0.08	0.01	0.10		
						14	0.08	0.02	0.10		
						28	0.05	<0.01	0.06		

Year (variety)	Application					PHI, days	Spinosyn residues, mg/kg				Ref.
	Form	kg ai/ha	kg ai/hl	water, l/ha	no.		A HPLC	D HPLC	Total HPLC	IA	
1999 (Hayward)	SC120		0.0048	2000	2	128	<0.01	<0.01	<0.01		GHF-P 1958 trial 98493- 04
						129	<0.01	<0.01	<0.01		
						131	<0.01	<0.01	<0.01		
						135	<0.01	<0.01	<0.01		
						142	<0.01	<0.01	<0.01		
						156	<0.01	<0.01	<0.01		
					3	0	0.19	0.04	0.23		
						1	0.03	<0.01	0.04		
						3	0.03	<0.01	0.04		
						7	0.03	<0.01	0.03		
						14	0.02	<0.01	0.02		
						28	0.02	<0.01	0.02		
					3	128	<0.01	<0.01	<0.01		
						129	<0.01	<0.01	<0.01		
						131	<0.01	<0.01	<0.01		
						135	<0.01	<0.01	<0.01		
						142	<0.01	<0.01	<0.01		
						156	<0.01	<0.01	<0.01		
						0	0.33	0.06	0.41		
						1	0.09	0.02	0.12		
						3	0.04	<0.01	0.05		
						7	0.05	0.01	0.07		
						14	0.03	<0.01	0.04		
						28	0.02	<0.01	0.02		
					3	130	<0.01	<0.01	0.01		GHF-P 1958 trial 98493- 05
						131	<0.01	<0.01	<0.01		
						133	<0.01	<0.01	0.01		
						137	<0.01	<0.01	<0.01		
						144	<0.01	<0.01	<0.01		
						158	<0.01	<0.01	<0.01		
						0	0.34	0.06	0.42		
						1	0.11	0.02	0.14		
						3	0.10	0.02	0.12		
						7	0.04	<0.01	0.05		
						14	0.02	<0.01	0.02		
						28	<0.01	<0.01	0.02		
					3	130	0.12	0.02	0.15		GHF-P 1958 trial 98493- 05
						131	<0.01	<0.01	0.01		
						133	<0.01	<0.01	0.01		
						137	<0.01	<0.01	<0.01		
						144	<0.01	<0.01	<0.01		
						158	<0.01	<0.01	<0.01		
						0	<0.01	<0.01	0.01		
						1	0.24	0.05	0.30		
						3	0.19	0.03	0.24		
						7	0.10	0.02	0.13		
						14	0.06	0.01	0.08		
						28	0.05	<0.01	0.06		

Year (variety)	Application					PHI, days	Spinosyn residues, mg/kg				Ref.
	Form	kg ai/ha	kg ai/hl	water, l/ha	no.		A HPLC	D HPLC	Total HPLC	IA	
1999 (Hayward)	SC120		0.0048	2000	2	111	0.01	0.01	0.02		GHF-P 1958 trial 98493- 06
						112	<0.01	0.01	0.02		
						114	<0.01	0.01	0.02		
						118	<u>0.01</u>	<u>0.01</u>	0.02		
						125	<0.01	<0.01	0.01		
						139	<0.01	<0.01	0.01		
					3	0	0.33	0.07	0.40		
						1	0.23	0.05	0.29		
						3	0.20	0.05	0.24		
						7	0.17	0.04	0.22		
						14	0.09	0.02	0.11		
						28	0.03	<0.01	0.04		
1999 (Hayward)	SC120		0.0096	2000	2	111	0.01	0.01	0.02		GHF-P 1958 trial 98493- 06
						112	0.01	0.01	0.03		
						114	0.01	0.01	0.03		
						118	<0.01	<0.01	0.02		
						125	0.01	<.01	0.02		
						139	0.01	<0.01	0.02		
					3	0	0.82	0.15	0.98		
						1	0.57	0.11	0.70		
						3	0.42	0.08	0.51		
						7	0.27	0.06	0.34		
						14	0.19	0.03	0.24		
						28	0.08	0.01	0.10		

Table 44. Spinosad residues in Brassica vegetables from supervised trials in Australia and New Zealand. Double-underlined residues are from treatments according to GAP and are valid for the estimation of maximum residue levels.

Crop, country, (location), year (variety)	Application					PHI, days	Residues, mg/kg spinosad	Ref.
	Form	kg ai/ha	kg ai/hl	water, l/ha	no.			
BROCCOLI								
Australia (NSW), 1997	SC 120 +adjuvant	0.048			4	3 7	0.05 0.01	GHF-P 1585
Australia (NSW), 1997	SC 120 +adjuvant	0.096			4	3 7	<u>0.08</u> 0.01	GHF-P 1585
Australia (Vic), 1996	SC480	0.05		253	4	0 3 7 14 21	1.1 0.09 0.01 <0.01 <0.01	GHF-P 1539
Australia (Vic), 1996	SC480	0.10		253	4	0 3 7 14 21	2.3 <u>0.39</u> 0.03 <0.01 <0.01	GHF-P 1539

Crop, country, (location), year (variety)	Application					PHI, days	Residues, mg/kg spinosad	Ref.
	Form	kg ai/ha	kg ai/hl	water, l/ha	no.			
Australia (Vic), 1997 (Bushido)	SC 120	0.048		250	4	0 1 3 7 14	0.17 0.12 0.01 <0.01 <0.01	GHF-P 1586 96322.3
Australia (Vic), 1997 (Bushido)	SC 120	0.096		250	4	0 1 3 7 14	0.70 0.49 <u>0.06</u> 0.01 <0.01	GHF-P 1586 96322.3
CAULIFLOWER								
Australia (WA), 1996 (Freemont)	SC480	0.05		250	4	0 3 7 14 21	1.0 0.01 <0.01 <0.01 <0.01	GHF-P 1539
Australia (WA), 1996 (Freemont)	SC480	0.10		250	4	0 3 7 14 21	1.7 <u>0.02</u> <0.01 <0.01 <0.01	GHF-P 1539
NZ, 1997 (All Year Hybrid)	SC 120	0.048		500	5	0 1 3 7 14	0.03 0.02 <u>0.01</u> <0.01 <0.01	GHF-P 1570 211571
NZ, 1997 (All Year Hybrid)	SC 120	0.096		500	5	0 1 3 7 14	0.09 0.07 0.03 0.01 0.01	GHF-P 1570 211571
CHINESE CABBAGE								
Australia (Qld), 1996 (SPS Maltida)	SC 120 +adjuvant	0.048 +0.048 +0.072 +0.072		250 +250 +380 +760	4	3 7	<u>0.10</u> 0.01	GHF-P 1585
Australia (Qld), 1996 (SPS Maltida)	SC 120 +adjuvant	0.096 +0.096 +0.14 +0.14		250 +250 +380 +760	4	3 7	0.09 0.02	GHF-P 1585
CABBAGE								
Australia (Vic), 1997	SC 120	0.048		250	4	0 1 3 7 14	0.01 <0.01 <0.01 <0.01 <0.01	GHF-P 1586 96322.4

Table 45. Spinosad residues in cabbage and Chinese cabbage from supervised trials in Japan in 1995. Double-underlined residues are from treatments according to GAP and are valid for the estimation of maximum residue levels. Residues were analysed by two laboratories.

Crop, Laboratory	Application					PHI, days	Spinosyn residues, mg/kg						Ref.
	Form	kg ai/ha	kg ai/hl	water, l/ha	no.		A	D	K	B	B of D	Total	
CABBAGE													



Crop, Laboratory	Application					PHI, days	Spinosyn residues, mg/kg						Ref.
	Form	kg ai/ha	kg ai/hl	water, l/ha	no.		A	D	K	B	B of D	Total	
DowElanco Japan	WG 250		0.01	3000	3	3 7 14	<u>&lt;0.01</u> <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	GHF-P- 1486 Iwate
Institute of Environmental Toxicology						3 7 14	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	GHF-P- 1486 Iwate
DowElanco Japan	WG 250		0.01	3000	3	3 7 14	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	GHF-P- 1486 Gunma
Institute of Environmental Toxicology						3 7 14	<u>0.01</u> <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	0.01 <0.01 <0.01	GHF-P- 1486 Gunma
CHINESE CABBAGE													
DowElanco Japan	WG 250		0.01	3000	3	3 6 14	<u>0.08</u> <0.01 <0.01	<u>0.01</u> <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	0.09 <0.01 <0.01	GHF-P- 1487 Miyagi
Institute of Environmental Toxicology						3 6 14	0.02 0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	0.02 0.01 <0.01	GHF-P- 1487 Miyagi
DowElanco Japan	WG 250		0.01	3000	3	3 7 14	<u>0.08</u> 0.04 <0.01	<u>0.01</u> 0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	0.09 0.05 <0.01	GHF-P- 1487 Nagano
Institute of Environmental Toxicology						3 7 14	0.08 0.04 0.01	0.02 0.01 <0.01	0.01 0.01 0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	0.11 0.06 0.02	GHF-P- 1487 Nagano

Table 46. Spinosad residues in Brassica vegetables from supervised trials in the USA in 1995. Double-underlined residues are from treatments according to GAP and are valid for the estimation of maximum residue levels. All WG 820 formulations.

Crop, Location (variety)	Application				PHI, days	Spinosyn residues, mg/kg						Ref.
	kg ai/ha	kg ai/hl	water, l/ha	no.		A	D	K	B	B of D	Total	
BROCCOLI												
AZ (Marathon)	2x0.10 +2x0.15		280	4	1 3	<u>0.17</u> 0.097	<u>0.024</u> 0.01	<0.01 <0.01	0.02 <0.01	<0.01 <0.01	0.22 0.12	RES95001
CA (Greenbelt)	2x0.10 +2x0.15		280	4	1 3	<u>0.35</u> 0.19	<u>0.04</u> 0.02	<0.01 <0.01	0.01 0.01	<0.01 <0.01	0.40 0.23	RES95001 CA1
CA (Marathont)	2x0.10 +2x0.15		280	4	1 3	<u>0.14</u> 0.097	<u>0.02</u> 0.01	<0.01 <0.01	0.01 <0.01	<0.01 <0.01	0.18 0.12	RES95001 CA2

Crop, Location (variety)	Application				PHI, days	Spinosyn residues, mg/kg						Ref.
	kg ai/ha	kg ai/hl	water, l/ha	no.		A	D	K	B	B of D	Total	
CA (Sultan)	2x0.10 +2x0.15		280	4	0 1 3 5 7 10	<u>0.47</u> 0.42 0.28 0.21 0.14 0.089	<u>0.057</u> 0.05 0.03 0.03 0.02 0.01	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01	0.03 0.02 0.02 0.01 0.01 0.01	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01	0.56 0.50 0.34 0.25 0.17 0.11	RES95001 CA3
CA (Green Valiant)	2x0.10 +2x0.15		280	4	1 3	<u>0.32</u> 0.20	<u>0.04</u> 0.02	<0.01 <0.01	0.03 0.02	<0.01 <0.01	0.39 0.25	RES95001 CA4
CA (Marathon)	2x0.10 +2x0.15		280	4	1 3	<u>0.32</u> 0.19	<u>0.03</u> 0.02	<0.01 <0.01	0.02 0.01	<0.01 <0.01	0.37 0.22	RES95001 CA5
OR (GEM)	2x0.10 +2x0.15		280	4	1 3	<u>0.39</u> 0.35	<u>0.052</u> 0.049	<0.01 <0.01	0.04 0.03	<0.01 <0.01	0.50 0.44	RES95001
TX (Green Comet)	2x0.10 +2x0.15		280	4	1 3	<u>0.11</u> 0.02	<u>0.01</u> <0.01	<0.01 <0.01	0.01 <0.01	<0.01 <0.01	0.14 0.02	RES95001
CABBAGE												
CA (Charmant) include wrapper leaves	2x0.10 +2x0.15		290	4	1 3	<u>0.078</u> 0.03	<u>0.01</u> <0.01	<0.01 <0.01	<0.01 <0.01	0.01 <0.01	0.11 0.04	RES95001
CA (Charmant) wrapper leaves removed	2x0.10 +2x0.15		290	4	1 3	0.02 0.02	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	0.03 0.02	RES95001
FL (Savoy) include wrapper leaves	2x0.10 +2x0.15		280	4	1 3 5	<u>0.93</u> 0.04 0.03	<u>0.16</u> <0.01 <0.01	<0.01 <0.01 <0.01	0.03 <0.01 <0.01	<0.01 <0.01 <0.01	1.1 0.052 0.04	RES95001
FL (Savoy) wrapper leaves removed	2x0.10 +2x0.15		280	4	1 3 5	0.15 <0.01 <0.01	0.02 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	0.18 <0.01 <0.01	RES95001
IN (Stonehead) include wrapper leaves	2x0.10 +2x0.15		290	4	1 3	<u>0.01</u> <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	0.01 0.01	RES95001 IN1
IN (Stonehead) wrapper leaves removed	2x0.10 +2x0.15		290	4	1 3	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	RES95001 IN1
IN (Golden Acre) include wrapper leaves	2x0.10 +2x0.15		280	4	1 3	<u>0.053</u> 0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	0.064 0.01	RES95001 IN2
IN (Golden Acre) wrapper leaves removed	2x0.10 +2x0.15		280	4	1 3	0.02 0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	0.03 0.01	RES95001 IN2
PA (Charmant) include wrapper leaves	2x0.10 +2x0.15		320	4	1 3	<u>0.070</u> 0.02	<u>0.01</u> <0.01	<0.01 <0.01	0.01 <0.01	<0.01 <0.01	0.089 0.03	RES95001 PA1

Crop, Location (variety)	Application				PHI, days	Spinosyn residues, mg/kg						Ref.
	kg ai/ha	kg ai/hl	water, l/ha	no.		A	D	K	B	B of D	Total	
PA (Charmant) wrapper leaves removed	2x0.10 +2x0.15		320	4	1 3	0.01 0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	0.01 0.01	RES95001 PA1
PA (Bravo) include wrapper leaves	2x0.10 +2x0.15		330	4	1 3	<u>0.02</u> 0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	0.03 0.01	RES95001 PA2
PA (Bravo) wrapper leaves removed	2x0.10 +2x0.15		330	4	1 3	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	RES95001 PA2
TX (Early Cabbage) include wrapper leaves	2x0.10 +2x0.15		290	4	1 3 5	<u>0.80</u> 0.32 0.14	<u>0.15</u> 0.04 0.02	0.01 0.01 <0.01	0.077 0.04 0.02	0.01 <0.01 <0.01	1.0 0.41 0.20	RES95001
TX (Early Cabbage) wrapper leaves removed	2x0.10 +2x0.15		290	4	1 3 5	0.32 0.097 0.11	0.04 0.01 0.01	<0.01 <0.01 <0.01	0.03 0.01 0.01	<0.01 <0.01 <0.01	0.39 0.13 0.15	RES95001
VA (Market Prize) include wrapper leaves	2x0.10 +2x0.15		290	4	1 3	<u>0.33</u> 0.13	<u>0.04</u> 0.02	<0.01 <0.01	0.03 <0.01	<0.01 <0.01	0.40 0.16	RES95001
VA (Market Prize) wrapper leaves removed	2x0.10 +2x0.15		290	4	1 3	0.03 0.02	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	0.03 0.03	RES95001
MUSTARD GREENS												
AZ (So. Giant Curled)	2x0.10 +2x0.15		280	4	1 3	<u>4.9</u> 4.3	<u>0.59</u> 0.52	<0.01 0.01	0.28 0.29	0.03 0.04	5.8 5.2	RES95001
CA (Florida Broadleaf)	2x0.10 +2x0.15		280	4	1 3	<u>5.1</u> 3.0	<u>0.64</u> 0.37	0.01 <0.01	0.25 0.19	0.04 0.03	6.1 3.6	RES95001
IN (Florida Broadleaf)	2x0.10 +2x0.15		280	4	0 1 3 5 7 10	<u>4.4</u> 2.1 0.078 0.02 0.02 <0.01	<u>0.55</u> 0.30 0.01 <0.01 <0.01 <0.01	0.01 0.01 <0.01 <0.01 <0.01 <0.01	0.18 0.16 <0.01 <0.01 <0.01 <0.01	0.02 0.01 <0.01 <0.01 <0.01 <0.01	5.2 2.5 0.10 0.02 0.02 <0.01	RES95001
MS (Florida Broadleaf)	2x0.10 +2x0.15		280	4	1 3 5	<u>3.6</u> 0.14 0.02	<u>0.40</u> 0.02 <0.01	<0.01 <0.01 <0.01	0.21 0.01 <0.01	0.03 <0.01 <0.01	4.2 0.17 0.02	RES95001
TX (Florida Broadleaf)	2x0.10 +2x0.15		280	4	1 3	<u>0.04</u> 0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	0.05 0.01	RES95001 TX1
TX (Florida Broadleaf)	2x0.10 +2x0.15		280	4	1 3	<u>3.1</u> 3.3	<u>0.43</u> 0.47	<0.01 <0.01	0.11 0.13	0.01 0.02	3.6 3.9	RES95001 TX2

Crop, Location (variety)	Application				PHI, days	Spinosyn residues, mg/kg						Ref.
	kg ai/ha	kg ai/hl	water, l/ha	no.		A	D	K	B	B of D	Total	
VA (Old Fashioned)	2x0.10 +2x0.15		280	4	1	<u>0.83</u>	<u>0.12</u>	<0.01	0.069	0.01	1.0	RES95001 VA1
					3	0.41	0.060	<0.01	0.03	<0.01	0.51	
					5	0.17	0.02	<0.01	0.02	<0.01	0.22	
VA (Old Fashioned)	2x0.10 +2x0.15		280	4	1	<u>5.0</u>	<u>0.62</u>	0.01	0.18	0.01	5.8	RES95001 VA2
					3	3.0	0.40	0.01	0.12	0.01	3.5	

Table 47. Spinosad residues in cucurbits from supervised trials in the USA in 1997. Double-underlined residues are from treatments according to GAP and are valid for the estimation of maximum residue levels.

Location (variety)	Application					PHI, days	Spinosyn residues, mg/kg immunoassay	Ref.
	Form	kg ai/ha	kg ai/hl	water, l/ha	no.			
CUCUMBER								
FL (Sunre 3775)	SC 240	5×0.075 +1×0.15		450	6	1	<u>0.046</u>	RES97002 FL1
MI (Marketmore)	SC 240	5×0.075 +1×0.15		270	6	1	<u>0.053</u>	RES97002 MI
NC (Straight Eight)	SC 240	5×0.075 +1×0.15		240	6	1	<u>0.01</u>	RES97002 NC1
NC (Burpless)	SC 240	5×0.075 +1×0.15		230	6	1	<u>0.059</u>	RES97002 NC2
OH (General Lee)	SC 240	5×0.075 +1×0.15		230	6	1	<u>0.052</u>	RES97002 OH1
TX (Dasher II)	SC 240	5×0.075 +1×0.15		360	6	1	<u>0.024</u>	RES97002 TX5
MUSKMELON								
CA (Jumbo Hales Best)	SC 240	5×0.075 +1×0.15		270	6	3 3	<u>0.12</u> f<0.01	RES97002 CA1
CA (Mission)	SC 240	5×0.075 +1×0.15		310	6	3	<u>0.036</u>	RES97002 CA2
CA (Primo cantaloupe)	SC 240	5×0.075 +1×0.15		320	6	3	<u>0.045</u>	RES97002 CA3
NC (Burpee Hybrid muskmelon)	SC 240	5×0.075 +1×0.15		220	6	3	<u>0.054</u>	RES97002 NC3
OH (Burpee Hybrid PMT Muskmelon)	SC 240	5×0.075 +1×0.15		220	6	3 3	<u>0.16</u> f<0.01	RES97002 OH2

Location (variety)	Application					PHI, days	Spinosyn residues, mg/kg immunoassay	Ref.
	Form	kg ai/ha	kg ai/hl	water, l/ha	no.			
TX Hales Best #36)	SC 240	5×0.075 +1×0.15		350	6	3 3	<u>0.092</u> f <0.01	RES97002 TX4
SUMMER SQUASH								
CA (Bennings Green tint)	SC 240	5×0.075 +1×0.15		290	6	3	<u>0.038</u>	RES97002 CA4
FL (Hurricane 9718)	SC 240	5×0.075 +1×0.15		450	6	3	<u>0.024</u>	RES97002 FL2
NC (Yellow Straightneck)	SC 240	5×0.075 +1×0.15		250	6	3	< <u>0.01</u>	RES97002 NC4

Table 48. Spinosad residues in tomatoes from supervised trials in Australia and New Zealand. Double-underlined residues are from treatments according to GAP and are valid for the estimation of maximum residue levels.

Country, year (variety)	Application				PHI, days	Residues, mg/kg	Ref.
	Form	kg ai/ha	water, l/ha	no.			
Australia (NSW), 1996 (82B)	SC480	0.10	99	4	0 1 3 7 13	0.03 <u>0.03</u> c 0.013 0.02 <0.01 <0.01	GHF-P 1535
Australia (NSW), 1996 (82B)	SC480	0.20	99	4	0 1 3 7 13	0.08 0.06 c 0.013 0.02 <0.01 0.01	GHF-P 1535
Australia (NSW), 1997 (Atlas)	SC 120	0.096	99	4	0 1 2 7	0.05 <u>0.03</u> 0.02 <0.01	GHF-P 1567
Australia (NSW), 1997 (Atlas)	SC 120	0.19	99	4	0 1 2 7	0.15 0.08 0.07 0.02	GHF-P 1567
Australia (NSW), 1997 (Pacesetter)	SC 120	0.096	99	4	0 1 2 7	0.05 <u>0.02</u> 0.03 <0.01	GHF-P 1567
Australia (NSW), 1997 (Pacesetter)	SC 120	0.19	99	4	0 1 2 7	0.13 0.09 0.06 0.02	GHF-P 1567

Country, year (variety)	Application				PHI, days	Residues, mg/kg	Ref.
	Form	kg ai/ha	water, l/ha	no.			
Australia (Vic), 1996 (Arcadia)	SC480	0.10	250	4	0 1 3 7 15	0.07 <u>0.04</u> 0.02 <0.01 <0.01	GHF-P 1535
Australia (Vic), 1996 (Arcadia)	SC480	0.20	250	4	0 1 3 7 15	0.13 0.08 0.04 <0.01 <0.01	GHF-P 1535
Australia (Vic), 1997 (Red Gem)	SC 120	0.096	250	4	0 1 3 7	0.05 <u>0.03</u> 0.01 <0.01	GHF-P 1567
Australia (Vic), 1997 (Red Gem)	SC 120	0.19	250	4	0 1 3 7	0.17 0.07 0.08 0.01	GHF-P 1567
NZ, 1997 (H232)	SC 120	0.096	470	4	0 1 3 7 14	0.06 <u>0.04</u> <0.01 <0.01 <0.01	GHF-P 1568 10650
NZ, 1997 (H232)	SC 120	0.048	470	4	0 1 3 7 14	0.03 0.02 <0.01 <0.01 <0.01	GHF-P 1568 510650

c: sample from control plot

Table 49. Spinosad residues in tomatoes from supervised trials in Argentina and Brazil. Replicate residues represent samples from split or replicate plots. Double-underlined residues are from treatments according to GAP and are valid for the estimation of maximum residue levels.

Country (location), year (variety)	Application				PHI, days	Residues, mg/kg		Ref.
	Form	kg ai/ha	water, l/ha	no.		spinosyn A	spinosyn D	
Argentina (Buenos Aires), 1996 (Lerika)	SC 480	0.12	1000	5	0 3 7 14	0.35 0.32 0.39 <u>0.05</u> <0.01 0.01 <0.01 <0.01 0.01 0.02 0.01 0.04	0.05 0.05 0.06 <u>0.01</u> <0.01 <0.01 <0.01(3) <0.01(3)	GHB-P 370
Argentina (Buenos Aires), 1996 (Lerika)	SC 480	0.24	1000	5	0 3 7 14	0.72 0.76 0.33 0.11 0.19 0.18 0.02 0.01 0.02 0.02 0.01 0.05	0.11 0.12 0.05 0.02 0.03 0.03 <0.01(3) <0.01 <0.01 0.01	GHB-P 370
Argentina (Buenos Aires), 1997 (Lerika)	SC 480	0.12	1000	5	1 4 8	0.11 0.07 0.07 <u>0.18</u> 0.09 0.08 0.09 0.04 0.07	0.02 0.01 0.01 <u>0.03</u> 0.02 0.01 0.02 <0.01 0.01	GHB-P 370

Country (location), year (variety)	Application				PHI, days	Residues, mg/kg		Ref.
	Form	kg ai/ha	water, l/ha	no.		spinosyn A	spinosyn D	
Argentina (Buenos Aires), 1997 (Lerika)	SC 480	0.24	1000	5	1 4 8	0.21 0.29 0.25 0.20 0.17 0.16 0.12 0.09 0.09	0.04 0.05 0.04 0.03 0.03 0.03 0.02 0.02 0.02	GHB-P 370
Argentina (Mendoza), 1996 (Presto)	SC 480	0.12	2600-3300	5	0 3 7 14	0.15 0.09 0.11 0.12 0.06 0.09 0.05 0.05 0.08 <u>0.15</u> 0.04 0.08	0.03 0.01 0.02 0.02 <0.01 0.01 <0.01 <0.01 0.01 <u>0.02</u> <0.01 0.01	GHB-P 370
Argentina (Mendoza), 1996 (Presto)	SC 480	0.24	2600-3300	5	0 3 7 14	0.47 0.36 0.41 0.24 0.33 0.38 0.16 0.22 0.15 0.09 0.12 0.08	0.07 0.06 0.06 0.04 0.05 0.06 0.03 0.04 0.03 0.02 0.02 0.01	GHB-P 370
Argentina (Mendoza), 1997 (Missouri)	SC 480	0.12	300-600	5	0 3 7 14	0.14 0.22 0.18 0.04 <u>0.06</u> 0.06 <0.01(3) <0.01(3)	0.02 0.03 0.03 <0.01(3) <0.01(3) <0.01(3)	GHB-P 370
Argentina (Mendoza), 1997 (Missouri)	SC 480	0.24	300-600	5	0 3 7 14	0.15 0.14 0.19 0.15 0.13 0.12 <0.01 <0.01 0.01 <0.01(3)	0.02 0.02 0.03 0.02 0.02 0.02 0.01 <0.01 <0.01 <0.01(3)	GHB-P 370
Argentina (Santiago del Estero), 1996 (6002)	SC 480	0.12	1200	5	0 3 7 14	0.02 0.33 0.02 <0.01 <u>0.01</u> <0.01 <0.01(3) <0.01(3)	<0.01 0.05 <0.01 <0.01(3) <0.01(3) <0.01(3)	GHB-P 370
Argentina (Santiago del Estero), 1996 (6002)	SC 480	0.24	1200	5	0 3 7 14	0.05 0.04 0.02 0.03 0.02 0.02 0.02 0.01 0.01 <0.01 <0.01 0.01	0.01 0.01 <0.01 <0.01(3) <0.01(3) <0.01(3)	GHB-P 370
Argentina (Santiago del Estero), 1997 (Rio Grande)	SC 480	0.12	1200	4	0 3 7 14	0.07 0.07 0.04 <u>0.02</u> 0.01 <0.01 <0.01 <0.01 0.01 0.01 0.01 <0.01	0.01 0.01 <0.01 <0.01(3) <0.01(3) <0.01(3)	GHB-P 370
Argentina (Santiago del Estero), 1997 (Rio Grande)	SC 480	0.24	1200	4	0 3 7 14	0.08 0.04 0.07 0.01 0.01 0.02 0.01 <0.01 0.01 <0.01 0.01 0.01	0.02 <0.01 0.01 <0.01(3) <0.01(3) <0.01(3)	GHB-P 370
Brazil (Paraná), 1995 (Grupo Santa Clara)	WG 800	0.20	1000	6	0 3 7 14 21	0.04 0.07 0.05 0.01 0.02 0.01 <0.01 (2) 0.02 <0.01 (2) 0.01 <0.01 (3)	<0.01 (3) <0.01 (3) <0.01 (3) <0.01 (3) <0.01 (3)	GHB P-331
Brazil (Paraná), 1995 (Grupo Santa Clara)	WG 800	0.40	1000	6	0 3 7 14 21	0.12 0.17 0.12 0.04 0.06 0.10 0.04 0.05 0.05 0.03 0.03 0.04 0.02 0.01 0.03	0.01 0.02 <0.01 <0.01 (2) 0.01 <0.01 (3) <0.01 (3) <0.01 (3)	GHB P-331

Country (location), year (variety)	Application				PHI, days	Residues, mg/kg		Ref.
	Form	kg ai/ha	water, l/ha	no.		spinosyn A	spinosyn D	
Brazil (São Paulo), 1995 (Angela 5200)	WG 800	0.20	1000	6	0 3 7 14 21	0.02 0.03 0.02 0.01 <0.01 (2) <0.01 (3) <0.01 (3) <0.01 (3)	<0.01 (3) <0.01 (3) <0.01 (3) <0.01 (3) <0.01 (3)	GHB P-331
Brazil (São Paulo), 1995 (Angela 5200)	WG 800	0.40	1000	6	0 3 7 14 21	0.10 0.07 0.09 0.04 <0.01 (2) <0.01 (3) <0.01 (3) <0.01 (3)	0.01 <0.01 0.01 <0.01 (3) <0.01 (3) <0.01 (3) <0.01 (3)	GHB P-331
Brazil (São Paulo), 1996 (Santa Clara)	SC 480	0.082	1000	6	0 1 3 7 14	0.02 0.01 0.02 0.01 0.01 <0.01 <0.01 (3) <0.01 (3) <0.01 (3)	<0.01 (3) <0.01 (3) <0.01 (3) <0.01 (3) <0.01 (3)	GHB P-337
Brazil (São Paulo), 1996 (Santa Clara)	SC 480	0.16	1000	6	0 1 3 7 14	0.04 (3) 0.02 0.03 <0.01 0.03 0.02 0.02 <0.01 (2) 0.02 <0.01 (3)	<0.01 (3) <0.01 (3) <0.01 (3) <0.01 (3) <0.01 (3)	GHB P-337
Brazil (São Paulo), 1996 (Santa Clara)	SC 480	0.082	1000	6	0 1 3	0.03 (3) 0.01 (3) <0.01 (2) 0.02	<0.01 (3) <0.01 (3) <0.01 (3)	GHB P-337
Brazil (São Paulo), 1996 (Santa Clara)	SC 480	0.16	1000	6	0 1 3	0.06 0.07 0.05 0.01 (3) <0.01 (2) 0.02	<0.01 (3) <0.01 (3) <0.01 (3)	GHB P-337

Table 50. Spinosad residues in tomatoes from supervised trials in Greece, Italy, Spain and the UK.

Country, year (variety)	Application					PHI, days	Spinosyn residues, mg/kg				Ref.
	Form	kg ai/ha	kg ai/hl	water, l/ha	no. <sup>3</sup>		A HPLC	D HPLC	Total <sup>1</sup> HPLC	IA <sup>2</sup>	
Greece, 1998 (Galli)	SC 480	0.58	0.094	620 +610 +620	2 3	14 0 2 3 6	0.01 0.10 0.04 0.02 0.02	<0.01 0.06 0.02 0.01 0.02	0.01 0.16 0.06 0.03 0.04		GHE-P-7858 R98-026B
Greece, 1998 (Noa F1)	SC 480	0.57 +0.58 +0.57	0.094	610 +610 +600	2 3	14 0 2 3 6	0.03 0.35 0.10 0.04 0.03	0.03 0.26 0.07 0.02 0.02	0.06 0.63 0.17 0.06 0.05		GHE-P-7858 R98-026A
Italy, 1997 (Erminia Peto Seed)	SC 480	0.54	0.067	800	5	3 4	0.30	0.08	0.40	0.38	GHE-P-7585



Country, year (variety)	Application					PHI, days	Spinosyn residues, mg/kg				Ref.
	Form	kg ai/ha	kg ai/hl	water, l/ha	no. <sup>3</sup>		A HPLC	D HPLC	Total <sup>1</sup> HPLC	IA <sup>2</sup>	
Italy, 1997 (ES 200)	SC 480	0.53	0.067	790	g 4 g 5	11	0.20	0.05	0.26	0.34	GHE-P-7580
		+0.55		+810		0	0.61	0.13	0.75	1.1	
		+0.55		+810		2	0.38	0.09	0.47	0.70	
		+0.54		+800		3	0.45	0.10	0.57	0.68	
		+0.55		+820		6	0.30	0.06	0.37	0.42	
						4					
Italy, 1997 (UC 82)	SC 480	0.53	0.066	810	5	3 6	0.11 0.20	0.03 0.05	0.12 0.24	0.11 0.26	GHE-P-7589
Italy, 1997 (UC 82R Peto seed)	SC 480	0.54	0.067	800	4 5	11	0.11	0.03	0.15	0.21	GHE-P-7583
		+0.55		+810		0	0.54	0.12	0.66	0.76	
		+0.54		+800		2	0.08	0.03	0.10	0.11	
		+0.53		+790		3	0.04	0.01	0.05	0.06	
		+0.54		+810		6	0.03	<0.01	0.04	0.06	
						4					
Italy, 1998 (Cauda)	SC 480	0.55	0.057	970	g 2 g 3	19	0.07	0.05	0.12		GHE-P-7860
		+0.57		+1000		0	0.22	0.15	0.37		
		+0.57		+1000		2	0.19	0.13	0.32		
						3	0.21	0.15	0.36		
						7	0.08	0.06	0.14		
Italy, 1998 (ES 580)	SC 480	0.55	0.067	810	g 3	3 6	0.08 0.06	0.06 0.04	0.14 0.10		GHE-P-7865 R98-033A
Italy, 1998 (EXH 98063)	SC 480	0.54	0.067	800	3	3 6	0.06 0.02	0.05 0.02	0.11 0.04		GHE-P-7861 R98-029A
Spain, 1997 (Daniella)	SC 480	0.43	0.043	990	g 5	3	0.25	0.08	0.32	0.35	GHE-P-7587
		+0.42		+980		6	0.29	0.08	0.36	0.41	
		+0.49		+900							
		+0.53		+980							
		+0.55		+1010							
Spain, 1997 (Durinta)	SC 480	0.58	0.077	750	g 4 g 5	10	0.07	0.02	0.09	0.14	GHE-P-7581
		+0.54		+810		0	0.15	0.05	0.21	0.36	
		+0.53		+880		2	0.15	0.06	0.22	0.37	
		+0.52		+970		3	0.17	0.05	0.23	0.36	
		+0.55		+1120		6	0.26	0.08	0.35	0.54	
Spain, 1997 (Durinta)	SC 480	0.54	0.054	1000	4 5	12	0.02	<0.01	0.03	0.04	GHE-P-7584
		+0.53		+980		0	0.14	0.03	0.18	0.22	
		+0.54		+1000		2	0.17	0.04	0.21	0.26	
		+0.55		+1010		3	0.22	0.06	0.29	0.37	
		+0.55		+1020		6	0.10	0.03	0.12	0.18	
Spain, 1997 (Durinta)	SC 480	0.55	0.054	1030	5	3	0.07	0.02	0.09	0.10	GHE-P-7588
		+0.55		+1030		6	0.02	<0.01	0.03	0.04	
		+0.51		+950							
		+0.51		+950							
		+0.54		+1000							
Spain, 1998 (Durinta)	SC 480	0.52	0.090	580	2 3	14	0.04	0.04	0.08		GHE-P-7857
		+0.53		+790		0	0.14	0.10	0.25		
		+0.57		+1050		2	0.09	0.06	0.15		
						3	0.09	0.07	0.17		
						6	0.08	0.06	0.14		



Country, year (variety)	Application				PHI, days	Spinosyn residues, mg/kg						Ref.
	Form	kg ai/ha	water, l/ha	no.		A	D	K	B	B of D	Total	
USA (CA), 1996 (Dimare 540)	WG 800	0.060 +0.058 +0.089 +0.087 +0.12 +0.17	520	6	1 3	<u>0.023</u> <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	0.026 <0.01	RES96009/ RES96008 CA1
USA (CA), 1996 (Dimare 540)	SC 480	0.056 +0.055 +0.087 +0.085 +0.12 +0.16	490	6	1 3	<u>0.013</u> 0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	0.013 0.01	RES96009/ RES96008 CA2
USA (CA), 1996 (Dimare 540)	WG 800	0.054 +0.053 +0.082 +0.083 +0.12 +0.17	530	6	1 3	<u>0.024</u> 0.018	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	0.027 0.020	RES96009/ RES96008 CA2
USA (FL), 1996 (Better Boy)	SC 480	0.050 +0.052 +0.075 +0.076 +0.10 +0.15	280	6	1 3	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	RES96009/ RES96008 FL
USA (FL), 1996 (Better Boy)	WG 800	0.048 +0.049 +0.074 +0.074 +0.10 +0.15	290	6	1 3	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	RES96009/ RES96008 FL
USA (CA), 1995 (Peelmech, processing)	WG 800	0.050 +3x0.10 +0.15	290	5	0 1 3	0.071 <u>0.062</u> 0.05	0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	0.085 0.070 0.05	RES95016 CA1
USA (CA), 1995 (Halley 3155, processing)	WG 800	0.050 +3x0.10 +0.15	290	5	0 1 3	0.062 <u>0.10</u> 0.058	<0.01 <u>0.01</u> <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	0.071 0.11 0.066	RES95016 CA2
USA (CA), 1995 (La Rossa, processing)	WG 800	0.050 +3x0.10 +0.15	280	5	0 1 3	0.057 <u>0.04</u> 0.03	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	0.069 0.05 0.03	RES95016 CA3
USA (CA), 1995 (Roma)	WG 800	0.050 +3x0.10 +0.15	280	5	0 1 3 7 10 14	0.03 <u>0.02</u> 0.01 <0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01	0.04 0.02 0.01 <0.01 <0.01 <0.01	RES95016 CA4

Country, year (variety)	Application				PHI, days	Spinosyn residues, mg/kg						Ref.
	Form	kg ai/ha	water, l/ha	no.		A	D	K	B	B of D	Total	
USA (CA), 1995 (Shady Lady)	WG 800	0.050 +3x0.10 +0.15	290	5	0	0.059	<0.01	<0.01	<0.01	<0.01	0.067	RES95016 CA5
					1	<u>0.03</u>	<0.01	<0.01	<0.01	<0.01	0.04	
					3	0.02	<0.01	<0.01	<0.01	<0.01	0.03	
USA (CA), 1996 (Shady Lady)	WG 800	0.050 +3x0.10 +0.15	280	5	0	0.058	<0.01	<0.01	<0.01	<0.01	0.064	RES95016 CA6
					1	<u>0.04</u>	<0.01	<0.01	<0.01	<0.01	0.05	
					3	0.05	<0.01	<0.01	<0.01	<0.01	0.059	
USA (CA), 1995 (Shady Lady)	WG 800	0.050 +3x0.10 +0.15	280	5	0	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	RES95016 CA7
					1	<u>0.03</u>	<0.01	<0.01	<0.01	<0.01	0.03	
					3	0.02	<0.01	<0.01	<0.01	<0.01	0.02	
USA (FL), 1995 (Fresh Market)	WG 800	0.050 +3x0.10 +0.15	280	5	0	0.04	<0.01	<0.01	<0.01	<0.01	0.05	RES95016 FL1
					1	<u>0.03</u>	<0.01	<0.01	<0.01	<0.01	0.04	
					3	0.02	<0.01	<0.01	<0.01	<0.01	0.02	
USA (FL), 1995 (Agriset)	WG 800	0.050 +3x0.10 +0.15	280	5	0	0.03	<0.01	<0.01	<0.01	<0.01	0.03	RES95016 FL2
					1	<u>0.02</u>	<0.01	<0.01	<0.01	<0.01	0.02	
					3	<u>0.03</u>	<0.01	<0.01	<0.01	<0.01	0.03	
USA (OH), 1995 (Heinz 8813, processing)	WG 800	0.050 +3x0.10 +0.15	180	5	0	0.060	<0.01	<0.01	<0.01	<0.01	0.067	RES95016
					1	<u>0.076</u>	<u>0.01</u>	<0.01	<0.01	<0.01	0.091	
					3	0.02	<0.01	<0.01	<0.01	<0.01	0.03	
USA (PA), 1995 (La Roma, processing)	WG 800	0.050 +3x0.10 +0.15	290	5	0	0.01	<0.01	<0.01	<0.01	<0.01	0.01	RES95016
					1	<u>0.02</u>	<0.01	<0.01	<0.01	<0.01	0.02	
					3	0.01	<0.01	<0.01	<0.01	<0.01	0.01	
USA (VA), 1995 (Mountain Pride)	WG 800	0.050 +3x0.10 +0.15	340	5	0	0.11	0.01	<0.01	<0.01	<0.01	0.13	RES95016
					1	<u>0.11</u>	<0.01	<0.01	<0.01	<0.01	0.13	
					3	0.05	<0.01	<0.01	<0.01	<0.01	0.059	

Table 52. Spinosad residues in peppers from supervised trials in the USA . Double-underlined residues are from treatments according to GAP and are valid for the estimation of maximum residue levels.

Location, year (variety)	Application				PHI, days	Spinosyn residues, mg/kg						Ref.
	Form	kg ai/ha	water, l/ha	no.		A	D	K	B	B of D	Total	
AZ, 1995 (S414 Peppers, hot)	WG 800	0.050 +3x0.10 +0.15	290	5	0	0.13	0.01	<0.01	<0.01	<0.01	0.15	RES95016
					1	<u>0.15</u>	<u>0.02</u>	<0.01	<0.01	<0.01	0.17	
					3	0.083	0.01	<0.01	<0.01	<0.01	0.097	
CA, 1995 (Jupiter, bell)	WG 800	0.050 +3x0.10 +0.15	290	5	0	0.058	<0.01	<0.01	<0.01	<0.01	0.065	RES95016 CA8
					1	<u>0.063</u>	<u>0.01</u>	<0.01	<0.01	<0.01	0.081	
					3	0.03	<0.01	<0.01	<0.01	<0.01	0.03	
CA, 1995 (Milta, hot)	WG 800	0.050 +3x0.10 +0.15	290	5	0	0.14	0.02	<0.01	<0.01	<0.01	0.16	RES95016 CA9
					1	<u>0.12</u>	<u>0.02</u>	<0.01	<0.01	<0.01	0.14	
					3	0.10	0.01	<0.01	<0.01	<0.01	0.12	

Location, year (variety)	Application				PHI, days	Spinosyn residues, mg/kg						Ref.
	Form	kg ai/ha	water, l/ha	no.		A	D	K	B	B of D	Total	
FL, 1995 (Jupiter, bell)	WG 800	0.050 +3x0.10 +0.15	290	5	0	0.093	0.01	<0.01	<0.01	<0.01	0.11	RES95016
					1	<u>0.05</u>	<0.01	<0.01	<0.01	<0.01	0.054	
					3	0.05	<0.01	<0.01	<0.01	<0.01	0.060	
OH, 1995 (Cal Wonders, bell)	WG 800	0.050 +3x0.10 +0.15	290	5	0	0.075	0.01	<0.01	<0.01	<0.01	0.084	RES95016
					1	<u>0.05</u>	<0.01	<0.01	<0.01	<0.01	0.055	
					3	0.03	<0.01	<0.01	<0.01	<0.01	0.03	
TX, 1995 (Wonder Rio 66, bell)	WG 800	0.050 +3x0.10 +0.15	290	5	0	0.075	<0.01	<0.01	<0.01	<0.01	0.084	RES95016 TX1
					1	<u>0.062</u>	<0.01	<0.01	<0.01	<0.01	0.069	
					3	0.03	<0.01	<0.01	<0.01	<0.01	0.03	
TX, 1995 (Jalapeno, hot)	WG 800	0.050 +3x0.10 +0.15	290	5	0	0.04	<0.01	<0.01	<0.01	<0.01	0.04	RES95016 TX2
					1	<u>0.03</u>	<0.01	<0.01	<0.01	<0.01	0.04	
					3	0.02	<0.01	<0.01	<0.01	<0.01	0.02	
VA, 1995 (Giant, bell)	WG 800	0.050 +3x0.10 +0.15	290	5	0	0.03	<0.01	<0.01	<0.01	<0.01	0.04	RES95016
					1	<u>0.02</u>	<0.01	<0.01	<0.01	<0.01	0.02	
					3	0.01	<0.01	<0.01	<0.01	<0.01	0.01	
					7	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	
					10	0.01	<0.01	<0.01	<0.01	<0.01	0.01	
					14	0.02	<0.01	<0.01	<0.01	<0.01	0.02	

Table 53. Spinosad residues in sweet peppers from supervised trials in Australia, Greece, Italy, Mexico, Spain and the UK. Double-underlined residues are from treatments according to GAP and are valid for the estimation of maximum residue levels.

Country, year (variety)	Application <sup>4</sup>					PHI, days	Spinosyn residues, mg/kg				Ref.
	Form	kg ai/ha	kg ai/hl	water, l/ha	no. <sup>3</sup>		A HPLC	D HPLC	Total <sup>1</sup> HPLC	IA <sup>2</sup>	
Australia (Qld), 1997 (Target capsicum)	SC 120	0.096		300 +450 +360 +360	4	0				0.05	GHF-P 1584
						1				<u>0.04</u>	
						3				0.03	
						7				0.03	
Australia (Qld), 1997 (Target capsicum)	SC 120	0.19		300 +450 +360 +360	4	0				0.24	GHF-P 1584
						1				0.14	
						3				0.09	
						7				0.06	
Australia (Vic), 1997 (Domino capsicum)	SC 120	0.096		250	4	0				0.12	GHF-P 1584
						1				<u>0.12</u>	
						3				0.06	
						7				0.02	
Australia (Vic), 1997 (Domino capsicum)	SC 120	0.19		250	4	0				0.20	GHF-P 1584
						1				0.23	
						3				0.23	
						7				0.10	

Country, year (variety)	Application <sup>4</sup>					PHI, days	Spinosyn residues, mg/kg				Ref.
	Form	kg ai/ha	kg ai/hl	water, l/ha	no. <sup>3</sup>		A HPLC	D HPLC	Total <sup>1</sup> HPLC	IA <sup>2</sup>	
Greece, 1998 (Stamboli)	SC 480	0.44	0.055	800	2 3	13 0 2 3 6	<0.01 0.41 0.15 0.06 0.05	<0.01 0.13 0.04 0.02 0.02	<0.01 0.56 0.20 0.08 0.07		GHE-P-7873
Italy, 1997 (Magister)	SC 480	0.43	0.054	800	4 5	11 0 2 3 6	<0.01 0.31 0.01 0.01 0.01	<0.01 0.09 <0.01 <0.01 <0.01	<0.01 0.43 0.02 0.02 0.01	<0.01 0.39 <0.01 0.01 0.01	GHE-P-7751
Italy, 1997 (Magister)	SC 480	0.43	0.054	810	5	3 6	0.06 <0.01	0.02 <0.01	0.08 <0.01	0.10 0.01	GHE-P-7912 R97-053B
Italy, 1997 (Moutero)	SC 480	0.41	0.052	790	g 4 g 5	7 0 2 3 6 3	0.79 1.1 1.07 1.2 0.96 c <0.01	0.20 0.27 0.28 0.32 0.23 c <0.01	1.1 1.5 1.4 1.7 1.3 c 0.02	1.1 1.2 1.1 2.0 0.88 c <0.01	GHE-P-7664
Italy, 1997 (Sanbor)	SC 480	0.37	0.052	700	5	3 6	0.13 0.07	0.04 0.02	0.16 0.10	0.19 0.14	GHE-P-7912 R97-053A
Italy, 1998 (Lipari)	SC 480	0.44	0.038	1160	3	3 6	0.08 0.03	0.07 0.04	0.15 0.08		GHE-P-7867
Italy, 1998 (Livor)	SC 480	0.43	0.054	790	g 3	3 6	0.35 0.28	0.25 0.19	0.63 0.49		GHE-P-7868R
Mexico, 1995 (Bell pepper, Galaxie)	WG 820	0.050 +0.11 +0.10 +0.092 +0.15		240 +360 +360 +350 +330	5	0 1 3 7	0.11 0.062 0.043 0.031	0.014 0.01 <0.01 <0.01	0.15 0.087 0.053 0.038		RES95076 MEX9502
Spain, 1997 (Carisma)	SC 480	0.43	0.33	1300	g 4 g 5	12 0 2 3 6	0.16 0.22 0.20 0.17 0.13	0.05 0.06 0.05 0.05 0.03	0.23 0.32 0.29 0.26 0.18	0.24 0.31 0.28 0.23 0.19	GHE-P-7685
Spain, 1997 (Lamuyo Largo)	SC 480	0.43	0.036	1200	4 5	11 0 2 3 6	0.04 0.12 0.10 0.17 0.06	0.01 0.03 0.03 0.05 0.02	0.05 0.17 0.13 0.23 0.09	0.04 0.25 0.17 0.35 0.10	GHE-P-7717
Spain, 1997 (Local)	SC 480	0.43	0.43	1000	5	3 6	0.18 0.11	0.05 0.03	0.24 0.14	0.32 0.18	GHE-P-7790
Spain, 1997 (Rosi)	SC 480	0.41	0.43	950	g 5	3 6	0.16 0.07	0.04 0.02	0.23 0.09	0.24 0.12	GHE-P-7784
Spain, 1998 (Atol)	SC 480	0.43	0.036	1180	g 3	3 6	0.20 0.16	0.05 0.04	0.25 0.20		GHE-P-7869R

Country, year (variety)	Application <sup>4</sup>					PHI, days	Spinosyn residues, mg/kg				Ref.
	Form	kg ai/ha	kg ai/hl	water, l/ha	no. <sup>3</sup>		A HPLC	D HPLC	Total <sup>1</sup> HPLC	IA <sup>2</sup>	
Spain, 1998 (Atol)	SC 480	0.43	0.036	1200	g 2 g 3	14 0 2 3 6 5	0.09 0.43 0.29 0.25 0.19	0.02 0.09 0.07 0.06 0.05	0.12 0.53 0.37 0.33 0.25		GHE-P-7872
Spain, 1998 (Dulce)	SC 480	0.45	0.045	1000	3	3 6	0.08 0.07	0.04 0.05	0.12 0.12		GHE-P-7866
Spain, 1998 (Lamuyo telier)	SC 480	0.44	0.045	980	2 3	13 0 2 3 6	0.02 <0.01 0.21 0.06 0.04	0.01 <0.01 0.08 0.04 0.02	0.03 <0.01 0.30 0.09 0.06		GHE-P-7870R R98-021B
Spain, 1998 (Lamuyo)	SC 480	0.47	0.038	1240	2 3	13 0 2 3 6	0.07 0.18 0.17 0.08 0.10	0.07 0.13 0.14 0.05 0.08	0.15 0.32 0.32 0.13 0.19		GHE-P-7870R R98-021A
UK, 1997 (Mazurka RZ)	SC 480	0.43	0.036	1190	g 4 g 5	10 0 2 3 6	0.35 0.45 0.35 0.38 0.41	0.10 0.12 0.09 0.10 0.10	0.50 0.65 0.49 0.54 0.59	0.67 0.78 0.55 0.73 0.80	GHE-P-7618
UK, 1997 (Mazurka RZ)	SC 480	0.44	0.036	1220	g 5	3 6	0.27 0.25	0.10 0.09	0.45 0.42	0.49 0.48	GHE-P-7626
UK, 1998 (Mazurka RZ)	SC 480	0.45	0.046	1000	g 2 g 3	14 1 2 3 6	0.08 0.24 0.19 0.17 0.18	0.06 0.16 0.13 0.13 0.14	0.15 0.42 0.34 0.31 0.33		GHE-P-7871

<sup>1</sup> Total includes spinosyns A, D, B, B of D and K

<sup>2</sup> Immunoassay

<sup>3</sup> g: glasshouse

<sup>4</sup> Application rate, spray concentration and volume per ha were not identical (variation mostly within 20%). The values reported are those for the final application.

<sup>5</sup> Samples stored frozen for approx. 15 months

Table 54. Residues of spinosad in sweet corn kernels and cob (with husk removed) from supervised trials in the USA. Double-underlined residues are from treatments according to GAP and are valid for the estimation of maximum residue levels.

Location, year (variety)	Application				PHI, days	Residues, mg/kg	Ref.
	Form	kg ai/ha	water, l/ha	no.			
CA, 1997 (Legend)	SC 240	0.10	190	5	1	< <u>0.01</u>	RES97037
FL, 1997 (Silver Queen)	SC 240	0.10	280	5	1	< <u>0.01</u>	RES97037

Location, year (variety)	Application				PHI, days	Residues, mg/kg	Ref.
	Form	kg ai/ha	water, l/ha	no.			
MI, 1997 (Sweet Chorus)	SC 240	0.10	230	5	1	< <u>0.01</u>	RES97037
MN, 1997 (Quickie Hybrid)	SC 240	0.10	180	5	1	< <u>0.01</u> <0.01 (HPLC)	RES97037
NC, 1997 (Silver Queen)	SC 240	0.10	240	5	1	< <u>0.01</u>	RES97037
NY, 1997 (Tuxedo)	SC 240	0.10	180	5	1	< <u>0.01</u>	RES97037
OR, 1997 (Jubilee)	SC 240	0.10	290	5	1	< <u>0.01</u>	RES97037
WA, 1997 (Silver Sweet Jubilee)	SC 240	0.10	290	5	1	< <u>0.01</u>	RES97037
WI, 1997 (Confection SH2 Bi-color)	SC 240	0.10	180	5	1	< <u>0.01</u>	RES97037

Table 55. Spinosad residues in egg plant from supervised glasshouse trials in Japan, in 1995[?].

Application				PHI, days	Spinosyn residues, mg/kg						Ref.
Form	kg ai/hl	water, l/ha	no.		A	D	K	B	B of D	Total	
WG 250	0.005	300	1	1	0.05	0.01	0.02	<0.01	<0.01	0.08	GHF-P-1485 N-NASU
				3	0.02	<0.01	0.01	<0.01	<0.01	0.03	
				7	0.01	<0.01	<0.01	<0.01	<0.01	0.01	
			3	1	0.14	0.02	0.01	<0.01	<0.01	0.17	
				3	0.15	0.02	0.01	<0.01	<0.01	0.18	
				7	0.07	0.01	0.01	<0.01	<0.01	0.09	
WG 250	0.005	300	1	1	0.08	0.01	<0.01	0.01	<0.01	0.10	GHF-P-1485 CHIKUYOU
WG 250	0.0025	300	1	1	0.06	0.01	0.01	0.01	<0.01	0.09	GHF-P-1485 CHIKUYOU
WG 250	0.0013	300	1	1	0.02	<0.01	<0.01	<0.01	<0.01	0.02	GHF-P-1485 CHIKUYOU

Table 56. Spinosad residues in lettuce from supervised trials in Australia. Double-underlined residues are from treatments according to GAP and are valid for the estimation of maximum residue levels.

Location, year (variety)	Application				PHI, days	Spinosad, mg/kg immunoassay	Ref.
	Form	kg ai/ha	water, l/ha	no.			
Qld, 1998 (Summertime)	SC120	0.096 +adjuvant	400	4	0	0.38	GHF-P-1797 984008GW
					1	0.10	
					3	<u>0.21</u>	
					7	0.05	
					14	<0.01	



Location, year (variety)	Application				PHI, days	Spinosad, mg/kg immunoassay	Ref.
	Form	kg ai/ha	water, l/ha	no.			
Qld, 1998 (Summertime)	SC120	0.19 +adjuvant	400	4	0 1 3 7 14	0.98 0.44 0.42 0.05 0.01	GHF-P-1797 984008GW
Qld, 1998 (Summertime)	SC120	0.096	400	4	0 3 7 14 21 28	2.2 <u>1.1</u> 0.04 <0.01 0.01 <0.01	GHF-P-1797 984016GW
Qld, 1998 (Summertime)	SC120	0.19	400	4	0 3 7 14 21 28	3.6 1.3 0.19 0.01 <0.01 <0.01	GHF-P-1797 984016GW
NSW, 1998 (Marksman)	SC120	0.096	250	4	0 1 3 7 14 21	3.5 3.4 <u>1.7</u> 0.50 0.01 <0.01	GHF-P-1797 97381.03
NSW, 1998 (Marksman)	SC120	0.19	250	4	0 1 3 7 14 21	8.3 7.7 3.8 0.61 0.10 <0.01	GHF-P-1797 97381.03

Table 57. Spinosad residues in leafy vegetables from supervised trials in the USA. Double-underlined residues are from treatments according to GAP and are valid for the estimation of maximum residue levels.

Location, year (variety)	Application				PHI, days	Spinosyn residues, mg/kg						Ref.
	Form	kg ai/ha	water, l/ha	no.		A	D	K	B	B of D	Total	
HEAD LETTUCE												
CA, 1996 (Pybas 251) include wrapper leaves	SC 480	0.049 +0.050 +0.073 +0.076 +0.099 +0.14	300	6	1 3	<u>0.094</u> 0.063	<u>0.015</u> 0.01	<0.0 1 <0.0 1	0.01 <0.01	<0.01 <0.01	0.12 0.081	RES96009/ RES96008 CA2

Location, year (variety)	Application				PHI, days	Spinosyn residues, mg/kg						Ref.
	Form	kg ai/ha	water, l/ha	no.		A	D	K	B	B of D	Total	
CA, 1996 (Pybas 251) wrapper leaves removed	SC 480	0.049 +0.050 +0.073 +0.076 +0.099 +0.14	300	6	1 3	0.01 <0.01	<0.01 <0.01	<0.0 1 <0.0 1	<0.01 <0.01	<0.01 <0.01	0.01 <0.01	RES96009/ RES96008 CA2
CA, 1996 (Pybas 251) include wrapper leaves	WG 800	0.050 +0.051 +0.075 +0.077 +0.098 +0.15	300	6	1 3	<u>0.052</u> 0.091	<0.01 0.015	<0.0 1 <0.0 1	<0.01 0.01	<0.01 <0.01	0.064 0.11	RES96009/ RES96008 CA2
CA, 1996 (Pybas 251) wrapper leaves removed	WG 800	0.050 +0.051 +0.075 +0.077 +0.098 +0.15	300	6	1 3	<0.01 0.01	<0.01 <0.01	<0.0 1 <0.0 1	<0.01 <0.01	<0.01 <0.01	<0.01 0.01	RES96009/ RES96008 CA2
CA, 1996 (Van May) include wrapper leaves	SC 480	0.048 +0.048 +0.078 +0.074 +0.099 +0.16	300	6	1 3	<u>0.67</u> 0.59	<u>0.10</u> 0.092	<0.0 1 <0.0 1	0.029 0.023	<0.01 <0.01	0.80 0.71	RES96009/ RES96008 CA1
CA, 1996 (Van May) wrapper leaves removed	SC 480	0.048 +0.048 +0.078 +0.074 +0.099 +0.16	300	6	1 3	0.019 0.018	<0.01 <0.01	<0.0 1 <0.0 1	<0.01 <0.01	<0.01 <0.01	0.019 0.021	RES96009/ RES96008 CA1
CA, 1996 (Van May) include wrapper leaves	WG 800	0.049 +0.048 +0.074 +0.076 +0.10 +0.15	300	6	1 3	<u>0.59</u> 0.37	<u>0.082</u> 0.055	<0.0 1 <0.0 1	0.024 0.016	<0.01 <0.01	0.70 0.44	RES96009/ RES96008 CA1
CA, 1996 (Van May) wrapper leaves removed	WG 800	0.049 +0.048 +0.074 +0.076 +0.10 +0.15	300	6	1 3	0.024 0.015	<0.01 <0.01	<0.0 1 <0.0 1	<0.01 <0.01	<0.01 <0.01	0.028 0.017	RES96009/ RES96008 CA1
FL, 1996 (Great Lakes) include wrapper leaves	SC 480	0.049 +0.050 +0.076 +0.077 +0.099 +0.15	280	6	1 3	<u>0.63</u> 0.25	<u>0.10</u> 0.041	<0.0 1 <0.0 1	0.048 0.034	<0.01 <0.01	0.79 0.33	RES96009/ RES96008 FL

Location, year (variety)	Application				PHI, days	Spinosyn residues, mg/kg						Ref.
	Form	kg ai/ha	water, l/ha	no.		A	D	K	B	B of D	Total	
FL, 1996 (Great Lakes) wrapper leaves removed	SC 480	0.049 +0.050 +0.076 +0.077 +0.099 +0.15	280	6	1 3	0.15 0.077	0.022 0.012	<0.0 1 <0.0 1	0.020 0.015	<0.01 <0.01	0.19 0.10	RES96009/ RES96008 FL
FL, 1996 (Great Lakes) include wrapper leaves	WG 800	0.051 +0.049 +0.075 +0.076 +0.098 +0.15	280	6	1 3	<u>0.72</u> 0.27	<u>0.13</u> 0.044	<0.0 1 <0.0 1	0.069 0.039	0.01 <0.01	0.94 0.35	RES96009/ RES96008 FL
FL, 1996 (Great Lakes) wrapper leaves removed	WG 800	0.051 +0.049 +0.075 +0.076 +0.098 +0.15	280	6	1 3	0.17 0.24	0.024 0.036	<0.0 1 <0.0 1	0.026 0.038	<0.01 <0.01	0.23 0.32	RES96009/ RES96008 FL
SPINACH												
CA, 1996 (St Helens)	SC 480	0.047 +0.050 +0.068 +0.074 +0.10 +0.15	290	6	1 3	<u>2.5</u> 0.97	<u>0.37</u> 0.14	0.01 <0.0 1	0.23 0.12	0.030 0.014	3.1 1.2	RES96009/ RES96008 CA1
CA, 1996 (St Helens)	WG 800	0.049 +0.050 +0.067 +0.074 +0.10 +0.15	290	6	1 3	<u>2.6</u> 1.4	<u>0.37</u> 0.19	<0.0 1 <0.0 1	0.22 0.14	0.022 0.018	3.3 1.7	RES96009/ RES96008 CA1
CA, 1996 (Bossa- nova)	SC 480	0.51 +0.48 +0.67 +0.74 +0.10 +0.15	300	6	1 3	<u>2.4</u> 0.48	<u>0.37</u> 0.079	<0.0 1 <0.0 1	0.17 0.060	0.024 <0.01	3.0 0.63	RES96009/ RES96008 CA2
CA, 1996 (Bossa- nova)	WG 800	0.052 +0.051 +0.077 +0.078 +0.10 +0.16	310	6	1 3	<u>3.5</u> 0.64	<u>0.49</u> 0.10	<0.0 1 <0.0 1	0.23 0.069	0.035 <0.01	4.3 0.82	RES96009/ RES96008 CA2
TX, 1996 (Skookum)	SC 480	0.050 +0.050 +0.075 +0.076 +0.10 +0.15	390	6	1 3	<u>2.1</u> 1.1	<u>0.33</u> 0.15	<0.0 1 <0.0 1	0.10 0.080	0.023 0.01	2.6 1.3	RES96009/ RES96008 TX

Location, year (variety)	Application				PHI, days	Spinosyn residues, mg/kg						Ref.
	Form	kg ai/ha	water, l/ha	no.		A	D	K	B	B of D	Total	
TX, 1996 (Skookum)	WG 800	0.051	390	6	1	<u>3.9</u>	<u>0.54</u>	<0.0	0.13	0.018	4.6	RES96009/ RES96008 TX
		+0.051 +0.075 +0.077 +0.10 +0.15			3	2.3	0.33	1 <0.0 1	0.14	0.015	2.7	

Table 58. Spinosad residues in leafy vegetables and celery from supervised trials in the USA. Double-underlined residues are from treatments according to GAP and are valid for the estimation of maximum residue levels.

Location, year (variety)	Application				PHI, days	Spinosyn residues, mg/kg				Ref.
	Form	kg ai/ha	water, l/ha	no.		A HPLC	D HPLC	Total <sup>1</sup> HPLC	IA <sup>2</sup>	
HEAD LETTUCE										
AZ, 1996 (Diamond) include wrapper leaves	SC 480	0.053 +0.054 +0.079 +0.080 +0.11 +0.15	290	6	1 3	<u>1.7</u>	<u>0.27</u>	2.1	3.1 2.1	RES96008 AZ LV9606
AZ, 1996 (Diamond) wrapper leaves removed	SC 480	0.053 +0.054 +0.079 +0.080 +0.11 +0.15	290	6	1 3	1.6 0.40	0.25 0.069	1.9 0.50	2.3 0.61	RES96008 AZ LV9606
CA, 1996 (Jupiter) include wrapper leaves	SC 480	0.048 +0.051 +0.075 +0.077 +0.10 +0.15	320	6	0 1 3 5				<u>0.93</u> 0.92 0.097 0.13	RES96008 CA3 LV9605
CA, 1996 (Jupiter) wrapper leaves removed	SC 480	0.048 +0.051 +0.075 +0.077 +0.10 +0.15	320	6	0 1 3 5				0.073 0.052 0.09 0.01	RES96008 CA3 LV9605
NJ, 1996 (Ithaca) include wrapper leaves	SC 480	0.052 +0.051 +0.077 +0.077 +0.10 +0.15	290	6	1 3	<0.01	<0.01	0.01	<u>0.12</u> 0.012	RES96008 NJ LV9601
NJ, 1996 (Ithaca) wrapper leaves removed	SC 480	0.052 +0.051 +0.077 +0.077 +0.10 +0.15	290	6	1 3				<0.01 <0.01	RES96008 NJ LV9601
LEAF LETTUCE										
AZ, 1996 (Green Vision)	SC 480	0.052 +0.053 +0.081 +0.079 +0.10 +0.15	290	6	1 3	<u>4.1</u>	<u>0.61</u>	5.0	4.8 3.8	RES96008 AZ

Location, year (variety)	Application				PHI, days	Spinosyn residues, mg/kg				Ref.
	Form	kg ai/ha	water, l/ha	no.		A HPLC	D HPLC	Total <sup>1</sup> HPLC	IA <sup>2</sup>	
CA, 1996 (Big Hoss)	SC 480	0.049 +0.050 +0.079 +0.076 +0.097 +0.15	280	6	1 3	0.35	0.062	0.45	<u>2.0</u> 0.53	RES96008 CA1
CA, 1996 (Deep Red)	SC 480	0.046 +0.050 +0.075 +0.077 +0.099 +0.15	370	6	1 3				<u>5.2</u> 2.8	RES96008 CA2
CA, 1996 (Romaine)	SC 480	0.051 +0.051 +0.076 +0.075 +0.10 +0.15	470	6	1 3				<u>1.9</u> 0.27	RES96008 CA3
FL, 1996 (Butter Crunch)	SC 480	0.051 +0.053 +0.074 +0.077 +0.10 +0.15	280	6	1 3	<u>1.2</u> 0.70	<u>0.17</u> 0.10	1.5 0.92	1.9 1.0	RES96008 FL
NJ, 1996 (Salad Bowl)	SC 480	0.049 +0.051 +0.077 +0.076 +0.10 +0.15	280	6	1 3				<u>4.9</u> 1.1	RES96008 NJ
SPINACH										
NJ, 1996 (Winter Bloomsdale)	SC 480	0.049 +0.051 +0.077 +0.077 +0.10 +0.15	280	6	1 3				<u>1.5</u> 0.31	RES96008 NJ
VA, 1996 (Long Standing Bloomsdale)	SC 480	0.051 +0.051 +0.075 +0.079 +0.10 +0.14	250	6	1 3	<u>1.7</u> 0.42	<u>0.23</u> 0.049	2.2 0.56	3.8 1.0	RES96008 VA
CO, 1996 (Melody F1 RS)	SC 480	0.049 +0.049 +0.075 +0.075 +0.10 +0.15	280	6	1 3	<u>4.0</u> 0.29	<u>0.53</u> 0.050	4.8 0.38	6.6 0.48	RES96008 CO

Location, year (variety)	Application				PHI, days	Spinosyn residues, mg/kg				Ref.
	Form	kg ai/ha	water, l/ha	no.		A HPLC	D HPLC	Total <sup>1</sup> HPLC	IA <sup>2</sup>	
CELERY										
FL, 1996 (M68-29-5) untrimmed	SC 480	0.051 +0.051 +0.075 +0.074 +0.10 +0.15	280	6	0 1 3 5	1.3	0.19	1.5	1.7 <u>1.1</u> 0.89 0.30	RES96008 FL
FL, 1996 (M68-29-5) trimmed	SC 480	0.051 +0.051 +0.075 +0.074 +0.10 +0.15	280	6	0 1 3 5	0.15	0.02	0.19	0.21 0.18 0.083 0.084	RES96008 FL
MI, 1996 (Florida 683K) untrimmed	SC 480	0.050 +0.050 +0.076 +0.075 +0.10 +0.15	280	6	1 3				<u>1.3</u> 0.19	RES96008 MI
MI, 1996 (Florida 683K) trimmed	SC 480	0.050 +0.050 +0.076 +0.075 +0.10 +0.15	280	6	1 3				0.31 0.14	RES96008 MI
CA, 1996 (T&A Special #1) untrimmed	SC 480	0.051 +0.051 +0.078 +0.075 +0.10 +0.15	320	6	1 3	<u>0.39</u> 0.27	<u>0.064</u> 0.042	0.48 0.32	0.78 0.63	RES96008 CA1
CA, 1996 (T&A Special #1) trimmed	SC 480	0.051 +0.051 +0.078 +0.075 +0.10 +0.15	320	6	1 3	0.034 0.067	<0.01 0.01	0.04 0.08	0.056 0.089	RES96008 CA1
CA, 1996 (5270R) untrimmed	SC 480	0.050 +0.054 +0.079 +0.073 +0.10 +0.13	350 +370 +380 +370 +400 +470	6	1 3				<u>0.84</u> 0.59	RES96008 CA2
CA, 1996 (5270R) trimmed	SC 480	0.050 +0.054 +0.079 +0.073 +0.10 +0.13	350 +370 +380 +370 +400 +470	6	1 3				0.087 0.097	RES96008 CA2

Location, year (variety)	Application				PHI, days	Spinosyn residues, mg/kg				Ref.
	Form	kg ai/ha	water, l/ha	no.		A HPLC	D HPLC	Total <sup>1</sup> HPLC	IA <sup>2</sup>	
CA, 1996 (Summit) untrimmed	SC 480	0.053 +0.049 +0.078 +0.075 +0.098 +0.14	450	6	1 3				<u>0.40</u> 0.18	RES96008 CA3
CA, 1996 (Summit) trimmed	SC 480	0.053 +0.049 +0.078 +0.075 +0.098 +0.14	450	6	1 3				0.11 0.096	RES96008 CA3
AZ, 1996 (Conquistador) untrimmed	SC 480	0.054 +0.055 +0.078 +0.079 +0.11 +0.16	290	6	1 3				<u>1.7</u> 1.2	RES96008 AZ
AZ, 1996 (Conquistador) trimmed	SC 480	0.054 +0.055 +0.078 +0.079 +0.11 +0.16	290	6	1 3				0.10 0.069	RES96008 AZ

<sup>1</sup> Total includes spinosyns A, D, B, N-demethyl-D and K. Results adjusted for procedural recoveries.

<sup>2</sup> HPLC only on selected samples as confirmation of immunoassay method. Results for HPLC are for single, and for immunoassay means of duplicate field samples.

Table 59. Spinosad residues in legume vegetables from supervised trials in the USA. Double-underlined residues are from treatments according to GAP and are valid for the estimation of maximum residue levels.

Location, year (variety)	Application				PHI, days	Residues, mg/kg spinosad	Ref.
	Form	kg ai/ha	water, l/ha	no.			
SNAP BEANS (seed and pod)							
CA, 1997 (Strike)	SC 240	0.060 +4x0.080 +0.10	290	6	3	<u>0.14</u>	RES97034 CA1
CA, 1997 (Roma)	SC 240	5x0.080 +0.10	410	6	3	<u>0.20</u>	RES97034 CA2
FL, 1997 (Harvester)	SC 240	6x0.080	480	6	3	<u>0.02</u>	RES97034 FL1
FL, 1997 (Harvester)	SC 240	0.060 +4x0.080 +0.10	470	6	3	<u>0.15</u>	RES97034 FL2



Location, year (variety)	Application				PHI, days	Residues, mg/kg spinosad	Ref.
	Form	kg ai/ha	water, l/ha	no.			
IN, 1997 (Blue lake 274)	SC 240	2x0.062 +3x0.080 +0.10	130	6	3	<u>0.02</u>	RES97034
MI, 1997 (Envy)	SC 240	5x0.080 +0.10	190	6	3	<u>0.042</u>	RES97034
NJ, 1997 (Florence)	SC 240	0.080 +4x0.060 +0.10	300	6	3	<u>0.085</u>	RES97034
OH, 1997 (Bush Tenderpod)	SC 240	5x0.080 +0.10	200	6	3	<u>0.077</u>	RES97034
PA, 1997 (Roma II)	SC 240	3x0.080 +2x0.060 +0.080	180	6	3	<u>&lt;0.01</u>	RES97034
WA, 1997 (Labrador)	SC 240	5x0.080 +0.10	280	6	3	<u>0.02</u>	RES97034
WI, 1997 (Florence)	SC 240	5x0.080 +0.10	180	6	3	<u>&lt;0.01</u>	RES97034
SNOW PEA (seed and pod)							
MI, 1997 (Oregon Sugar)	SC 240	5x0.080 +0.10	190	6	3	<u>0.20</u>	RES97034
OH, 1997 (Oregon Sugar)	SC 240	3x0.060 +2x0.080 +0.10	210	6	3	<u>0.21</u>	RES97034
OR, 1997 (Green Arrow)	SC 240	3x0.060 +2x0.080 +0.10	190	6	3	<u>0.063</u>	RES97034
PA, 1997 (Dwarf White Sugar)	SC 240	5x0.080 +0.10	190	6	3	<u>0.03</u>	RES97034
WA, 1997 (Perfection)	SC 240	2x0.080 +3x0.060 +0.080	280	6	3	<u>0.039</u>	RES97034
WI, 1997 (Oregon Sugar Pod II)	SC 240	3x0.080 +2x0.060 +0.080	190	6	3	<u>&lt;0.01</u>	RES97034
WI, 1997 (Super Sugar Mel)	SC 240	5x0.080 +0.10	180	6	3	<u>0.01</u>	RES97034

Table 60. Spinosad residues in navy beans stored in a freezer for approximately 28 months in supervised trials in Australia. .

Location, year (variety)	Application			PHI, days	Residues, mg/kg spinosad	Ref.
	Form	kg ai/ha	no.			
Qld, 1995 (Kerman)	SC480	0.096	2	14	<0.01	GHF-P 1672 Walkamin
Qld, 1995 (Kerman)	SC480	0.19	2	14	<0.01	GHF-P 1672 Walkamin
Qld, 1995 (Kerman)	SC480	0.096	2	14	<0.01	GHF-P 1672 Mareeba
Qld, 1995 (Kerman)	SC480	0.19	2	14	<0.01	GHF-P 1672 Mareeba

Table 61. Spinosad residues in soya beans from supervised trials in the USA. Double-underlined residues are from treatments according to GAP and are valid for the estimation of maximum residue levels.

Location, year (variety)	Application				PHI, days	Residues, mg/kg spinosad	Ref.
	Form	kg ai/ha	water, l/ha	no.			
IA, 1997 (Pioneer 9294)	SC 480	0.38	190	3	28	< <u>0.01</u>	RES97034
IL, 1997 (Asgrow AG4401)	SC 480	0.38	150	3	28	< <u>0.01</u>	RES97034
IN, 1997 (Pioneer 9273)	SC 480	0.38	190	3	28	< <u>0.01</u>	RES97034
MO, 1997 (Pioneer 9363)	SC 480	0.38	160	3	28	< <u>0.01</u>	RES97034
MS, 1997 (DP3588)	SC 480	0.38	230	3	28	< <u>0.01</u>	RES97034
NC, 1997 (Hartz H5566)	SC 480	0.38	330	3	28	< <u>0.01</u>	RES97034
WI, 1997 (DeKalb CX 173)	SC 480	0.38	190	3	28	< <u>0.01</u>	RES97034

Table 62. Spinosad residues in soya beans from supervised trials in Argentina and Brazil. Double-underlined residues are from treatments according to GAP and are valid for the estimation of maximum residue levels.

Country, year (variety)	Application				PHI, days	Residues, mg/kg		Ref.
	Form	kg ai/ha	water, l/ha	no.		spinosyn A	spinosyn D	
Argentina, 1995 (Asgrow 5308)	SC 480	0.050	100	1	75 <sub>1</sub>	<0.01 (3)	<0.01 (3)	GHB P-344
Argentina, 1995 (Asgrow 5308)	SC 480	0.10	100	1	75 <sub>1</sub>	<0.01 (3)	<0.01 (3)	GHB P-344

Country, year (variety)	Application				PHI, days	Residues, mg/kg		Ref.
	Form	kg ai/ha	water, l/ha	no.		spinosyn A	spinosyn D	
Argentina, 1995 (Bataca 54)	SC 480	0.050	120	1	70 <sub>1</sub>	<0.01 (3)	<0.01 (3)	GHB P-344
Argentina, 1995 (Bataca 54)	SC 480	0.10	120	1	70 <sub>1</sub>	<0.01 (3)	<0.01 (3)	GHB P-344
Argentina, 1995 (DK 458)	SC 480	0.050	160	1	61 <sub>1</sub>	<0.01 (3)	<0.01 (3)	GHB P-344
Argentina, 1995 (DK 458)	SC 480	0.10	160	1	61 <sub>1</sub>	<0.01 (3)	<0.01 (3)	GHB P-344
Argentina, 1996 (Asgrow 4422)	SC 480	0.050	100	1	86	<0.01 (3)	<0.01 (3)	GHB P-344
Argentina, 1996 (Asgrow 4422)	SC 480	0.10	100	1	86	<0.01 (3)	<0.01 (3)	GHB P-344
Argentina, 1996 (Asgrow 4422)	SC 480	0.050	100	1	61	<0.01 (3)	<0.01 (3)	GHB P-344
Argentina, 1996 (Asgrow 4422)	SC 480	0.10	100	1	61	<0.01 (3)	<0.01 (3)	GHB P-344
Argentina, 1996 (Asgrow 5401)	SC 480	0.050	110	1	98	<0.01 (3)	<0.01 (3)	GHB P-344
Argentina, 1996 (Asgrow 5401)	SC 480	0.10	110	1	98	<0.01 (3)	<0.01 (3)	GHB P-344
Brazil (Paraná), 1995 (BR-4)	WG 800	0.048	100	2	19	<0.01 (3)	<0.01 (3)	GHB P-296
Brazil (Paraná), 1995 (BR-4)	WG 800	0.096	100	2	19	<0.01 (3)	<0.01 (3)	GHB P-296
Brazil (Paraná), 1995 (BR-4)	SC 480	0.048	100	2	19	<0.01 (3)	<0.01 (3)	GHB P-296
Brazil (Paraná), 1995 (BR-4)	SC 480	0.096	100	2	19	<0.01 (3)	<0.01 (3)	GHB P-296
Brazil (São Paulo), 1994 (Garimpo)	SC 480	0.048	100	2	0 9 20 <sub>1</sub>	<0.01 (3) <0.01 (3) <0.01 (3)	<0.01 (3) <0.01 (3) <0.01 (3)	GHB P-296
Brazil (São Paulo), 1994 (Garimpo)	SC 480	0.096	100	2	0 9 20 <sub>1</sub>	<0.01 (3) <0.01 (3) <0.01 (3)	<0.01 (3) <0.01 (3) <0.01 (3)	GHB P-296

<sup>1</sup> Samples stored in a freezer for 19-20 months.

Table 63. Spinosad residues in potatoes from supervised trials in Brazil and the USA. Double-underlined residues are from treatments according to GAP and are valid for the estimation of maximum residue levels.

country, year (variety)	Application				PHI, days	Residues, mg/kg			Ref.
	Form	kg ai/ha	water, l/ha	no.		spinosyn A	spinosyn D	IA <sup>1</sup>	
Brazil, 1995 (Achat)	WG 800	0.20	400	4	15	<0.01 (3)	<0.01 (3)		GHB P-295
Brazil, 1995 (Achat)	WG 800	0.40	400	4	15	<0.01 (3)	<0.01 (3)		GHB P-295
Brazil (São Paulo), 1996 (Achat)	SC 480	0.20	400	5	0	<0.01	<0.01		GHB P-346
					1	<0.01	<0.01		
					3	<u>&lt;0.01</u>	<0.01		
					5	<0.01	<0.01		
					10	<0.01	<0.01		
Brazil (São Paulo), 1996 (Achat)	SC 480	0.40	400	5	0	<0.01	<0.01		GHB P-346
					1	<0.01	<0.01		
					3	<u>&lt;0.01</u>	<0.01		
					5	<0.01	<0.01		
					10	<0.01	<0.01		
Brazil (Paraná), 1996 (Elvira)	SC 480	0.20	300	5	0	<0.01	<0.01		GHB P-346
					1	<0.01	<0.01		
					3	<u>&lt;0.01</u>	<0.01		
					5	<0.01	<0.01		
					10	<0.01	<0.01		
Brazil (Paraná), 1996 (Elvira)	SC 480	0.40	300	5	0	<0.01	<0.01		GHB P-346
					1	<0.01	<0.01		
					3	<u>&lt;0.01</u>	<0.01		
					5	<0.01	<0.01		
					10	<0.01	<0.01		
USA (ME), 1997 (FL-1533)	SC 230	0.12	330	3	7			< <u>0.005</u>	IR-4 06653 97-ME03
USA (NJ), 1997 (Superior)	SC 230	0.12	410	3	7			< <u>0.005</u>	IR-4 06653 97-NJ29
USA (NJ), 1997 (Superior)	SC 230	0.62	410	3	7			< <u>0.005</u>	IR-4 06653 97-NJ29
USA (ID), 1997 (Russet Burbank)	SC 230	0.12	190	3	8			< <u>0.005</u>	IR-4 06653 97-ID13
USA (ID), 1997 (Russet Burbank)	SC 230	0.62	190	3	8			< <u>0.005</u>	IR-4 06653 97-ID13
USA (ID), 1997 (Russet Burbank)	SC 230	0.12	190	3	8			< <u>0.005</u>	IR-4 06653 97-ID14
USA (ID), 1997 (Russet Burbank)	SC 230	0.12	190	3	8			< <u>0.005</u>	IR-4 06653 97-ID15
USA (WI), 1997 (Superior)	SC 230	0.12	190	3	8			< <u>0.005</u>	IR-4 06653 97-WI18
USA (WI), 1997 (Russet Burbank)	SC 230	0.12	240	3	7			< <u>0.005</u>	IR-4 06653 97-WI19

<sup>1</sup> Immunoassay

Laboratory	Application				PHI, days	Spinosyn residues, mg/kg						Ref.
	Form	kg ai/hl	water, l/ha	no.		A	D	K	B	B of D	Total	
<b>Roots</b>												
Institute of Environmental Toxicology	WG 250	0.01	3000	3	7 15 22 31	<0.01 <0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01	GHF-P- 1488 Chiba
Japan Chemical Analysis Consultant					7 15 22 31	<0.01 <0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01	GHF-P- 1488 Chiba
Institute of Environmental Toxicology	WG 250	0.01	3000	3	7 14 21 30	<0.01 <u>0.01</u> <0.01 <0.01	<0.01 <0.01 <0.01 <0.01	0.01 <0.01 <0.01 <0.01	0.01 <0.01 <0.01 <0.01	0.01 <0.01 <0.01 <0.01	<u>0.03</u> 0.01 <0.01 <0.01	GHF-P- 1488 Niigata
Japan Chemical Analysis Consultant					7 14 21 30	<0.01 <0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01	GHF-P- 1488 Niigata

Laboratory	Application				PHI, days	Spinosyn residues, mg/kg						Ref.
	Form	kg ai/hl	water, l/ha	no.		A	D	K	B	B of D	Total	
Leaves												
Institute of Environmental Toxicology	WG 250	0.01	3000	3	7	<u>0.06</u>	<u>0.01</u>	0.01	<0.01	<0.01	0.08	GHF-P-1488 Chiba
					15	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	
					22	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	
					31	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	
Japan Chemical Analysis Consultant					7	0.04	<0.01	<0.01	<0.01	<0.01	0.04	GHF-P-1488 Chiba
					15	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	
					22	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	
					31	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	
Institute of Environmental Toxicology	WG 250	0.01	3000	3	7	<u>0.20</u>	<u>0.03</u>	0.02	0.02	<0.01	0.27	GHF-P-1488 Niigata
					14	0.02	<0.01	0.01	0.01	<0.01	0.04	
					21	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	
					30	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	
Japan Chemical Analysis Consultant					7	0.12	0.02	0.02	0.02	<0.01	0.18	GHF-P-1488 Niigata
					14	0.02	<0.01	0.01	<0.01	<0.01	0.03	
					21	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	
					30	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	

Table 65. Spinosad residues in cereals measured by an immunoassay method from supervised trials in the USA. Double-underlined residues are from treatments according to GAP and are valid for the estimation of maximum residue levels.

Location, year (variety)	Application				PHI, days	Residues, mg/kg	Ref.
	Form	kg ai/ha	water, l/ha	no.			
MAIZE							
MN, 1997 (Pioneer 3751)	SC 480	0.50	200	2	27	< <u>0.01</u>	RES97037
NE, 1997 (Pioneer 3751)	SC 480	0.50	260	2	30	< <u>0.01</u>	RES97037
NC, 1997 (Pioneer 3394)	SC 480	0.50	190	2	30	< <u>0.01</u>	RES97037
OH, 1997 (GL 276)	SC 480	0.50	210	2	28	< <u>0.01</u> <0.01 (HPLC)	RES97037
PA, 1997 (DK 385B)	SC 480	0.50	190	2	28	< <u>0.01</u>	RES97037
SORGHUM							
CO, 1997 (Cargill 577)	SC 480	0.10	250	5	7	<u>0.68</u> 0.62 (HPLC)	RES97037
KS, 1997 (DK 47)	SC 480	0.10	210	5	7	<u>0.088</u>	RES97037
MO, 1997 (Pioneer 8305)	SC 480	0.10	220	5	7	<u>0.030</u>	RES97037

Location, year (variety)	Application				PHI, days	Residues, mg/kg	Ref.
	Form	kg ai/ha	water, l/ha	no.			
MS, 1997 (G-522DR, Mycogen Hybrid)	SC 480	0.10	270	5	0 9 14 21 0 9	0.85 0.086 0.099 0.037 c 0.90 <sup>1</sup> c 0.02	RES97037
NE, 1997 (Northrup King NK1210)	SC 480	0.10	190	5	7	<u>0.47</u>	RES97037 NE1
NE, 1997 (Northrup King NK1210)	SC 480	0.10	190	5	8	<u>0.17</u>	RES97037 NE2
OK, 1997 (T-E Eden)	SC 480	0.10	200	5	7	<u>0.18</u>	RES97037
TX, 1997 (DPol 1558)	SC 480	0.10	250	5	7	<u>0.16</u>	RES97037 TX1
TX, 1997 (Y363)	SC 480	0.10	190	5	7	<u>0.12</u>	RES97037 TX2
WHEAT							
IL, 1997 (Clemens 8530, winter wheat)	SC 480	0.50	190	3	21	0.01	RES97037
IN, 1997 (Pioneer 2571, winter wheat)	SC 480	0.50	190	3	21	<0.01	RES97037
ND, 1997 (2375 NDSU, spring wheat)	SC 480	0.50	290	3	21	0.051 0.05 (HPLC) 0.074 cleaned grain 0.57 grain trash	RES97037
OK, 1997 (Tam 200, winter wheat)	SC 480	0.50	240	3	21	0.061	RES97037
SD, 1997 (Hard Red 2375, spring wheat)	SC 480	0.50	190	3	21	0.054	RES97037
TX, 1997 (Thunderbird, winter wheat)	SC 480	0.50	250	3	21	0.084	RES97037

<sup>1</sup> Sample from control plot apparently swapped with a treated sample. Data from this trial are not included in the evaluation.

Table 66. Spinosad residues in maize from supervised trials in Argentina and Brazil. Double-underlined residues are from treatments according to GAP and are valid for the estimation of maximum residue levels.

Country, year (variety)	Application				PHI, days	Residues, mg/kg		Ref.
	Form	kg ai/ha	water, l/ha	no.		spinosyn A	spinosyn D	
Argentina (Buenos Aires), 1999 (DK752)	SC 480	0.029		1	137	<0.01 (3)	<0.01 (3)	GHB P-448

Country, year (variety)	Application				PHI, days	Residues, mg/kg		Ref.
	Form	kg ai/ha	water, l/ha	no.		spinosyn A	spinosyn D	
Argentina (Buenos Aires), 1999 (DK752)	SC 480	0.058		1	137	<0.01 (3)	<0.01 (3)	GHB P-448
Argentina (Buenos Aires), 1999 (Tilcara)	SC 480	0.029		1	138	<0.01 (3)	<0.01 (3)	GHB P-448
Argentina (Buenos Aires), 1999 (Tilcara)	SC 480	0.058		1	138	<0.01 (3)	<0.01 (3)	GHB P-448
Argentina (La Virginia-Tucumán), 1999 (Hercules)	SC 480	0.029		1	122	<0.01 (2)	<0.01 (2)	GHB P-448
Argentina (La Virginia-Tucumán), 1999 (Hercules)	SC 480	0.058		1	122	<0.01 (3)	<0.01 (3)	GHB P-448
Brazil (Paraná), 1995 (G-85)	WG 800	0.060	250	3	0 7	<0.01 (3) <0.01 (3)	<0.01 (3) <0.01 (3)	GHB P-287
Brazil (Paraná), 1995 (G-85)	WG 800	0.12	250	3	0 7	<0.01 (3) <0.01 (3)	<0.01 (3) <0.01 (3)	GHB P-287
Brazil (Paraná), 1995 (G-85)	SC 480	0.048	250	3	0 7	<0.01 (3) <0.01 (3)	<0.01 (3) <0.01 (3)	GHB P-292
Brazil (Paraná), 1995 (G-85)	SC 480	0.096	250	3	0 7	<0.01 (3) <0.01 (3)	<0.01 (3) <0.01 (3)	GHB P-292
Brazil (São Paulo), 1995 (C-701)	WG 800	0.060	400	3	0 7 14 28 56	<0.01 (3) <0.01 (3) <0.01 (3) <0.01 (3) <0.01 (3)	<0.01 (3) <0.01 (3) <0.01 (3) <0.01 (3) <0.01 (3)	GHB P-287
Brazil (São Paulo), 1995 (C-701)	WG 800	0.120	400	3	0 7 14 28 56	<0.01 (3) <0.01 (3) <0.01 (3) <0.01 (3) <0.01 (3)	<0.01 (3) <0.01 (3) <0.01 (3) <0.01 (3) <0.01 (3)	GHB P-287
Brazil (São Paulo), 1995 (C-701)	SC 480	0.048	400	3	0 7 14 28 56	<0.01 (3) <0.01 (3) <0.01 (3) <0.01 (3) <0.01 (3)	<0.01 (3) <0.01 (3) <0.01 (3) <0.01 (3) <0.01 (3)	GHB P-292
Brazil (São Paulo), 1995 (C-701)	SC 480	0.096	400	3	0 7 14 28 56	<0.01 (3) <0.01 (3) <0.01 (3) <0.01 (3) <0.01 (3)	<0.01 (3) <0.01 (3) <0.01 (3) <0.01 (3) <0.01 (3)	GHB P-292



Table 67. Spinosad residues in almonds from supervised trials in California, USA, in 1996. Double-underlined residues are from treatments according to GAP and are valid for the estimation of maximum residue levels.

[illegible]

Application				PHI, days	Spinosyn residues, mg/kg						Ref.
Form	kg ai/ha	water, l/ha	no.		A	D	K	B	B of D	Total	
SC 450	0.070 +0.10 +0.15 +0.18	1900	4	14	<u>&lt;0.01</u>	<0.01	<0.01	<0.01	<0.01	<0.01	RES96004 CA6

Table 68. Spinosad residues in cotton seed from supervised trials in Argentina, Australia, Brazil and the USA. Double-underlined residues are from treatments according to GAP and are valid for the estimation of maximum residue levels.

Country, year (variety)	Application					PHI, days	Residues, mg/kg		Ref.
	Form	kg ai/ha	kg ai/hl	water, l/ha	no.		spinosyn A	spinosyn D	
Argentina (Salta), 1996 (Guazuncho 2)	SC 480	0.05		90	4	71	<0.01(3)	<0.01(3)	GHB-P 358
Argentina (Salta), 1996 (Guazuncho 2)	SC 480	0.10		90	4	71	<0.01(3)	<0.01(3)	GHB-P 358
Argentina (Salta), 1997 (Guazuncho 2)	SC 480	0.05		90	4	65	<0.01(3)	<0.01(3)	GHB-P 358
Argentina (Salta), 1997 (Guazuncho 2)	SC 480	0.10		90	4	65	<0.01(3)	<0.01(3)	GHB-P 358
Argentina (Santiago del Estero), 1996 (Guazuncho inta)	SC 480	0.05		108	4	41	<0.01(3)	<0.01(3)	GHB-P 358
Argentina (Santiago del Estero), 1996 (Guazuncho inta)	SC 480	0.10		108	4	41	<0.01(3)	<0.01(3)	GHB-P 358
Argentina (Santiago del Estero), 1997 (Guazuncho 2)	SC 480	0.05		190	4	41	<0.01(3)	<0.01(3)	GHB-P 358
Argentina (Santiago del Estero), 1997 (Guazuncho 2)	SC 480	0.10		190	4	41	<0.01(3)	<0.01(3)	GHB-P 358
Australia (NSW), 1994 (Deltapine 90)	UL 125	0.075		50	2	28	<0.01	<0.01	GHF-P 1371 93200
Australia (NSW), 1994 (Deltapine 90)	UL 125	0.15		50	2	28	<u>&lt;0.01</u>	<0.01	GHF-P 1371 93200
Australia (NSW), 1994 (Deltapine 90)	UL 125	0.075	<sup>1</sup>	4	2	28	<0.01	<0.01	GHF-P 1371 93200
Australia (NSW), 1994 (Deltapine 90)	UL 125	0.15	<sup>1</sup>	4	2	28	<u>&lt;0.01</u>	<0.01	GHF-P 1371 93200
Australia (NSW), 1996 (CS8S)	SC 125	0.10			3	28	<u>&lt;0.01</u>	<0.01	GHF-P 1628

Country, year (variety)	Application					PHI, days	Residues, mg/kg		Ref.
	Form	kg ai/ha	kg ai/hl	water, l/ha	no.		spinosyn A	spinosyn D	
Australia (NSW), 1996 (CS8S)	SC 125	0.20			3	28	<u>0.01</u>	<0.01	GHF-P 1628
Australia (Qld), 1996 (65)	SC 125	0.10			3	28	<u>0.01</u>	<0.01	GHF-P 1628
Australia (Qld), 1996 (65)	SC 125	0.20			3	28	< <u>0.01</u>	<0.01	GHF-P 1628
Australia (Qld), 1996 (L22)	SC 480	0.10			3	28	< <u>0.01</u> <sup>2</sup>	<0.01	GHF-P 1629 Emerald
Australia (Qld), 1996 (L22)	SC 125	0.10			3	28	< <u>0.01</u> <sup>2</sup>	<0.01	GHF-P 1629 Emerald
Australia (Qld), 1996 (L22)	SC 125	0.20			3	28	< <u>0.01</u> <sup>2</sup>	<0.01	GHF-P 1629 Emerald
Australia (Qld), 1996 (SK 1-4)	SC 480	0.10			3	28	< <u>0.01</u>	<0.01	GHF-P 1629 Gatton
Australia (Qld), 1996 (SK 1-4)	SC 125	0.10			3	28	< <u>0.01</u>	<0.01	GHF-P 1629 Gatton
Australia (Qld), 1996 (SK 1-4)	SC 125	0.20			3	28	< <u>0.01</u>	<0.01	GHF-P 1629 Gatton
Brazil (PR), 1994 (IAC-20)	SC 480	0.072		100	4	25	<0.01	<0.01	GHB-P 279
Brazil (PR), 1994 (IAC-20)	SC 480	0.14		100	4	25	<0.01	<0.01	GHB-P 279
Brazil (SP), 1994 (IAC-20)	SC 480	0.072		100	4	0 3 7 14 21 0	0.01 <0.01 < <u>0.01</u> <0.01 <0.01 c 0.01	<0.01 <0.01 <0.01 <0.01 <0.01 c <0.01	GHB-P 279
Brazil (SP), 1994 (IAC-20)	SC 480	0.14		100	4	0 3 7 14 21 0	0.02 <0.01 < <u>0.01</u> <0.01 <0.01 c 0.01	<0.01 <0.01 <0.01 <0.01 <0.01 c <0.01	GHB-P 279
USA (AR), 1993 (Deltapine 20)	SC 480	0.076 +0.097 +0.101 +0.101 +0.125		130 +140 +150 +110 +140	5	27	< <u>0.01</u>	<0.01	RES93026R/ RES92024R
USA (AR), 1993 (Stoneville 453)	SC 480	0.076 +0.101 +0.101 +0.101 +0.126		130 +140 +150 +110 +140	5	27	< <u>0.01</u>	<0.01	RES93026R/ RES92024R

Country, year (variety)	Application					PHI, days	Residues, mg/kg		Ref.
	Form	kg ai/ha	kg ai/hl	water, l/ha	no.		spinosyn A	spinosyn D	
USA (AZ), 1993 (Deltapine 20)	SC 480	0.074 +0.101 +0.102 +0.102 +0.125		140	5	23	<0.01	<0.01	RES93026R/ RES92024R
USA (AZ), 1993 (Deltapine 50)	SC 480	0.073 +0.099 +0.099 +0.097 +0.124		140	5	28	<0.01	<0.01	RES93026R/ RES92024R
USA (CA), 1992 (Acala SJ-2)	SC 480	0.20		280	5	14 28	<0.01 <0.01	<0.01 <0.01	RES93026R/ RES92024R
USA (CA), 1993 (Maxxa)	SC 480	0.079 +0.100 +0.099 +0.099 +0.127		200 +190 +190 +190 +190	5	28	<0.01	<0.01	RES93026R/ RES92024R
USA (CA), 1993 (Maxxa)	SC 480	0.47 +0.61 +0.60 +0.60 +0.76			5	28	<0.01	<0.01	RES93026R/ RES92024R
USA (CA), 1993 (Maxxa)	SC 480	0.076 +0.102 +0.101 +0.102 +0.126		190	5	28	<0.01	<0.01	RES93026R/ RES92024R
USA (CA), 1993 (Maxxa)	SC 480	0.076 +0.101 +0.100 +0.100 +0.124		190	5	28	<0.01	<0.01	RES93026R/ RES92024R
USA (GA), 1993 (DP 5415)	SC 480	0.076 +0.101 +0.101 +0.101 +0.126		190	5	28	<0.01	<0.01	RES93026R/ RES92024R
USA (LA), 1993 (Deltapine 20)	SC 480	0.076 +0.101 +0.102 +0.101 +0.12		150 +150 +150 +130 +120	5	27	<0.01	<0.01	RES93026R/ RES92024R
USA (MS), 1992 (Deltapine 50)	SC 480	0.20		120	5	14 28	<0.01 <0.01	<0.01 <0.01	RES93026R/ RES92024R
USA (MS), 1993 (Des119 Sure Grow)	SC 480	0.076 +0.099 +0.099 +0.099 +0.14		160 +100 +200 +200 +170	5	28	<0.01	<0.01	RES93026R/ RES92024R

Country, year (variety)	Application					PHI, days	Residues, mg/kg		Ref.
	Form	kg ai/ha	kg ai/hl	water, l/ha	no.		spinosyn A	spinosyn D	
USA (NC), 1993 (Deltapine 50)	SC 480	0.074 +0.100 +0.100 +0.100		190 +180 +180 +180	4	28	<u>&lt;0.01</u>	<0.01	RES93026R/ RES92024R
USA (OK), 1993 (Cascot C-13)	SC 480	0.075 +0.100 +0.100 +0.100 +0.125		140 +130 +140 +130 +150	5	28	<u>&lt;0.01</u>	<0.01	RES93026R/ RES92024R
USA (TX), 1993 (Deltapine 50)	SC 480	0.075 +0.100 +0.100 +0.101 +0.126		170	5	28	<u>&lt;0.01</u>	<0.01	RES93026R/ RES92024R
USA (TX), 1993 (Deltapine 51)	SC 480	0.075 +0.101 +0.099 +0.099 +0.127		170	5	28	<u>&lt;0.01</u>	<0.01	RES93026R/ RES92024R
USA (TX), 1993 (DP 5415)	SC 480	0.076 +0.099 +0.100 +0.097 +0.124		140	5	28	<u>&lt;0.01</u>	<0.01	RES93026R/ RES92024R
USA (TX), 1993 (HS- 200)	SC 480	0.075 +0.101 +0.100 +0.101 +0.125		190	5	28	<u>&lt;0.01</u>	<0.01	RES93026R/ RES92024R
USA (TX), 1993 (Southland M1)	SC 480	0.075 +0.100 +0.100 +0.100 +0.125		190	5	28	<u>&lt;0.01</u>	<0.01	RES93026R/ RES92024R

<sup>1</sup> Spinning disc applicator; diluent was a crop oil, not water.

<sup>2</sup> Samples in stored in freezer for approx. 14 months

Table 69. Spinosad residues in cereal forage and fodder from supervised trials in the USA in 1997. Residues were measured by an immunoassay method. Double-underlined residues are from treatments according to GAP and are valid for the estimation of maximum residue levels.

Location, year (variety)	Application				PHI, days	% moisture	mg/kg	Ref.
	Form	kg ai/ha	water, l/ha	no.				
SWEET CORN FORAGE								
CA (Legend)	SC 240	0.10	190	5	7	79	<u>0.48</u>	RES97037
FL (Silver Queen)	SC 240	0.10	280	5	7		<u>0.12</u> c 0.02	RES97037

Location, year (variety)	Application				PHI, days	% moisture	mg/kg	Ref.
	Form	kg ai/ha	water, l/ha	no.				
IL (Sensor)	SC 240	0.10	280	5	7	80	<u>0.099</u>	RES97037
MI (Sweet Chorus)	SC 240	0.10	230	5	7	86	<u>0.17</u>	RES97037
MN (Quickie Hybrid)	SC 240	0.10	180	5	7	84	<u>0.49</u>	RES97037
NC (Silver Queen)	SC 240	0.10	240	5	7	75	<u>0.18</u>	RES97037
NY (Tuxedo)	SC 240	0.10	180	5	7	85	<u>0.074</u>	RES97037
OH (Silver Queen)	SC 240	0.10	220	5	7	72	<u>0.36</u>	RES97037
OR (Jubilee)	SC 240	0.10	290	5	7	87	<u>0.087</u>	RES97037
PA (Breeders Bi-Color)	SC 240	0.10	230	5	7	76	<u>0.098</u>	RES97037
WA (Silver Sweet Jubilee)	SC 240	0.10	290	5	7	70	<u>0.16</u>	RES97037
WI (Confection SH2 Bi-color)	SC 240	0.10	180	5	7	84	<u>0.44</u>	RES97037
SWEET CORN STOVER								
CA (Legend)	SC 240	0.10	190	5	28	68	<u>0.68</u>	RES97037
FL (Silver Queen)	SC 240	0.10	280	5	28	65	<u>0.074</u>	RES97037
IL (Sensor)	SC 240	0.10	280	5	28	76	<u>0.099</u>	RES97037
MI (Sweet Chorus)	SC 240	0.10	230	5	28	77	<u>0.03</u>	RES97037
MN (Quickie Hybrid)	SC 240	0.10	180	5	28	72	<u>0.17</u>	RES97037
NC (Silver Queen)	SC 240	0.10	240	5	28	77	<u>0.12</u>	RES97037
NY (Tuxedo)	SC 240	0.10	180	5	28	72	<u>0.17</u>	RES97037
OH (Silver Queen)	SC 240	0.10	220	5	28	75	<u>0.23</u>	RES97037
OR (Jubilee)	SC 240	0.10	290	5	28	65	<u>0.053</u>	RES97037
PA (Breeders Bi-Color)	SC 240	0.10	230	5	28	71	<u>0.11</u>	RES97037
WA (Silver Sweet Jubilee)	SC 240	0.10	290	5	28	43	<u>0.46</u>	RES97037
WI (Confection SH2 Bi-color)	SC 240	0.10	180	5	28	66	<u>0.097</u>	RES97037
SORGHUM FORAGE								
KS (DK 47)	SC 480	0.10	210	5	14	68	<u>0.078</u>	RES97037
MS (G-522DR, Mycogen Hybrid)	SC 480	0.10	270	5	0 3 7 14 14	66	0.68 1.1 0.11 0.084 c 0.04 <sup>1</sup>	RES97037

Location, year (variety)	Application				PHI, days	% moisture	mg/kg	Ref.
	Form	kg ai/ha	water, l/ha	no.				
NE (Northrup King NK1210)	SC 480	0.10	190	5	14	66	<u>0.095</u>	RES97037 NE2
OK (T-E Eden)	SC 480	0.10	200	5	14	71	<u>0.052</u>	RES97037
TX (Y363)	SC 480	0.10	190	5	14	62	<u>0.18</u>	RES97037 TX2
SORGHUM STOVER								
KS (DK 47)	SC 480	0.10	210	5	15	64	0.060	RES97037
MS (G-522DR, Mycogen Hybrid)	SC 480	0.10	270	5	0 9 14 21	68	1.6 0.11 0.068 0.17	RES97037
NE (Northrup King NK1210)	SC 480	0.10	190	5	14	75	0.11	RES97037 NE2
OK (T-E Eden)	SC 480	0.10	200	5	14	67	0.27	RES97037
TX (Y363)	SC 480	0.10	190	5	14	67	0.097	RES97037 TX2
WHEAT FORAGE								
IL (Clemens 8530, winter wheat)	SC 480	0.10	190	1	14	76	0.054	RES97037
IN (Pioneer 2571, winter wheat)	SC 480	0.10	190	1	14	83	<0.01	RES97037
ND (2375 NDSU, spring wheat)	SC 480	0.10	290	1	14	83	<0.01	RES97037
OK (Tam 200, winter wheat)	SC 480	0.10	240	1	14	73	0.01	RES97037
SD (Hard Red 2375, spring wheat)	SC 480	0.10	190	1	14	82	0.01	RES97037
TX (Thunderbird, winter wheat)	SC 480	0.10	250	1	14	77	0.050	RES97037
WHEAT HAY								
IL (Clemens 8530, winter wheat)	SC 480	0.10	190	2	14	15	<u>0.050</u>	RES97037
IN (Pioneer 2571, winter wheat)	SC 480	0.10	190	2	14	53	< <u>0.01</u>	RES97037
ND (2375 NDSU, spring wheat)	SC 480	0.10	290	2	14	29	<u>0.019</u>	RES97037
OK (Tam 200, winter wheat)	SC 480	0.10	240	2	14	60	<u>0.052</u>	RES97037
SD (Hard Red 2375, spring wheat)	SC 480	0.10	190	2	14	30	<u>0.15</u>	RES97037
TX (Thunderbird, winter wheat)	SC 480	0.10	250	2	14	36	<u>0.17</u>	RES97037
WHEAT STRAW								
IL (Clemens 8530, winter wheat)	SC 480	0.10	190	3	21	15	<u>0.19</u>	RES97037

Location, year (variety)	Application				PHI, days	% moisture	mg/kg	Ref.
	Form	kg ai/ha	water, l/ha	no.				
IN (Pioneer 2571, winter wheat)	SC 480	0.10	190	3	21	48	<0.01	RES97037
ND (2375 NDSU, spring wheat)	SC 480	0.10	290	3	21	12	0.73	RES97037
OK (Tam 200, winter wheat)	SC 480	0.10	240	3	21	10	0.53	RES97037
SD (Hard Red 2375, spring wheat)	SC 480	0.10	190	3	21	13	0.56	RES97037
TX (Thunderbird, winter wheat)	SC 480	0.10	250	3	21	10	0.37	RES97037

<sup>1</sup> Contaminated control sample; data from this trial not included in the evaluation.

Table 70. Spinosad residues in cotton trash from supervised trials in Australia. <sup>1</sup>

Location, year (variety)	Application			PHI, days	Residues, mg/kg <sup>2</sup>			Ref.
	Form	kg ai/ha	no.		spinosyn A	spinosyn D	Total	
NSW, 1996 (CS8S)	SC 125	0.10	3	0	2.9 (11.2)	0.46 (1.8)	3.3 (13)	GHF-P 1628
				8	0.32 (1.2)	0.04 (0.13)	0.36 (1.3)	
				14	0.56 (2.0)	0.05 (0.18)	0.61 (2.2)	
				28	0.11 (0.13)	0.01 (0.01)	0.12 (0.14)	
NSW, 1996 (CS8S)	SC 125	0.20	3	0	6.2 (26)	0.90 (3.8)	7.1 (30)	GHF-P 1628
				8	0.99 (4.0)	0.10 (0.39)	1.1 (4.4)	
				14	1.2 (4.3)	0.11 (0.38)	1.3 (4.7)	
				28	0.37 (0.43)	0.04 (0.04)	0.41 (0.47)	
Qld, 1996 (65)	SC 125	0.10	3	0	12.8 (47)	2.6 (9.3)	15 (56)	GHF-P 1628
				7	2.0 (7.1)	0.31 (1.1)	2.3 (8.3)	
				14	0.95 (3.6)	0.09 (0.33)	1.0 (3.9)	
				28	0.59 (1.9)	0.04 (0.13)	0.63 (2.0)	
Qld, 1996 (65)	SC 125	0.20	3	0	27 (103)	4.3 (16)	32 (119)	GHF-P 1628
				7	4.0 (14)	0.61 (2.2)	4.6 (17)	
				14	1.7 (6.6)	0.14 (0.55)	1.8 (7.1)	
				28	1.6 (5.5)	0.08 (0.27)	1.6 (5.7)	
Qld, 1996 (L22)	SC 480	0.10	3	0	7.5 (32)	1.2 (5.0)	8.6 (37)	GHF-P 1629 Emerald
				7	0.72 (3.1)	0.09 (0.39)	0.81 (3.5)	
				14	0.23 (0.99)	0.04 (0.17)	0.27 (1.2)	
				28	0.08 (0.09)	0.01 (0.01)	0.09 (0.11)	
Qld, 1996 (SK 1-4)	SC 480	0.10	3	0	8.0 (29)	1.3 (4.7)	9.3 (34)	GHF-P 1629 Gatton
				7	0.93 (3.1)	0.15 (0.51)	1.1 (3.6)	
				14	0.16 (0.57)	0.03 (0.09)	0.18 (0.66)	
				28	0.05 (0.07)	0.01 (0.01)	0.06 (0.08)	
Qld, 1996 (L22)	SC 125	0.10	3	0	7.2 (32)	1.2 (5.3)	8.4 (37)	GHF-P 1629 Emerald
				7	0.90 (3.9)	0.11 (0.47)	1.0 (4.4)	
				14	0.54 (2.3)	0.07 (0.30)	0.61 (2.6)	
				28	0.18 (0.21)	0.03 (0.04)	0.21 (0.24)	



Location, year (variety)	Application			PHI, days	Residues, mg/kg <sup>2</sup>			Ref.
	Form	kg ai/ha	no.		spinosyn A	spinosyn D	Total	
Qld, 1996 (L22)	SC 125	0.20	3	0 7 14 28	16 (71) 1.1 (5.3) 0.50 (2.3) 0.21 (0.24)	2.5 (11) 0.13 (0.60) 0.05 (0.20) 0.04 (0.04)	18 (82) 1.3 (5.9) 0.55 (2.5) 0.24 (0.29)	GHF-P 1629 Emerald
Qld, 1996 (SK 1-4)	SC 125	0.10	3	0 7 14 28	7.2 (27) 1.2 (4.0) 0.71 (2.6) 0.04 (0.05)	1.3 (4.8) 0.19 (0.63) 0.11 (0.39) 0.01 (0.01)	8.5 (32) 1.4 (4.6) 0.81 (3.0) 0.05 (0.06)	GHF-P 1629 Gatton
Qld, 1996 (SK 1-4)	SC 125	0.20	3	0 7 14 28	11 (43) 1.6 (5.8) 1.4 (5.2) 0.08 (0.10)	2.0 (7.6) 0.27 (0.95) 0.18 (0.66) 0.01 (0.01)	13 (51) 1.9 (6.7) 1.6 (5.9) 0.09 (0.11)	GHF-P 1629 Gatton

<sup>1</sup> Gin trash is typically dried cotton foliage that has adhered to the lint during machine picking. For the 28-day samples, only dried leaves and cotton bracts that remained after the lint harvest were taken, and for earlier samples, green or partially green cotton leaves and bracts from the upper two-thirds of the plant were taken.

<sup>2</sup> Residues in parentheses are on a dry weight basis.

Table 71. Spinosad residues in navy bean forage and hay from supervised trials in Australia.

Location, year (variety)	Application				PHI, days	Commodity	Spinosad, mg/kg <sup>1</sup>	Ref.
	Form	kg ai/ha	water, l/ha	no.				
Qld, 1995 (Kerman)	SC480	0.096	230	1	14	whole plant	0.01 (0.02)	GHF-P 1672 Walkamin
Qld, 1995 (Kerman)	SC480	0.19	230	1	14	whole plant	0.02 (0.06)	GHF-P 1672 Walkamin
Qld, 1995 (Kerman)	SC480	0.096	?	1	14	whole plant	0.01 (0.05)	GHF-P 1672 Mareeba
Qld, 1995 (Kerman)	SC480	0.19	?	1	14	whole plant	0.03 (0.13)	GHF-P 1672 Mareeba
Qld, 1995 (Kerman)	SC480	0.096	230	2	14	hay	0.02 (0.02)	GHF-P 1672 Walkamin
Qld, 1995 (Kerman)	SC480	0.19	230	2	14	hay	0.6 (0.07)	GHF-P 1672 Walkamin
Qld, 1995 (Kerman)	SC480	0.096	?	2	14	hay	0.02 (0.02)	GHF-P 1672 Mareeba
Qld, 1995 (Kerman)	SC480	0.19	?	2	14	hay	0.04 (0.04)	GHF-P 1672 Mareeba

<sup>1</sup> Residues in parentheses are on a dry weight basis.

Table 72. Spinosad residues in almond hulls from supervised trials in California, USA, in 1996. Double-underlined residues are from treatments according to GAP and are valid for the estimation of maximum residue levels.

Application				PHI, days	Spinosyn residues, mg/kg						Ref.
Form	kg ai/ha	water, l/ha	no.		A	D	K	B	B of D	Total	
SC 450	0.070 +0.10 +0.15 +0.18	480	4	14	<u>0.59</u>	<u>0.080</u>	<0.01	0.02	<0.01	0.70	RES96004 CA1
SC 450	0.070 +0.10 +0.15 +0.18	1800	4	14	<u>0.64</u>	<u>0.087</u>	<0.01	0.02	<0.01	0.75	RES96004 CA1
SC 450	0.070 +0.10 +0.15 +0.18	490	4	14	<u>0.54</u>	<u>0.077</u>	<0.01	0.02	<0.01	0.64	RES96004 CA2
SC 450	0.070 +0.10 +0.15 +0.18	1900	4	14	<u>0.98</u>	<u>0.14</u>	<0.01	0.03	<0.01	1.2	RES96004 CA2
SC 450	0.070 +0.10 +0.15 +0.18	470	4	14	<u>0.43</u>	<u>0.058</u>	<0.01	0.02	<0.01	0.50	RES96004 CA3
SC 450	0.070 +0.10 +0.15 +0.18	2000	4	14	<u>0.72</u>	<u>0.098</u>	<0.01	0.030	<0.01	0.86	RES96004 CA3
SC 450	0.070 +0.10 +0.15 +0.18	470	4	14	<u>0.25</u>	<u>0.03</u>	<0.01	<0.01	<0.01	0.28	RES96004 CA4
SC 450	0.070 +0.10 +0.15 +0.18	1900	4	14	<u>0.33</u>	<u>0.04</u>	<0.01	0.01	<0.01	0.38	RES96004 CA4
SC 450	0.070 +0.10 +0.15 +0.18	470	4	0 5 14 21	0.27 0.48 <u>0.18</u> 0.17	0.037 0.055 <u>0.02</u> 0.02	<0.01 <0.01 <0.01 <0.01	0.01 0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01	0.32 0.56 0.21 0.20	RES96004 CA5
SC 450	0.070 +0.10 +0.15 +0.18	1900	4	0 5 14 21	0.34 0.49 <u>0.24</u> 0.27	0.047 0.068 <u>0.032</u> 0.038	<0.01 <0.01 <0.01 <0.01	0.01 0.02 <0.01 0.01	<0.01 <0.01 <0.01 <0.01	0.40 0.58 0.28 0.32	RES96004 CA5
SC 450	0.070 +0.10 +0.15 +0.18	470	4	14	<u>0.40</u>	<u>0.054</u>	<0.01	0.02	<0.01	0.47	RES96004 CA6

Application				PHI, days	Spinosyn residues, mg/kg						Ref.
Form	kg ai/ha	water, l/ha	no.		A	D	K	B	B of D	Total	
SC 450	0.070 +0.10 +0.15 +0.18	1900	4	14	<u>0.60</u>	<u>0.085</u>	<0.01	0.02	<0.01	0.71	RES96004 CA6

### Direct animal treatments

The Meeting received information on residues in the tissues and milk of sheep and cattle treated directly with spinosad.

**Sheep.** In two trials in Australia short-wool sheep were plunge-dipped in a solution of spinosad and long-wool sheep were sprayed with a jet (Ridley, 1999). At each withholding period (whp) 5 animals were slaughtered. Residues in the tissues are shown in Table 73. The short-wool sheep (wool length 1.6-2.6 cm) were one and a half years old and their live weight on the day of treatment was 36-46.5 kg, and the long-wool sheep (wool length 6.8-7.5 cm) were two and a half years old with live weights of 41-60 kg. The former sheep were shorn 45 days and the latter 9 months before treatment.

In the plunge dipping the sheep's heads were immersed twice, each animal remaining in the fluid for 20 seconds, and on exit taking with them 12-15 l of fluid. Animals were weighed before dipping and again after 30-60 min when they had stopped dripping, demonstrating that the average weight of dipping fluid retained was 2.6 kg.

Similarly the long-wool sheep treated for 21 seconds with a hand-held jetting applicator supported by a motor driven diaphragm pump received 5.1 l each, retaining an average weight of fluid of 1.3 kg.

Tissue samples were analysed by the immunoassay method. Recoveries ranged from 59-149%, n=30, averaging 87%, 92%, 83% and 93% for kidney, liver, muscle and fat respectively.

In a further plunge-dipping trial on short-wool Dorset Horn ewes the sheep's heads were immersed twice, each animal remaining in the fluid for 30 seconds (Ridley, 2000). On exiting the sheep each removed approximately 12-15 l of fluid. The animals were shorn 42 days before treatment. On the day of treatment, wool length was 1.5-1.9 cm and live weight 36-67 kg. Residues in the tissues are shown in Table 73.

Table 73. Spinosad residues in the tissues of sheep from supervised trials in Australia in 1999 (analyses by immunoassay method).

Location (breed)	Application			Sample	whp days	Spinosad, mg/kg	Ref.
	Form	Method	Spray or dip conc, kg ai/hl				
NSW (Merino) short wool	aq susp 25 g ai/l	plunge dip	0.001	muscle	5 12 15	<0.01 (5) <0.01 (5) <0.01 (5)	ELANCO /GLP/980 9/1-1
				kidney	5 12 15	<0.01 0.013 0.01 <0.01 0.014 <0.01 (5) <0.01 (5)	

Location (breed)	Application			Sample	whp days	Spinosad, mg/kg	Ref.
	Form	Method	Spray or dip conc, kg ai/hl				
				liver	5 12 15	<0.01 (5) <0.01 (5) <0.01 (5)	
				back fat	5 12 15 21 39 56	0.020 0.029 0.014 <0.01 (2) 0.016 0.033 0.017 0.017 0.018 0.017 0.024 0.026 0.024 0.013 0.033 0.018 <0.01 0.011 <0.01 0.023 <0.01 (4) <0.01 (5)	
				perirenal fat	5 12 15 21 39 56	0.042 0.032 0.014 0.024 0.042 <0.01 0.011 0.021 0.040 0.017 0.027 0.021 0.030 0.029 0.024 0.032 0.021 0.025 0.020 0.024 <0.01 (5) <0.01 (5)	
NSW (Merino) long wool	aq susp 25 g ai/l	jetting, 5.1 l per sheep	0.0025	muscle	5 12 15	<0.01 (5) <0.01 (5) <0.01 (5)	ELANCO /GLP/980 9/1-1
				kidney	5 12 15	<0.01 (5) <0.01 (5) <0.01 (5)	
				liver	5 12 15	<0.01 (5) <0.01 (5) <0.01 (5)	
				back fat	5 12 15 21	<0.01 (5) <0.01 (5) <0.01 (5) <0.01 (5)	
				perirenal fat	5 12 15 21	<0.01 (5) <0.01 (5) <0.01 (5) <0.01 (5)	
NSW (Dorset Horn ewes) short wool	aq susp 25 g ai/l	plunge dip	0.001	muscle	5 15	<0.01 (5) <0.01 (5)	ELANCO /GLP/990 2a
				kidney	5 15	<0.01 (5) <0.01 (5)	
				liver	5 15	<0.01 (5) <0.01 (5)	
				back fat	5 15 21 56	<0.01 (3) 0.0100.017 <0.01 (5) <0.01 (5) <0.01 (5)	

Location (breed)	Application			Sample	whp days	Spinosad, mg/kg	Ref.
	Form	Method	Spray or dip conc, kg ai/hl				
				perirenal fat	5 15 21 56	0.01 <0.01 0.013 0.015 0.027 <0.01 (3) 0.01 0.011 <0.01 (5) <0.01 (5)	

**Dairy cattle.** In trials in the USA Holstein dairy cows were subjected to 3 kinds of direct treatment with spinosad and a premises treatment (Spurlock-Brouwer *et al.*, 2000). All treatments were applied 5 times.

1. Treatment every 7 days with 2 l of a 400 mg ai/l spray to cover the entire body (group of 9 cows). Lactating animals (groups of 3) were slaughtered 2, 7 and 14 days after the last treatment.
2. Treatment every 21 days with 5 l of a 400 mg ai/l spray to cover the entire body (group of 9 cows). Lactating animals (groups of 3) were slaughtered 2, 7 and 14 days after the last treatment.
3. Treatment every 14 days with 2 mg ai/kg bw, poured down the back from withers to tail (15 cows). Lactating animals (groups of 3) were slaughtered 2 and 14 days after the last treatment. The remaining (non-lactating) animals were slaughtered in groups of 3 21, 28 and 35 days after the last treatment.
4. Walls and ceiling of the premises were treated with a 800 mg ai/l spray every 7 days after the treated animals came in.

Milk was collected twice daily during the trial. Milk, muscle, kidney and liver were analysed by the immunoassay method and fat by HPLC. The results are shown in Table 74. Residues were higher in the fat than in other tissues, and the pour-on treatment produced the highest residues. Decline of the residues in fat was slow, with estimated half-lives of 20-40 days.

Spinosad residues were measured in 119 samples of milk and cream from the 3 treatments and followed the cycle of treatments with the level spiking the day after treatment and then falling over the next 3-5 days. Residues did not accumulate from one cycle to the next for the spray treatments, but did substantially for pour-on treatment. Spinosad does not partition totally into the fat fraction of milk. The mean quotient for concentration in cream divided by concentration in milk was 4.2, SD 1.6, max 9.0, min 1.2.

Table 74. Spinosad residues in the tissues of Holstein dairy cows from supervised trials in the USA in 1999. (Spurlock-Brouwer *et al.*, 2000). Muscle, kidney and liver were analysed by an immunoassay method and fat by an HPLC method (sum of spinosyns A, D, B and B of D).

Application				Sample	whp days	Spinosad, mg/kg	Ref.
Form	Method	Spray or dip conc, kg ai/hl	no				
aq susp 25 g ai/l	spray 2 l every 7 days	0.040	5	muscle	2 7 14	0.043 0.027 0.030 0.01 0.016 0.015 0.011 0.01 0.01	T9C739904
				kidney	2 7 14	0.083 0.13 0.13 0.036 0.045 0.037 0.021 0.018 0.030	

Application				Sample	whp days	Spinosad, mg/kg	Ref.
Form	Method	Spray or dip conc, kg ai/hl	no				
				liver	2 7 14	0.14 0.21 0.22 0.083 0.079 0.049 0.022 0.023 0.054	
				renal fat	2 7 14	0.37 0.47 0.30 0.27 0.32 0.25 0.21 0.19 0.20	
				sc fat	2 7 14	0.26 0.47 0.31 0.18 0.18 0.18 0.26 0.14 0.30	
aq susp 25 g ai/l	spray 5 l every 21 days	0.040	5	muscle	2 7 14	0.013 0.031 0.028 0.019 0.043 0.022 <0.01 <0.01 0.014	T9C739904
				kidney	2 7 14	0.075 0.11 0.089 0.049 0.058 0.055 0.011 0.022 0.030	
				liver	2 7 14	0.14 0.17 0.13 0.094 0.087 0.11 0.019 0.041 0.049	
				renal fat	2 7 14	0.19 0.21 0.26 0.36 0.33 0.30 0.056 0.17 0.25	
				sc fat	2 7 14	0.089 0.17 0.18 0.27 0.27 0.22 0.11 0.15 0.20	
aq susp 25 g ai/l	neat pour-on along backline at 2 mg/kg bw, every 21 days		5	muscle	2 14 21 28 35	<u>0.28</u> 0.074 0.070 0.078 0.13 <0.01 0.021 0.096 0.046 0.012 0.14 0.075 <0.01 0.075 0.018	T9C739904
				kidney	2 14 21 28 35	<u>0.87</u> 0.21 0.31 0.20 0.22 0.026 0.039 0.15 0.065 0.029 0.31 0.078 0.023 0.046 0.051	
				liver	2 14 21 28 35	<u>1.2</u> 0.38 0.66 0.23 0.35 0.040 0.052 0.080 0.17 0.059 0.38 0.075 0.031 0.077 0.065	
				renal fat	2 14 21 28 35	2.4 0.55 0.84 1.6 2.3 0.13 0.46 0.89 0.39 0.23 <u>2.7</u> 0.66 0.20 0.56 0.33	

Application				Sample	whp days	Spinosad, mg/kg	Ref.
Form	Method	Spray or dip conc, kg ai/hl	no				
				sc fat	2 14 21 28 35	1.7 0.22 0.50 1.6 <u>2.2</u> 0.12 0.57 0.94 0.50 0.26 0.27 0.79 0.26 0.79 0.58	

Table 75. Spinosad residues in the milk of groups of dairy cattle subject to 3 different treatments through 5 cycles of each treatment (Spurlock-Brouwer *et al.*, 2000). Treatment occurred on days marked 't'. Milk samples were analysed by an immunoassay method.

Days	Spinosad, mg/l milk		
	2-l spray each 7 days	5-l spray each 21 days	pour-on each 14 days
0	t 0.002	t 0.002	t 0.001
1	0.061	0.091	0.041
2	0.046	0.061	0.090
5	0.014	0.020	0.058
7	t 0.017		
8	0.061		
9	0.053		
12	0.017		
14	t 0.012		t 0.011
15	0.069		0.161
16	0.066		0.214
19	0.025		0.096
21	t 0.016	t 0.004	
22	0.060	0.083	
23	0.045	0.054	
26	0.017	0.015	
28	t 0.013		t 0.024
29	0.061		0.30
30	0.052		0.34
33	0.033		0.11
42		t 0.005	t 0.036
43		0.084	0.37
44		0.078	0.43
47		0.028	0.14
56		0.008	t 0.04
57		0.008	0.40
58		0.008	<u>0.65</u>
61		0.009	0.18
63		0.006	0.11
64		0.078	0.11
65		0.072	
68		0.029	
84		t 0.005	
85		0.064	
86		0.092	
89		0.035	

### Farm animal feeding studies

**Dairy cows.** Groups of Holstein dairy cows (weighing 410-631 kg) were dosed orally daily with gelatine capsules at nominal levels of 1, 3 and 10 ppm technical spinosad equivalent in the diet for 28 days (Rutherford and Robb, 1996b). The technical spinosad was characterized as 76% spinosyn A and 12% spinosyn D. Doses were expressed as pure spinosad in dry feed matter. The cows ate 8 kg dairy ration, 16 kg alfalfa hay cubes and 2 kg baled hay each day. There were 3 cows in the lower dose groups and 7 in the 10 ppm group. Three animals from each group were slaughtered on day 29, and the four remaining animals on the 10 ppm diet were put on a residue-free diet and slaughtered on days 36, 43, 57 and 85. Milk was collected twice daily and the morning and evening milk was pooled to provide a daily sample for each cow.

Residues in the milk reached a plateau after 7-14 days. The residues in the milk of the cows eventually fed a residue-free diet decreased with a half-life of approximately 4 days. The results reported for residues in whole and skimmed milk and cream from day 14 and day 28 are shown in Table 78. The relationship between levels in cream and in whole milk seems anomalous for a fat-soluble compound. Levels of residue in cream were only 3-5 times the level in the whole milk (expect 20-25 times).

Spinosad residues were higher in the fat than in the other tissues, as in the metabolism studies, but residues were also found in the other tissues. Transfer factors were reasonably consistent for each tissue at all doses (Table 79), which adds confidence in interpolating to feeding levels that might occur in practice. Levels in fat decreased with an initial half-life of 14 days once dosing stopped, but decreased more slowly longer term.

Table 76. Spinosyn residues measured by immunoassay and HPLC in the tissues of dairy cows dosed with technical spinosad for 28 days at nominal levels of 1, 3 and 10 ppm in the diet (Rutherford and Robb, 1996b). Four animals from the highest dose group were given a residue-free diet after day 28 and slaughtered on days 36, 43, 57 and 85.

Tissue	Dose – 1 ppm			Spinosyn, mg/kg Dose – 3 ppm				Dose – 10 ppm				Day	Spinosyn, mg/kg Dose – 10 ppm					
	IA <sup>1</sup>		HPLC	IA		HPLC		IA		HPLC			IA	HPLC				
Muscle	0.02	0.037	0.011	0.026	0.042	0.051	0.03	0.035	0.35	0.18	0.25	0.14	36	0.27	0.23			
	0.035		0.024		0.095		0.069		0.43		0.30		43	0.028	0.022			
													57	<0.01	<0.01			
													85	0.03	0.022			
Kidney	0.063	0.097	0.048	0.082	0.28	0.37	0.32	0.22	0.26	0.86	0.94	1.2	0.75	0.61	36	0.37	0.23	
	0.082		0.065				0.26					0.83		43	0.074	0.038		
														57	<0.01	<0.01		
														85	0.051	0.034		
Liver	0.14	0.22	0.089	0.15	0.51	0.80	0.52	0.32	0.44	1.9	2.1	3.2	0.99	1.0	1.7	36	0.84	0.34
	0.20		0.15				0.29							43	0.085	0.047		
														57	0.013	<0.01		
														85	0.026	0.01		
Fat			0.62	0.66			0.81	0.78	1.7			6.1	3.6	7.5	36		3.7	
			0.66												43		0.31	
														57		0.026		
														85		0.18		

<sup>1</sup> IA: immunoassay method.



Table 77. Spinosyn residues in the milk of dairy cows dosed with technical spinosad for 28 days at nominal levels of 1, 3 and 10 ppm in the diet (Rutherford and Robb, 1996b). Four animals from the highest dose group were given a residue-free diet after day 28 and slaughtered on days 36, 43, 57 and 85.

Milk	Spinosyn, mg/kg					
	Dose - 1 ppm		Dose - 3 ppm		Dose - 10 ppm	
	IA <sup>1</sup>	HPLC	IA	HPLC	IA	HPLC
Day 1	<0.01 (3)	<0.01 (3)	<0.01 0.01 <0.01	<0.01 (3)	0.02 0.02 0.01 0.039 0.024 0.023 0.014	0.02 0.02 0.01 0.035 0.02 0.03 0.01
Day 2	0.031 0.02 0.02		0.062 0.079 0.067		0.21 0.26 0.18 0.32 0.23 0.20 0.18	
Day 3	0.037 0.031 0.034	0.035 0.037 0.035	0.095 0.13 0.090	0.074 0.12 0.090	0.32 0.47 0.32 0.42 0.40 0.32 0.35	0.33 0.48 0.26 0.41 0.36 0.31 0.37
Day 4	0.051 0.029 0.034		0.092 0.16 0.12		0.38 0.45 0.32 0.54 0.45 0.38 0.37	
Day 5	0.039 0.033 0.03	0.044 0.044 0.03	0.096 0.12 0.11	0.11 0.13 0.12	0.44 0.48 0.38 0.45 0.45 0.37 0.34	0.40 0.53 0.36 0.47 0.46 0.38 0.32
Day 6	0.049 0.044 0.031		0.094 0.18 0.14		0.46 0.58 0.46 0.60 0.52 0.41 0.42	
Day 7	0.047 0.039 0.045	0.039 0.051 0.03	0.096 0.15 0.14	0.13 0.15 0.14	0.47 0.69 0.52 0.68 0.55 0.41 0.42	0.42 0.61 0.39 0.57 0.47 0.36 0.35
Day 10	0.041 0.063 0.047	0.041 0.051 0.044	0.10 0.18 0.18	0.096 0.15 0.14	0.42 0.60 0.56 0.95 0.61 0.37 0.56	0.36 0.52 0.41 0.63 0.46 0.29 0.43
Day 12	0.071 0.066 0.046		0.13 0.21 0.16		0.46 0.28 0.67 0.46 0.56 0.42 0.40	
Day 14	0.076 0.071 0.065	0.042 0.036 0.044	0.19 0.21 0.14	0.13 0.16 0.13	0.79 0.61 0.99 1.3 0.76 0.55 1.2	0.44 0.58 0.82 1.3 0.63 0.43 1.1
Day 16	0.075 0.053 0.084		0.12 0.19 0.12		0.44 0.85 0.57 0.74 0.44 0.30 0.47	
Day 21	0.044 0.043 0.041	0.032 0.046 0.041	0.13 0.16 0.13	0.11 0.17 0.12	0.47 0.58 0.53 0.75 0.43 0.26 0.54	0.41 0.53 0.48 0.65 0.48 0.35 0.46
Day 28	0.052 0.054 0.041		0.15 0.20 0.13		0.39 0.55 0.58 0.70 0.43 0.31 0.50	
Day 29					0.82 0.57 0.28 0.51	
Day 30					0.54 0.29 0.15 0.42	
Day 31					0.41 0.26 0.14 0.35	
Day 32					0.34 0.17 0.071 0.30	
Day 33					0.32 0.15 0.062 0.25	
Day 34					0.26 0.11 0.038 0.20	
Day 35					0.24 0.11 0.042 0.20	
Day 36					0.074 0.031 0.17	
Day 37					0.059 0.027 0.17	
Day 38					0.060 0.02 0.15	
Day 39					0.064 0.02 0.12	
Day 40					0.051 0.02 0.12	
Day 41					0.040 0.01 0.093	
Day 42					0.034 0.01 0.098	
Day 49					<0.01 0.054	
Day 56					<0.01 0.032	
Day 70					0.01	
Day 84					<0.01	

<sup>1</sup> IA immunoassay method

Table 78. Spinosad residues in milk and cream from day 14 and day 28 milkings (Rutherford and Robb, 1996b).

	Spinosad residues, mg/kg												
	1 ppm feeding level			3 ppm feeding level			10 ppm feeding level						
Animal	15	11	1	9	6	4	3	14	8	12	2	5	16
Day 14 milk, HPLC	0.044	0.037	0.052	0.133	0.15	0.10	0.55	0.52	0.82	1.3	1.6	0.43	1.1
Day 14 milk	0.076	0.071	0.065	0.19	0.21	0.14	0.79	0.61	0.99	1.31	0.76	0.55	1.16
Day 14 cream	0.22	0.20	0.10	0.46	0.53	0.47	2.1	2.2	2.0	3.1	2.5	1.5	2.1
Day 14 skimmed milk	0.008	0.005	0.004	0.01	0.011	0.01	0.042	0.043	0.065	0.082	0.055	0.024	0.038
Day 28 milk	0.032	0.046	0.041	0.11	0.17	0.12	0.41	0.53	0.48	0.65	0.48	0.35	0.46
Day 28 milk, HPLC	0.052	0.054	0.041	0.15	0.20	0.13	0.39	0.55	0.58	0.70	0.43	0.31	0.50
Day 28 cream	0.15	0.22	0.17	0.48	0.71	0.58	1.4	2.2	2.1	2.9	2.1	1.6	1.1
Day 28 skimmed milk	0.005	0.004	0.003	0.009	0.021	0.013	0.064	0.059	0.13	0.11	0.067	0.058	0.083

Table 79. Transfer factors for spinosad residues from feed to tissues and milk of dairy cows (Rutherford and Robb, 1996b).

Feeding level	Mean residue, mg/kg			Transfer factor = conc in tissue ÷ conc in feed			
	1 ppm	3 ppm	10 ppm	1 ppm	3 ppm	10 ppm	Mean
Muscle	0.026	0.054	0.28	0.026	0.018	0.028	0.024
Kidney	0.073	0.29	0.87	0.073	0.095	0.087	0.085
Liver	0.16	0.48	1.8	0.16	0.16	0.18	0.17
Fat	0.65	1.1	5.7	0.65	0.37	0.57	0.53
Milk 28 days	0.044	0.15	0.49	0.044	0.048	0.049	0.047
Cream 28 days	0.18	0.59	1.9	0.18	0.20	0.19	0.19
Skimmed milk, 28 days	0.004	0.014	0.082	0.004	0.005	0.008	0.006

**Laying hens.** Groups of 12 White Leghorn laying hens each bird weighing c.1.5 kg were dosed by gelatine capsules at nominal levels of 0.1, 0.3, 1 and 5 ppm technical spinosad equivalent in the diet for 41 days (Gardner and Dolder, 1998). The technical spinosad was characterized as 76% spinosyn A and 12% spinosyn D. Birds ate an average 123 g of feed per day. Eggs were collected daily and were composited in groups of 3. On day 42, 4-9 hours after the last dose, hens from each group were killed. Samples were analysed by HPLC; the LOQ was 0.03 mg/kg for fat and 0.01 mg/kg for other tissues.

Residues were often below the LOQ at the lower feeding levels, so differences between tissues are clearer at the higher feeding level. At 5 ppm, spinosad residues were highest in the abdominal fat, 1.4 mg/kg, 1.0 mg/kg in subcutaneous fat, 0.18 mg/kg in whole body including fat and skin, 0.092 mg/kg in liver and 0.062 and 0.027 mg/kg in dark and light muscle meats respectively. Residues in fat from the 5 ppm dosing group were 8.7 and 7.0 times as high as levels from the 1 ppm group in abdominal and subcutaneous fat respectively, slightly more than the expected 5 times. Residues in eggs reached a plateau by day 13 in the highest dose group, and were generally below the LOQ (0.01 mg/kg) from the other groups.

Table 80. Spinosyn residues in the tissues and eggs of laying hens dosed with technical spinosad for 41 days at nominal levels of 0.1, 0.3, 1 and 5 ppm in the diet (Gardner and Dolder, 1998).

Sample	Residues, mg/kg							
	Dose – 0.1 ppm		Dose – 0.3 ppm		Dose – 1 ppm		Dose – 5 ppm	
	spinosyn A	spinosyn D	spinosyn A	spinosyn D	spinosyn A	spinosyn D	spinosyn A	spinosyn D
Whole body <sup>1</sup>	<0.01 (3)	<0.01 (3)	<0.01 (3)	<0.01 (3)	0.02 <0.01 0.01	<0.01 (3)	0.12 0.15 0.15	0.031 0.045 0.047
Light muscle	<0.01 (3)	<0.01 (3)	<0.01 (3)	<0.01 (3)	<0.01 (3)	<0.01 (3)	0.021 0.039 0.021	<0.01 (3)
Dark muscle	<0.01 (3)	<0.01 (3)	<0.01 (3)	<0.01 (3)	<0.01 (3)	<0.01 (3)	0.047 0.049 0.051	0.013 0.014 0.013
Fat, abdominal	<0.03 0.03 <0.03	<0.03 (3)	<0.03 0.047 <0.03	<0.03 (3)	0.13 0.12 0.11	0.049 0.040 0.040	1.0 1.2	0.30 0.35
Fat, SC	<0.03 (2) 0.04	<0.03 (3)	<0.03 0.05 <0.03	<0.03 (3)	0.12 0.12 0.095	0.04 0.04 0.03	0.66 1.2 0.55	0.18 0.32 0.19
Liver	<0.01 (3)	<0.01 (3)	<0.01 (3)	<0.01 (3)	0.01 <0.01 <0.01	<0.01 (3)	0.082 0.067 0.043	0.035 0.031 0.019
Eggs, day 1							<0.01 (3)	<0.01 (3)
Eggs, day 4							0.053 0.042 0.069	0.01 <0.01 0.01
Eggs, day 7							0.081 0.11	0.02 0.025
Eggs, day 10							0.14 0.14 0.16	0.035 0.036 0.041
Eggs, day 13							0.13 0.13 0.26	0.032 0.034 0.073
Eggs, day 20							0.13 0.14 0.18	0.037 0.036 0.030
Eggs, day 28	<0.01 (3)	<0.01 (3)	<0.01 (3)	<0.01 (3)	0.01 <0.01 0.01	<0.01 (3)	0.098 0.14 0.074	0.027 0.038 0.020
Eggs, day 35	<0.01 (3)	<0.01 (3)	<0.01 (3)	<0.01 (3)	0.01 0.01 <0.01	<0.01 (3)	0.16 0.11 0.13	0.042 0.029 0.037
Eggs, day 41	<0.01 (3)	<0.01 (3)	<0.01 (2) 0.02	<0.01 (3)	<0.01 0.01 <0.01	<0.01 (3)	0.12 0.14 0.15	0.036 0.040 0.044

<sup>1</sup> Whole body: the carcass was cut in half lengthways and bones removed. Whole body represents the whole commodity with overlying skin and fat, but with liver and abdominal fat removed.

Table 81. Transfer factors for spinosad residues from feed to tissues and eggs of laying hens (Gardner and Dolder, 1998).

Feeding level	Mean residue, mg/kg		Transfer factor = conc in tissue ÷ conc in feed	
	1 ppm	5 ppm	1 ppm	5 ppm
Whole body	0.010	0.18	0.01	0.04
Light muscle	<0.01	0.027	<0.01	0.01
Dark muscle	<0.01	0.062	<0.01	0.01
Fat, abdominal	0.16	1.43	0.16	0.29
Fat, SC	0.15	1.03	0.15	0.21
Liver	<0.01	0.092	<0.01	0.018
Eggs day 41	0.01	0.18	0.01	0.035

## FATE OF RESIDUES IN STORAGE AND PROCESSING

### In processing

The Meeting received information on the fate of spinosad residues during the processing of apples, oranges, grapes, tomatoes and cotton seed.

Apples. In a supervised trial in France Khoshab and Hastings (1999c) treated apples with 4 applications of spinosad (Table 82). 20 kg were processed into juice and wet pomace, and 6 kg into purée (Figure ). In the juicing operation spinosad residues were partitioned into the pomace rather than into the juice.

Bolles and Robb (1996) treated Red Chief apples on 5 occasions with an exaggerated spinosad application rate ( $\times 5$ ) before harvesting the apples 7 days after the last application and processing them to juice and pomace (Table 82). An apple RAC sample was analysed as well as the bulk apple sample (30 kg) sent for processing.

Procedures simulated commercial practices. The apples were washed in cold water for 5 minutes and then leaves, stems and other debris were removed. Apples were crushed to pulp and heated with steam to raise the temperature of the pulp to 40-50°C. Pectic enzyme was mixed in. After standing for 2 hours, the pulp was pressed to produce juice and wet pomace. The juice was further screened through a standard milk filter to provide the final processed juice. Approximately 30% of the residue was removed in the washing step. Residues partitioned into pomace rather than to juice.

Oranges. Hamlin oranges were treated with an exaggerated spinosad application rate ( $\times 5$ ) and harvested 4 days after the last application (approx. 400 kg) for processing to juice (Gardner and Phillips, 1997, RES96023 FL01). Much of the residue disappeared in the washing step. Residues were not detected in the juice, but were concentrated in the oil (Table 82).

Table 82. Spinosad residues in fruits and processed products.

CROP country, year, (variety)	Application					PHI, days, sample	Spinosyn residues, mg/kg						Ref.
	Form	kg ai/ha	kg ai/hl	water, l/ha	no.		A	D	K	B	B of D	Total	
APPLES France, 1998 (Golden)	SC 480	0.29	0.030	960	4	7							GHE- P-8252
						apples	0.09	0.02	<0.01	0.01	<0.01	0.12	
						pomace	0.17	0.06	<0.01	0.01	<0.01	0.23	
						purée	0.01	<0.01	<0.01	<0.01	<0.01	0.01	
ORANGES USA (FL), 1996 (Hamlin oranges)	SC 480	0.35 +0.50 +0.75 +0.90		480	4	4							RES96 023 FL01
						orange unw	0.065	0.010	<0.01	0.011	<0.01	0.088	
						orange washed	0.014	<0.01	<0.01	<0.01	<0.01	0.020	
						juice	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	
						dried pulp <sup>1</sup>	0.15	0.017	<0.01	0.024	<0.01	0.19	
APPLES USA (WA), 1995 (Red Chief)	WG 820	0.25 +0.35 +0.50 +0.50 +0.90		1900	5	7							RES95 041
						apples RAC	0.25	0.035	<0.01	<0.01	<0.01	0.30	
						apples unw	0.21	0.03	<0.01	<0.01	<0.01	0.25	
						apples washed	0.15	0.02	<0.01	<0.01	<0.01	0.18	
						juice	0.017	<0.01	<0.01	<0.01	<0.01	0.024	
ORANGES USA (FL), 1996 (Hamlin oranges)	SC 480	0.35 +0.50 +0.75 +0.90		480	4	oil	0.80	0.14	<0.01	0.15	0.018	1.3	RES96 023 FL01
APPLES USA (WA), 1995 (Red Chief)	WG 820	0.25 +0.35 +0.50 +0.50 +0.90		1900	5	wet pomace	1.1	0.15	0.01	0.042	0.01	1.3	RES95 041

<sup>1</sup> Dried citrus processing pulp: contains peel, membrane, rag and seeds that are chopped, limed and dried to 8-10% moisture.

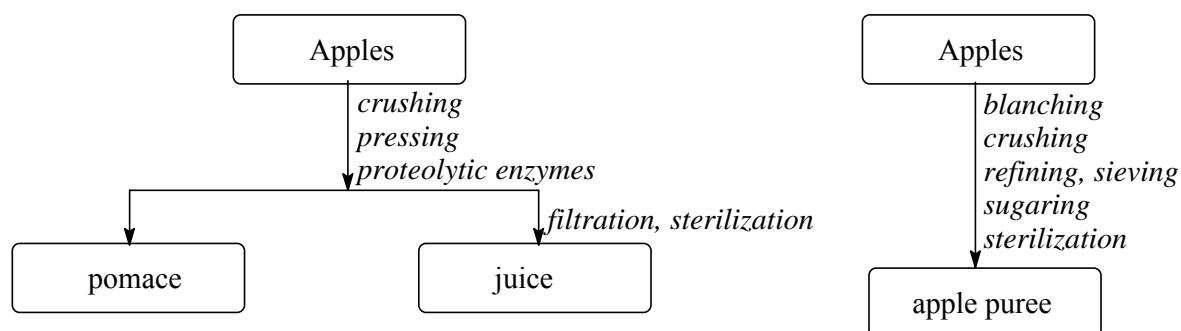


Figure 5. Apple processing (Khoshab and Hastings, 1999c).

**Grapes.** Khoshab and Volle (1999, report GHE-P-7575) treated grapes with spinosad before processing them into pomace, must and wine. Unfortunately residues in the white grapes (Chenin) were too low to provide useful information about the fate of spinosad during processing (Table 83). Residues in the red wine grapes (Gamay) were low, but provided evidence that residues tend to partition to the pomace rather than the wine.

Khoshab *et al.* (1999d, report GHE-P-7856) treated red wine grapes with spinosad before processing them into pomace, must and wine. Residues in the pomace were higher than in the grapes while residues in the must and wine were all below the LOQ of 0.01 mg/kg (Table 83).

Table 83. Spinosad residues in grapes, pomace, must and wine from supervised trials in France.

Year (variety)	Application <sup>4</sup>					PHI, days sample	Spinosyn residues, mg/kg				Ref.
	Form	kg ai/ha	kg ai/hl	water, l/ha	no.		A HPLC	D HPLC	Total <sup>1</sup> HPLC	IA <sup>2</sup>	
1997 (Chenin, Riparia Gloire)	SC 480	0.060	0.029	210	5	14 grapes	<0.01	<0.01	<0.01	<0.01	GHE-P-7575
						pomace	<0.01	<0.01	<0.01	0.01	
						must	<0.01	<0.01	<0.01	<0.01	
						wine <sup>3</sup>	<0.01	<0.01	<0.01	<0.01	
						wine <sup>4</sup>	<0.01	<0.01	<0.01	<0.01	
						c pomace <sub>5 6</sub>	<0.01	<0.01	<0.01	0.01	
1997 (Gamay)	SC 480	0.060	0.032	190	5	15 grapes	0.01	<0.01	0.02	0.02	GHE-P-7575
						pomace	0.03	<0.01	0.05	0.06	
						must	0.02	<0.01	0.03	0.03	
						wine <sup>3</sup>	<0.01	<0.01	<0.01	<0.01	
						wine <sup>4</sup>	<0.01	<0.01	<0.01	<0.01	
1998 (Red wine grape, Cot)	SC 480	0.096	0.042	230	5	15 grapes	0.02	0.01	0.03		GHE-P-7856
		+0.096	+0.042			pomace	0.04	0.03	0.07		
		+0.048	+0.021			must	<0.01	<0.01	<0.01		
		+0.048	+0.021			wine <sup>3</sup>	<0.01	<0.01	<0.01		
		+0.048	+0.021			wine <sup>4</sup>	<0.01	<0.01	<0.01		

Year (variety)	Application <sup>4</sup>					PHI, days sample	Spinosyn residues, mg/kg				Ref.
	Form	kg ai/ha	kg ai/hl	water, l/ha	no.		A HPLC	D HPLC	Total <sup>1</sup> HPLC	IA <sup>2</sup>	
Italy, 1998 (Trebiano)	SC 480	0.098	0.016	610	5	15 grapes pomace must wine <sup>3</sup> wine <sup>4</sup>					GHE-P-7855
		+0.091	+0.016	+570			0.01	0.01	0.03		
		+0.049	+0.005	+1010			<0.01	<0.01	0.01		
		+0.046	+0.005	+950			0.01	<0.01	0.02		
		+0.050	+0.005	+1030			<0.01	<0.01	<0.01		

<sup>1</sup> Total includes spinosyns A, D, B, B of D and K

<sup>2</sup> Immunoassay

<sup>3</sup> wine at bottling

<sup>4</sup> wine after 4 months

<sup>5</sup> c pomace produced from control grapes

Tomatoes. Khoshab (1999v, report GHE-P-7585) treated tomatoes with spinosad and then produced canned tomatoes, juice and purée in a small-scale process (25 kg tomatoes available for the two processes). Residues in the processed commodities were much less than in the whole tomatoes (Table 84). The initial washing ( Figure 6) probably removed much of the residue.

In a processing trial in the USA, tomatoes were treated with an exaggerated rate (×5) of spinosad and 350 kg harvested 1 day after the last treatment were converted into juice, purée, pomace and paste by a simulated commercial process (Rutherford and Robb, 1996d, RES95000). The tomatoes were washed with fresh water and then with chlorinated water, then passed through a grinder and heated to approximately 93°C before the peel and seeds were screened out as wet pomace. The wet pomace was dried in a food dehydrator to dry pomace containing approximately 92% dry matter. The juice was collected, canned, heated for at least 50 min at a minimum of 115°C, and concentrated in a vacuum evaporator to provide purée. A portion of purée was further condensed to paste in a vacuum kettle. The results are shown in Table 84. Washing removed much of the residue, which tended to concentrate in the pomace fractions.

The residue in the tomato RAC (0.53 mg/kg) taken directly for analysis was quite different from the residue found in the unwashed tomatoes (0.077 mg/kg) supplied for processing. It was found that the tomatoes plants had had very dense foliage and many of the fruit were on the ground sheltered from the spray. Visible fruit were picked for the RAC sample. For processing, whole plants were pulled from the ground and all pink or red tomatoes were picked. A higher proportion of sheltered fruit would have been collected for processing than for the RAC sample.

Table 84. Spinosad residues in tomatoes and their processed products from supervised trials in Italy and the USA.

Country, year (variety)	Application <sup>4</sup>					PHI, days sample	Spinosyn residues, mg/kg				Ref.
	Form	kg ai/ha	kg ai/hl	water, l/ha	no.		A HPLC	D HPLC	Total <sup>1</sup> HPLC	IA <sup>2</sup>	
Italy, 1997 (Erminia Peto Seed)	SC 480	0.54	0.067	800	5	3 tomatoes juice purée canned tomatoes					GHE-P-7585
							0.30	0.08	0.40	0.38	
							0.01	<0.01	0.01	0.02	
							0.06	0.01	0.07	0.11	
							<0.01	<0.01	<0.01	0.02	

Country, year (variety)	Application <sup>4</sup>					PHI, days sample	Spinosyn residues, mg/kg				Ref.
	Form	kg ai/ha	kg ai/hl	water, l/ha	no.		A HPLC	D HPLC	Total <sup>1</sup> HPLC	IA <sup>2</sup>	
USA (CA), 1995 (La Rossa)	WG 800	0.25		900	5	1					RES95000
		+0.50		+890		tomato RAC	0.41	0.053	0.53		
		+0.52		+960		tomato unwashed	0.062	0.01	0.077		
		+0.54		+980		tomato washed	0.021	<0.01	0.027		
		+0.83		+1000		juice	0.018	<0.01	0.024		
						wet pomace	0.55	0.070	0.66		
						dry pomace	0.91	0.11	1.1		
						purée	0.042	<0.01	0.054		
						paste	0.12	0.020	0.15		

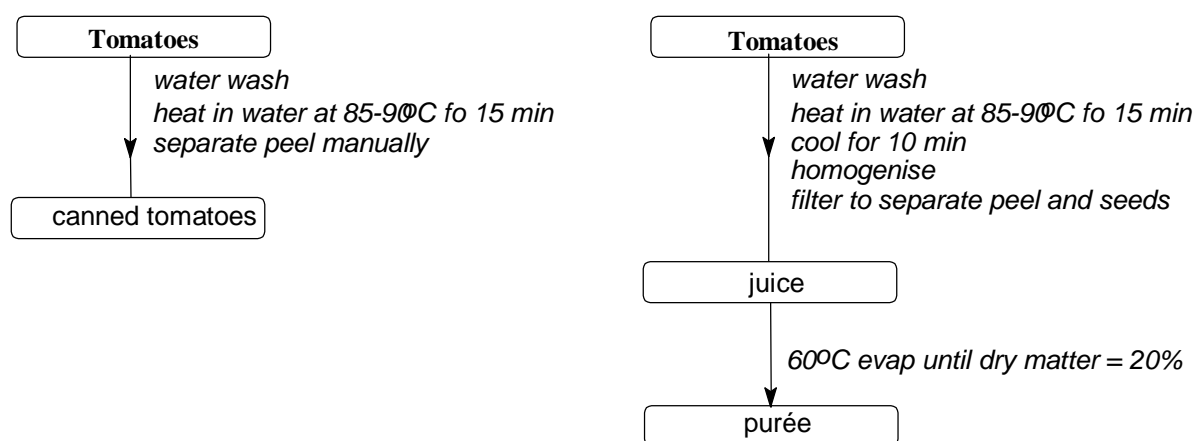


Figure 6. Tomato processing (Khoshab, 1999v, GHE-P-7585)

Cotton was treated at an exaggerated rate and approximately 18 kg of harvested seed was processed (Figure 7) to hulls, meal, oil and soapstock (Gardner and West, 1994b). Spinosad residues generally decreased during processing, but residues were apparent in the hulls and oil (Table 85).

Table 85. Spinosad residues in cotton seed and its processed products from supervised trials in the USA (Gardner and West, 1994b).

Location, year (variety)	Application				PHI, days sample	Residues, mg/kg			Ref.
	Form	kg ai/ha	water, l/ha	no.		spinosyn A	spinosyn D	total	
MS, 1993 (Des119 Sure Grow)	SC 480	0.54	560	5	28				RES93026. 01
		+0.60	+560		cotton seed	0.060	<0.01	0.067	
		+0.60	+410		hulls	0.012	<0.01	0.014	
		+0.60	+560		meal	<0.01	<0.01	<0.01	
		+0.84	+510		crude oil	0.011	<0.01	0.011	
					refined oil	0.012	<0.01	0.013	
					soapstock	<0.01	<0.01	<0.01	

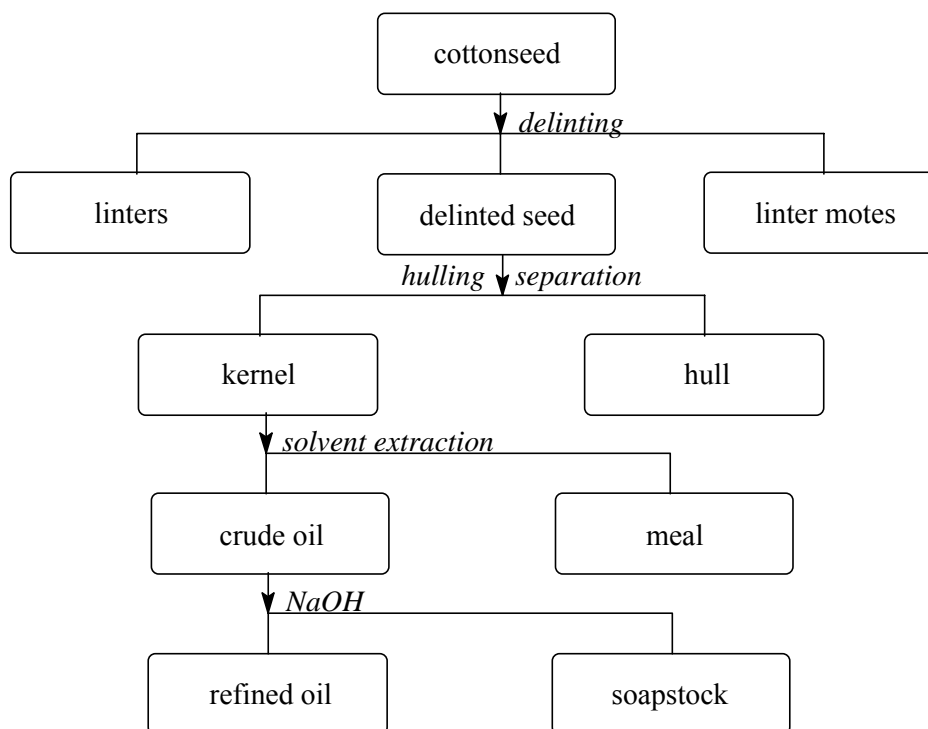


Figure 7. Cotton seed processing (Gardner and West, 1994b).

Processing factors were calculated from the residue data in the studies (Table 86) in three ways: spinosyn A residues, sum of spinosyns A and D by HPLC or immunoassay. Generally the results are similar except where residues are at or close to the 0.01 mg/kg LOQ, and they were lower in the processed commodity the calculated factor is shown with a “<” sign although the value of this is very limited when the residue in the raw commodity is close to the LOQ.

The residue is a surface one and washing removes some or most of it. In juicing operations the residue tends to attach itself to the solids, and in the production of oilseed it partitions to the oil rather than the meal.

Table 86. Calculated processing factors for apples, oranges, grapes, tomatoes and cotton seed.

Crop	Commodity	Residues, mg/kg			Processing factors			Ref.
		Spinosyn A	Sum (A+D)	IA	Spinosyn A	Sum (A+D)	IA	
Apples	apples	0.09	0.11					GHE-P-8252
	pomace	0.17	0.23		1.9	2.1		
	purée	0.01	0.01		0.11	0.09		
	juice	<0.01	<0.01		<0.11	<0.09		
Apples	apples unwashed	0.21	0.24					RES95041
	apples washed	0.15	0.17		0.71	0.71		
	juice	0.017	0.017		0.08	0.07		
	wet pomace	1.1	1.25		5.2	5.2		
Oranges	orange unwashed	0.065	0.075					RES96023 FL01
	orange washed	0.014	0.014		0.22	0.19		
	juice	<0.01	<0.01		<0.15	<0.13		
	dried pulp	0.15	0.167		2.3	2.2		
	oil	0.8	0.94		12	13		



Crop	Commodity	Residues, mg/kg			Processing factors			Ref.
		Spinosyn A	Sum (A+D)	IA	Spinosyn A	Sum (A+D)	IA	
Grapes	grapes	0.01	0.01	0.02				GHE-P-7575
	pomace	0.03	0.03	0.06	3	3.0	3	
	must	0.02	0.02	0.03	2	2.0	1.5	
	wine at bottling	<0.01	<0.01	<0.01	<1	<1	<0.5	
	wine after 4 months	<0.01	<0.01	<0.01	<1	<1	<0.5	
Grapes	grapes	0.02	0.03					GHE-P-7856
	pomace	0.04	0.07		2.0	2.3		
	must	<0.01	<0.01		<0.5	0.3		
	wine at bottling	<0.01	<0.01		<0.5	<0.3		
	wine after 4 months	<0.01	<0.01		<0.5	<0.3		
Grapes	grapes	0.01	0.02					GHE-P-7855
	pomace	<0.01	0.01		<1	<0.5		
	must	0.01	0.01		1	0.5		
	wine at bottling	<0.01	<0.01		<1	<0.5		
	wine after 4 months	<0.01	<0.01		<1	<0.5		
Tomato	tomatoes	0.3	0.38	0.38				GHE-P-7585
	juice	0.01	0.01	0.02	0.033	0.026	0.053	
	purée	0.06	0.07	0.11	0.20	0.18	0.29	
	canned tomatoes	<0.01	<0.01	0.02	<0.03	<0.03	0.053	
Tomato	tomato unwashed	0.062	0.072					RES95000
	tomato washed	0.021	0.021		0.34	0.29		
	juice	0.018	0.018		0.29	0.25		
	wet pomace	0.55	0.62		8.9	8.6		
	dry pomace	0.91	1.02		15	14		
	purée	0.042	0.042		0.68	0.58		
	paste	0.12	0.14		1.94	1.94		
Cotton seed	cotton seed	0.06	0.06					RES93026.01
	hulls	0.012	0.012		0.20	0.20		
	meal	<0.01	<0.01		<0.17	<0.17		
	crude oil	0.011	0.011		0.18	0.18		
	refined oil	0.012	0.012		0.20	0.20		
	soapstock	<0.01	<0.01		<0.17	<0.17		

### Residues in the edible portion of food commodities

Information was reported on residues in head cabbages, head lettuce and celery from residue trials.

In a trial on mandarins in Japan (Table 34), residues of spinosyn A were 0.09 and 0.05 mg/kg in the peel, but none were found in the pulp.

In US trials the residue was essentially on the peel of oranges, and was undetectable in the pulp of muskmelons. Residues in head cabbages were reduced by averages of 78% in head cabbages, and of 77% in head lettuce when wrapper leaves were removed (calculations based on those cases where residues exceeded the LOQ). Residues in trimmed celery were approximately 15% of those in untrimmed celery.

Table 87. Spinosad residues in trade commodities and edible portions of citrus fruits, melons, cabbages, head lettuce and celery (Table 34, Table 46, Table 47, Table 57 and Table 58).

Sample	Trade commodity, mg/kg	Edible portion, mg/kg	Peel, mg/kg	Residue in edible portion ÷ residue in commodity	Ref.
	<b>whole fruit</b>	<b>peeled fruit</b>			
Mandarin		<0.01	0.09		GHF-P-1683
Orange	0.046	<0.01	0.10	<0.22	RES96023
Orange	0.022	<0.01	0.052	<0.45	RES96023

Sample	Trade commodity, mg/kg	Edible portion, mg/kg	Peel, mg/kg	Residue in edible portion ÷ residue in commodity	Ref.
Orange	0.017	<0.01	0.021	<0.59	RES96023
Orange	0.01	<0.01	0.035	<1.00	RES96023
Orange	0.14	0.01	0.64	0.07	RES96023
Orange	0.06	<0.01	0.30	<0.17	RES96023
Orange	0.20	<0.01	0.78	<0.05	RES96023
Orange	0.072	<0.01	0.32	<0.14	RES96023
Orange	0.053	<0.01	0.08	<0.19	RES96023
Orange	0.011	<0.01	0.054	<0.91	RES96023
Orange	0.031	<0.01	0.046	<0.32	RES96023
Orange	0.016	<0.01	0.11	<0.63	RES96023
Musk melon	0.16	<0.01		<0.063	RES97002
Musk melon	0.092	<0.01		<0.11	RES97002
	<b>include wrapper leaves</b>	<b>wrapper leaves removed</b>			
Head cabbage	0.089	0.02		0.22	RES95001 CA
Head cabbage	1.09	0.17		0.16	RES95001 FL
Head cabbage	0.01	<0.01		<1	RES95001 IN1
Head cabbage	0.053	0.02		0.38	RES95001 IN2
Head cabbage	0.08	0.01		0.13	RES95001 PA1
Head cabbage	0.02	<0.01		<0.5	RES95001PA2
Head cabbage	1.0	0.36		0.38	RES95001 TX
Head cabbage	0.37	0.03		0.08	RES95001 VA
Head lettuce	1.97	1.85		0.94	RES96008AZ LV9606
Head lettuce	0.93	0.073		0.08	RES96008CA3 LV9605
Head lettuce	0.12	<0.01		<0.08	RES96008NJ LV9601
Head lettuce	0.109	0.01		0.09	RES96009/RES96008CA2
Head lettuce	0.052	<0.01		<0.19	RES96009/RES96008CA2
Head lettuce	0.77	0.019		0.02	RES96009/RES96008CA1
Head lettuce	0.672	0.024		0.04	RES96009/RES96008CA1
Head lettuce	0.73	0.172		0.24	RES96009/RES96008FL
Head lettuce	0.85	0.194		0.23	RES96009/RES96008FL
	<b>untrimmed</b>	<b>trimmed</b>			
Celery	1.1	0.18		0.16	RES96008 FL
Celery	1.3	0.31		0.24	RES96008 MI
Celery	0.454	0.034		0.07	RES96008 CA1
Celery	0.84	0.087		0.10	RES96008 CA2
Celery	0.4	0.11		0.28	RES96008 CA3
Celery	1.7	0.1		0.06	RES96008 AZ

## RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

No information was available on residue monitoring data for spinosad.

## NATIONAL MAXIMUM RESIDUE LIMITS

The Meeting was aware that the following national MRLs had been established.

Country	MRL, mg/kg	Commodity
Argentina	0.03	Tomato
	<0.001*	Soya bean; cotton seed
Australia	5	Cabbage, Chinese; leafy vegetables (except Chinese cabbage); lettuce; spinach
	0.5	Brassica vegetables (cole or cabbage); broccoli; cabbage; cauliflower; flowerhead Brassica (broccoli, cauliflower)
	0.5 (T)	Strawberry
	0.2	Cattle, fat
	0.2 (T)	Melon

Country	MRL, mg/kg	Commodity
	0.2	Peppers; sheep fat; tomato
	0.1 (T)	Apple; citrus; egg plant; grapes; pome fruits (apples, pears)
	0.05	Edible mammalian offal
	0.02	Maize grain, sweet; milk of cattle, goats, and sheep
	0.01	Cotton seed
	0.01*	Egg; poultry offal, poultry meat
Brazil	0.1	Tomato
	0.01	Potato; soya bean
Japan	5	Japanese radish (leaf); lettuce
	2	Egg plant; peppers; tea; tomato
	1	Cabbage; Chinese cabbage
	0.5	Apple
	0.2	Japanese radish (root); peach
Mexico	2	Flowerhead Brassica (broccoli, cauliflower)
	0.4	Peppers; tomato
	0.02	Cotton seed
New Zealand	0.1	Apple; avocado; broccoli; Brussels sprouts; cabbage; cabbage, Chinese; cauliflower; Brassica vegetables (cole or cabbage); flowerhead Brassica (broccoli, cauliflower); kiwifruit; pome fruits (apples, pears); tomato
Philippines	2	Cabbage
Switzerland	1	Cabbage; pepper
	0.5	Tomato
	0.2	Cucumber
	0.1	Grapes
Taiwan	1	Cabbage
Thailand	0.05	Cabbage
USA	20	Aspirated grain fractions
	10	Ti palm; turnip greens; vegetables, leafy Brassica – crop group 5-b
	10 (T)	Beet, sugar (tops)
	8	Cilantro; vegetables, leafy (except Brassica) – crop group 4; watercress
	5	Milk, fat
	4 (T)	Alfalfa forage; alfalfa hay
	3.5	Cattle, fat; goats, fat; hogs, fat; horses, fat; sheep, fat
	3	Citrus oil
	2	Almond hulls; vegetables, head and stem Brassica- crop group 5-a
	1.5	Cotton gin by products
	1	Amaranth, grain; cattle, mby; corn, fodder; corn, forage; corn, hay; corn, stover; corn, straw; corn, sweet, forage; corn, sweet, stover; goats, mby; hogs, mby; horses, mby; pearl, millet; proso, millet; sheep, mby; sorghum, fodder; sorghum, forage; sorghum, grain; sorghum, hay; sorghum, stover; sorghum, straw; wheat, fodder; wheat, forage; wheat, hay; wheat, stover; wheat, straw
	1 (T)	Peanut hay
	0.5	Apple pomace, wet; citrus pulp, dried; milk, whole
	0.4	Vegetables, fruiting except cucurbits – crop group 8
	0.3	Acerola; atemoya; avocado; biriba; canistel; cherimoya; citrus fruits group; cucurbit vegetables group; custard apple; feijoa; guava; ilama; jaboticaba; legume vegetables, edible podded – crop subgroup 6-a; longan; lychee; mango; papaya; passionfruit; pulasan; rambutan; sapodilla; sapote, black; sapote, mamey; sapote, white; soursop; Spanish lime; star apple; starfruit; sugar apple; wax jambu
	0.2	Apples; poultry, fat; stone fruit group
	0.15	Cattle, meat; goats, meat; hogs, meat; horses, meat; sheep, meat; wheat, bran; wheat, flour; wheat, middlings; wheat, shorts
	0.02	Almond; animal feed, non-grass- crop group 18; barley, grain; buckwheat, grain; corn, field, grain; corn, grain; corn, pop, grain; corn, sweet, k+cwhr; cotton seed; grass, fodder, forage, hay –crop group 17; legume veg, dried shell pea and bean (except soya beans)– crop subgroup 6-c; legume vegetables, succulent shelled pea and bean – crop subgroup 6-b; oats, grain; pistachio; popcorn, grain; poultry, eggs; poultry, meat; poultry, mby; rye, grain; soya beans; teosinte, grain; tuberous and corm vegetables – crop group 1-c; wheat, grain
	0.02 (T)	Beet, sugar (root); grapes; kiwifruit; nectarines; peanuts; strawberries

\* indicates lower limit of determination (LOD) or MRL set at or about limit of analytical quantification.

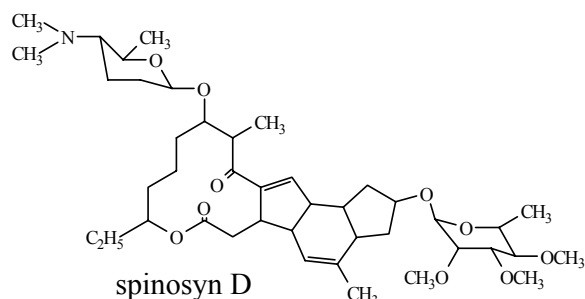
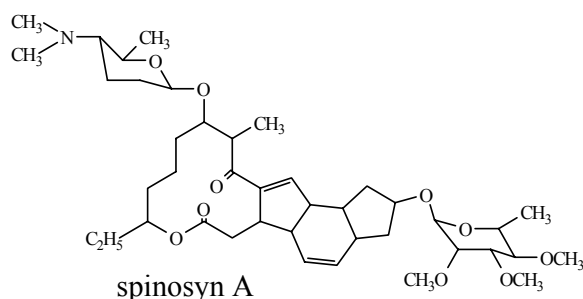
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The definition of the residue is spinosyn A + spinosyn D in Argentina, Australia, Brazil, Canada, EU, Malaysia, NZ, PRC, Taiwan and the USA.

## APPRAISAL

Residue and analytical aspects of spinosad were considered for the first time by the present Meeting.

Spinosad is a naturally derived fermentation product, which has demonstrated insect control activity against a large number of pests, including members of the insect orders Lepidoptera, Coleoptera and Thysanoptera. The product contains a mixture of two structurally similar molecules which are both active insecticidally and have been designated spinosyn A and spinosyn D. *N*-Demethyl-spinosyn A is called spinosyn B, and the analogous product from spinosyn D is called spinosyn B of D.



The Meeting received extensive information on the metabolism and environmental fate of spinosad, methods of analysis for residues, stability in freezer storage, national registered use patterns, the results of supervised trials, direct animal treatments, farm animal feeding studies, the fate of residues in processing and national MRLs.

## Metabolism

### Animals

Spinosyns A and D, reasonably uniformly radiolabelled at 23 carbons in the aglycone ring system, were used in studies of metabolism and environment fate. The amino and rhamnose sugars did not contain the  $^{14}\text{C}$  label.

When lactating goats were dosed orally with [ $^{14}\text{C}$ ]spinosyn A or [ $^{14}\text{C}$ ]spinosyn D at the equivalent of 10 ppm in the feed for 3 consecutive days, a considerable portion of the residue (45% spinosyn A and 20% spinosyn D) transferred to tissues and milk. Excretion occurred mainly via the faeces. The parent compounds (spinosyns A and D) were major components of the residue in tissues and milk and constituted an especially high percentage of the total residue in fat (86 and 85%) and milk (71 and 81%). The concentrations of spinosyn A in fat and milk were 3.1 and 0.45 mg/kg, respectively. A number of metabolites were identified and were most prevalent in kidney and liver. The metabolites resulted from *N*-demethylation and hydroxylation of the macrolide ring.

Most of a dose of [ $^{14}\text{C}$ ]spinosyn A (69%) or [ $^{14}\text{C}$ ]spinosyn D (82%) appeared in the excreta of laying hens dosed at the equivalent of 10 ppm in feed for 5 consecutive days. The concentrations in eggs were apparently still increasing at the end of the study. The highest concentrations occurred in fat, parent compound constituting most of the residue; spinosyns A and D constituted 81% and 79%

of the fat residue, respectively, at concentrations of 1.8 and 0.81 mg/kg. The parent compounds were also the main or important constituents of the residue in muscle and eggs. Substantial metabolism occurred in liver, where the metabolites were identified as deriving from *N*-demethylation, *O*-demethylation and loss of the forosamine sugar moiety.

When goats were treated dermally once along the backline with [ $^{14}\text{C}$ ]spinosyn A or [ $^{14}\text{C}$ ]spinosyn D, more residue was found in liver and fat than in other tissues. The parent compound was the predominant component of the residue, particularly in fat and milk. The metabolites were produced by *N*-demethylation and hydroxylation of the macrolide ring, a process also identified after oral dosing. The concentrations of residues in milk reached a peak 40–70 h after treatment.

### *Plants*

The Meeting received information on the fate of spinosyns after foliar application to apples, cabbage, tomatoes, turnips, grapes and cotton. The residues of spinosad on fruits, vegetables and other crops are usually at the surface, and the main primary degradation step is photolysis.

Apple trees were sprayed with [ $^{14}\text{C}$ ]spinosyn A or [ $^{14}\text{C}$ ]spinosyn D, one branch being protected from the spray and some apples being protected from light immediately after spraying. The total amount of radiolabel in the apples decreased by about half during the 42 days of sampling, most likely because of growth dilution. The residue occurred mostly on the surface; even after 42 days, about 60% of the remaining residue could be rinsed from the surface. The concentrations of parent spinosyns A and D declined quickly (more than 50% during the first 3 days). The only metabolites that were characterized were spinosyn B and spinosyn B of D, both resulting from *N*-demethylation of the parent compound. The nature of the radiolabelled residues was extensively investigated: they were shown to be polar and to have multiple components. Fractions taken on day 14 from apples sprayed with spinosyn A had low sensitivity in the spinosyn immunoassay, suggesting that the residues did not contain structures similar to those to which the immunoassay is sensitive (spinosyns A, B, C, E, F, K or pseudoaglycone of A).

Both spinosyns A and D were more persistent on apples kept from the light, indicating that photolysis is a major process of degradation. The radiolabelled residues in apples protected from spraying represented only 1.3% of those on apples that had been sprayed directly on day 42, indicating that translocation was minimal. The radiolabel was shown to be incorporated into structural carbohydrates in both treated and untreated leaves.

When grapes were treated separately with [ $^{14}\text{C}$ ]spinosyn A and [ $^{14}\text{C}$ ]spinosyn D, a high percentage of residue was found on the surface, even when aged. When the grapes reached maturity (49 days after treatment), spinosyn A accounted for about 35% of its residue and spinosyn D for 22%. Other components of the residue were polar and numerous and were probably products of photolysis. Hydroxy-spinosyn A and hydroxy-spinosyn D were tentatively identified in the residues.

After cabbage was treated with [ $^{14}\text{C}$ ]spinosyn A and [ $^{14}\text{C}$ ]spinosyn D, the parent compounds disappeared rapidly, most likely by photolysis, and accounted for only 10 and 13% of the residue 3 days after treatment. In a study in which cabbages were treated with [ $^{14}\text{C}$ ]spinosyn A, spinosyns B and K were identified in the residue, which also comprised numerous polar compounds and some incorporation of radiolabel into natural compounds.

When tomato plants were treated four times with [ $^{14}\text{C}$ ]spinosyn A 0 and 3 days before harvest, spinosyn A accounted for 65% and 24% of the radiolabel in the fruit. A portion of the tomatoes (TRR, 0.080 mg/kg as spinosyn A) was processed to juice and seeds plus peel. The concentrations of residue in the juice (TRR, 0.048 mg/kg as spinosyn A) and seeds plus peel (TRR, 0.28 mg/kg as spinosyn A) indicated that most of the radiolabelled residue was on the surface.

Turnip plants were treated with [ $^{14}\text{C}$ ]spinosyn A and [ $^{14}\text{C}$ ]spinosyn D, and the leaves and roots were subsequently sampled for analysis. By day 10, the parent compounds constituted a minor proportion of the radiolabelled residue in the foliage; however, the residues of parent compounds that reached the root and were protected from sunlight were more persistent. By day 24, the concentrations of parent compound were higher in the roots (A: leaf and root, 0.075 and 0.084 mg/kg; D: leaf and root, 0.016 and 0.036 mg/kg) than in the foliage and constituted a much higher percentage of the total radiolabel in the roots. Spinosyns B, K and B of D, which are products of photolysis, appeared as components of the residue in leaf and root from day 0.

Cotton plants were treated five times with [ $^{14}\text{C}$ ]spinosyn A and [ $^{14}\text{C}$ ]spinosyn D. Cotton seed and fibre were collected from the plots 48 or 49 days after the final treatment and were ginned to separate seed from fibre. The concentrations of radiolabel were 0.29 mg/kg in seed and 0.22 mg/kg in fibre after spinosyn A treatment and 0.11 and 0.075 mg/kg (seed and fibre) after spinosyn D treatment. Despite persistent attempts to identify spinosyn-related compounds, none were identified in cotton seed. Further attempts on separated fractions of the seeds showed that at least some of the radiolabel had become incorporated into natural compounds; the other residues had multiple components and were highly polar. The radiolabel in the fibre was incorporated into cellulose.

### ***Environmental fate***

#### ***Soil***

The losses of spinosad by volatilization from soil and foliar surfaces were too small to be observed at 20 °C in a wind tunnel with air flowing at 1–1.5 m/s.

When [ $^{14}\text{C}$ ]spinosyn A and [ $^{14}\text{C}$ ]spinosyn D on a soil surface were exposed to sunlight in August–September at 39.8° N, the initial disappearance half-lives were 17 and 7 days, respectively. Subsequent disappearance was slow (estimated half-lives > 100 days), indicating that the residues had become absorbed into the soil particles and unavailable for exposure to UV radiation. Spinosyns B and B of D were identified as photoproducts. In another study, spinosyn B was shown to be the primary photoproduct of spinosyn A. Spinosyns A and B disappeared with a half-life of about 20 days. Other photoproducts were characterized as parent compound with a hydroxyl attached to the macrolide ring and an *N*-demethyl derivative, also hydroxylated on the macrolide ring.

Spinosyns A and D were quite persistent under aerobic soil conditions at 20 °C in the dark, with estimated half-lives of 40–75 days and 65–85 days, respectively, in four different soils. Spinosyn B and spinosyn B of D were the main degradation products and were more persistent than the parent compounds, with concentrations exceeding those of the parents after 56 days. The amount of mineralization of spinosyn A ranged from 5.8% within 1 year in a sandy silt to 26% within 6 months in a sandy loam, while that of spinosyn D ranged from 4.8% to 25% within 8 months. In another study of aerobic soil degradation, additional products of spinosyn A were characterized as a hydroxy-spinosyn A and a hydroxy-spinosyn B.

In a series of studies of soil adsorption and desorption, spinosyn A and its metabolite spinosyn B were rated as unlikely to leach in most agricultural soils.

The leaching behaviour of fresh, microbially aged and photolytically aged [ $^{14}\text{C}$ ]spinosyn A and [ $^{14}\text{C}$ ]spinosyn D in soil columns was tested on a loamy sand. The fresh residues were not leached at all. Some products of ageing were leached down the column and into the leachate. The compounds could not be fully identified but were substantially modified from the starting spinosyns.

In a study of confined rotational crops, lettuce, radish and wheat seed were sown into a soil that had been treated 30, 120 and 365 days previously with [ $^{14}\text{C}$ ]spinosyn A at 1.1 kg ai/ha. Radiolabel was present in lettuce leaf, radish root and leaf and wheat forage from crops grown to

maturity. No spinosyns or closely related metabolites were identified in the crops. At least some of the radiolabel had been incorporated into natural compounds.

Spinosyn A residues disappeared very quickly (> 70% within 1 day) in field studies of dissipation on a silty clay and a sandy loam. Three metabolites were formed at low concentrations, which declined within 2 months to undetectable levels. Very little of the residue penetrated below the top 15 cm. The mineralization half-life was about 7 months at both sites.

#### *Water-sediment systems*

In a study of photolysis, [ $^{14}\text{C}$ ]spinosyn A and [ $^{14}\text{C}$ ]spinosyn D dissolved in sterile pH 7 buffers at 2 mg/l in borosilicate glass tubes were subjected to natural sunlight at 39.9° N in summer. The disappearance half-lives for spinosyns A and D were 22.3 and 19.7 h of sunlight, respectively. The photoproducts were characterized as parent compounds with changes such as saturation of a double-bond and addition of a water molecule. The disappearance half-life for spinosyns A and D in pond water was 4.3 h. The photodegradates were identified as spinosyn B and spinosyn B of D.

In a study of anaerobic sediment water, [ $^{14}\text{C}$ ]spinosyn A and [ $^{14}\text{C}$ ]spinosyn D rapidly became attached to the sediment and were relatively persistent (50% decrease within 6 months). Little mineralization occurred (< 2% within 1 year). The main metabolites were spinosyn B (from A) and spinosyn B of D (from D).

Spinosad in the form of a diluted suspension concentrate formulation was applied to the surface of an aquatic microcosm (1200-l open tank) at a nominal rate of 0.10 kg ai/ha. The concentrations of residues declined rapidly in the water, with a half-life of 1–2 days. Small amounts of spinosyn A reached the sediment, generally accounting for only about 10–15% of that applied. The results suggest that spinosad dissipates principally by degradation (photolysis) and then by adsorption to the sediment.

#### *Methods of analysis*

Methods for the analysis of residues of the spinosyns fall into two main categories: HPLC and immunoassay. The methods have been extensively validated on a wide range of substrates.

The HPLC methods, after an extraction specific to the matrix, follow a reasonably standard clean-up, with determination based on UV or MS detection. These methods allow measurement of the individual spinosyns and provide data on spinosyns A, D, K, B and B of D in residue trials. Spinosyn A usually contributes most of the residue, and some HPLC methods are designed to concentrate on spinosyns A and D. The LOQ for most substrates was 0.01 mg/kg.

Immunoassay methods, again after an extraction designed for the matrix, may or may not require clean-up before the final colorimetric determination. The method is specific and represents the sum of the spinosyns and their metabolites. When the HPLC and immunoassay methods were tested side-by-side, the agreement was usually good. The method is based on a commercially available test kit in which the antibody is sensitive to several spinosyns. A portion of a cleaned-up sample extract is incubated with enzyme-conjugated spinosad and magnetic particles coated with antibodies specific to spinosad. The spinosad in the sample and enzyme-conjugated spinosad compete for antibody sites on the magnetic particles. When a magnetic field is applied to the particles at the end of the incubation period, the spinosad and enzyme-conjugated spinosad bound to antibodies on the particles are held in the sample tube by the magnetic field, while the unbound reagents are decanted. A coloured product, produced by incubating the antibody-bound enzyme conjugate with hydrogen peroxide and 3,3',5,5'-tetramethylbenzidine, is measured by its absorbance at 450 nm. The assay is sensitive to spinosyn analogues with little or no modification to the trimethylpyranosyl ring, but it is relatively insensitive

to analogues or degradates in which the trimethylpyranosyl ring has been modified or is missing. The LOQ for most substrates was 0.01 mg/kg.

### ***Stability of residues in stored analytical samples***

Stability in freezer storage was tested for a range of representative substrates. Residues of spinosyns A, D, K, B and B of D were generally stable for the intervals tested:

18 months: grapes, peppers, strawberries, wine (estimated 30% decrease in spinosyn D residues in wine within 12 months)

12 months: tomatoes, cabbage, cotton seed, potato, maize grain, sweet corn forage, sweet corn stover

6 months: apples, almond kernels, almond hulls, celery, spinach; incurred residues in liver, kidney, muscle and fat.

5 months: milk

3 months: apple juice

### ***Definition of the residue***

Spinosad is a mixture of spinosyn A and spinosyn D. After it has been applied to crops, the closely related compounds spinosyn B, spinosyn K and spinosyn B of D are formed, principally by photolysis. HPLC methods can be used to measure all these compounds separately, whereas immunoassay allows measurement of these spinosyns and also some other metabolites. Spinosyn A constitutes approximately 85% of the residue initially and in practice represents most of the spinosyn residue; in 482 of 624 (77%) measurements in the residue trials, spinosyn A constituted  $\geq 70\%$  of the measured residue. Spinosyn A and spinosyn D together generally constitute more than 90% of the total spinosyn residue.

Spinosyns A and D were major identifiable components of the residue in fat, muscle, kidney, liver and milk of goats dosed orally or treated dermally with spinosyns A and D.

In some trials the residue was measured by the immunoassay method; the residue so measured may be considered sufficiently close to the sum of spinosyn A and spinosyn D for the purpose of estimating maximum residues levels or dietary intake.

The log  $P_{ow}$  of 4 and 4.5 (pH 7) and the studies of animal metabolism indicate that spinosyns A and D should be described as soluble in body fat. However, spinosad residues are incompletely partitioned into the fat of milk. In the study with direct treatment of dairy cows, the ratio of residue in cream to that in milk was 4.2 (mean of 119 observations). In the feeding study in dairy cows, the concentrations of residue in cream were three to five times those in milk.

The Meeting recommended that spinosad be described as fat-soluble for the purposes of residues in meat but not for residues in milk.

The Meeting was aware that national governments had already adopted the sum of spinosyn A and spinosyn D as the residue definition for spinosad.

The Meeting recommended that the residue definition for compliance with MRLs and for estimation of dietary intake be the sum of spinosyn A and spinosyn D.

The proposed definition of the residue for compliance with MRLs and for estimation of dietary intake is the sum of spinosyn A and spinosyn D. The residue is fat-soluble, but residues in milk should be measured in whole milk.



### *Results of supervised trials*

The results of supervised trials were available for use of spinosad on almonds, apples, blueberries, Brassica vegetables, celery, citrus, cotton, cucurbits, egg plant, grapes, Japanese radish, kiwifruit, leafy vegetables, legume vegetables, lettuce, maize, navy beans, peppers, potatoes, sorghum, soya beans, stone fruit, strawberries, sweet corn, tomatoes and wheat. No relevant GAP was available to evaluate the data for blueberries, egg plant, grapes, navy beans and strawberries, and only those trials with relevant GAP are discussed below.

The residue definition for spinosad requires addition of residues of spinosyns A and D. In this calculation, when the concentration of residue of spinosyn D was below the LOQ, it was assumed to be zero, except when the concentrations of residues of both spinosyns A and D were below the LOQ. In that case, the total was taken as below the LOQ, which is a reasonable assumption because the concentration of spinosyn D is usually much lower than that of spinosyn A. For example:

Spinosyn A	Spinosyn D	Sum of spinosyns A and D
0.59	0.082	0.67
0.052	< 0.01	0.052
< 0.01	< 0.01	< 0.01

When residues had been measured in a sample by both HPLC and immunoassay, the results from the HPLC method were preferentially chosen for evaluation.

### *Citrus*

Spinosad is registered in the USA for use on citrus fruits at 0.18 kg ai/ha with a PHI of 1 day. The concentrations of residues resulting from trials in the USA in 1996 that met those conditions were grapefruit, 0.013, 0.021, 0.03, 0.061, 0.086 and 0.19 mg/kg; lemon, 0.021, 0.037, 0.056 and 0.14 mg/kg; and oranges, 0.01, 0.017, 0.031, 0.044, 0.046, 0.053, 0.053, 0.07, 0.11, 0.13, 0.14, 0.14 and 0.15 mg/kg. The residues in the three fruits appeared to be from the same population and were therefore evaluated together. The concentrations of spinosad residues in citrus in 23 trials that matched GAP in the USA, in ranked order (median underlined), were 0.01, 0.013, 0.017, 0.021, 0.021, 0.03, 0.031, 0.037, 0.044, 0.046, 0.053 (2), 0.056, 0.061, 0.07, 0.086, 0.11, 0.13, 0.14 (3), 0.15 and 0.19 mg/kg.

The Meeting estimated a maximum residue level of 0.3 mg/kg and an STMR value of 0.053 mg/kg for spinosad in citrus whole fruit.

Six samples of orange from the trials were peeled, and residues were measured in the peeled oranges. In five of the peeled oranges, the concentrations of residues were < 0.01 mg/kg (0.017, 0.031, 0.046, 0.053 and 0.20 mg/kg in the whole oranges). In one peeled orange (0.14 mg/kg in the whole orange), the concentration was 0.01 mg/kg, and this finding was taken as evidence that the concentrations in the edible portion were usually below the LOQ but occasionally reached 0.01 mg/kg.

The Meeting estimated an STMR value for spinosad in citrus edible portion of 0.01 mg/kg.

### *Apple*

In Japan, spinosad is registered for use on apple at a spray concentration of 0.01 kg ai/hl and harvesting 3 days after the final application. In two trials in Japan that matched GAP, the concentrations of residues of spinosyn A were 0.03 and 0.17 mg/kg.

GAP in the USA permits application of spinosad at 0.18 kg ai/ha on apples with a PHI of 7 days. The concentrations of spinosad residues, in ranked order, in apples from 32 trials that matched GAP were < 0.01 (8), 0.01 (4), 0.014, 0.015 (2), 0.016, 0.017, 0.020, 0.024 (3), 0.025, 0.028, 0.032, 0.033 (2), 0.036, 0.041 (2), 0.045, 0.078 and 0.080 mg/kg.

The results of the Japanese trials were not included in the evaluation because they probably did not represent the same population as those from the USA. The Meeting estimated a maximum residue level of 0.1 mg/kg and an STMR value of 0.0165 mg/kg for spinosad in apples.

#### *Stone fruits*

The results of trials in Japan on peach could not be evaluated because the results were for peel and flesh rather than fruit. Trials on nectarine in Chile could not be evaluated because the conditions did not match GAP.

GAP in the USA permits application of spinosad at 0.14 kg ai/ha with harvesting 7 days after the final application for cherries, plums and prunes or 14 days for peach, nectarine and apricot. The concentrations of residues, in ranked order, from eight trials on cherry that met GAP in the USA were < 0.02, 0.023, 0.03, 0.04, 0.06, 0.083 and 0.11 (2) mg/kg; those in seven trials on peach were < 0.02 (3), 0.03, 0.05 and 0.055 (2) mg/kg; and those in five trials on plum were < 0.02 (5) mg/kg.

The Meeting agreed that cherries, peaches and plums represent the stone fruit group and that cherries and peaches would usually have the highest concentrations of residues in the group. Therefore, a maximum residue level could be recommended for stone fruit. The concentrations in ranked order in the 20 trials (median underlined) were < 0.02 (9), 0.023, 0.03 (2), 0.04, 0.05, 0.055 (2), 0.06, 0.083 and 0.11 (2) mg/kg. The Meeting estimated a maximum residue level of 0.2 mg/kg and an STMR value of 0.0265 mg/kg for spinosad in stone fruits.

#### *Kiwifruit*

GAP in New Zealand permits application of spinosad at a spray concentration of 0.0048 kg ai/hl and harvesting 120 days after the final application on kiwifruit. In seven trials in New Zealand in 1998–99 which matched the application rate and with PHIs of 118–142 days, the concentrations of spinosad residues, in ranked order, were < 0.01 (3) 0.02 (2) and < 0.05 (2) mg/kg.

The Meeting estimated a maximum residue level of 0.05 mg/kg and an STMR value of 0.02 mg/kg for spinosad in kiwifruit.

#### *Brassica vegetables*

Spinosad is registered in Australia for use on broccoli, cauliflower, cabbage and Brussels sprouts at 0.096 kg ai/ha with a PHI of 3 days. In trials in Australia that matched GAP conditions, the concentrations of spinosad residues were 0.06, 0.08 and 0.39 mg/kg in broccoli; 0.02 mg/kg in cauliflower; < 0.01 mg/kg in cabbage; and 0.02 and 0.03 mg/kg in Brussels sprouts.

Spinosad is registered in New Zealand for use on Brassica vegetables at 0.048 kg ai/ha with a PHI of 3 days. In trials in New Zealand that matched GAP, the concentrations were 0.02 mg/kg in cauliflower and < 0.01 mg/kg in cabbage.

Spinosad is registered in Japan for use on cabbage at a spray concentration of 0.01 kg ai/hl and a PHI of 3 days. In trials in Japan that matched GAP conditions, the concentrations of spinosad residues in cabbage were < 0.01 and 0.01 mg/kg.

In the USA, spinosad is registered for use on cole crops at 0.18 kg ai/ha with a 1-day PHI. In trials in the USA that matched GAP (0.15 kg ai/ha is sufficiently close to 0.18 kg ai/ha) for cole crops, the concentrations of spinosad residues were 0.12, 0.16, 0.19, 0.35, 0.36, 0.39, 0.44 and 0.53 mg/kg in broccoli and 0.01, 0.02, 0.053, 0.080, 0.088, 0.37, 0.95 and 1.1 mg/kg in cabbage.

The data from the USA appeared to represent a different population from those from Australia, Japan and New Zealand. The Meeting agreed that the data from the USA for cabbage and broccoli represented the same population and could be combined for Brassica vegetables. The concentrations of spinosad residues in Brassica vegetables from the 16 trials in the USA, in ranked order, were 0.01, 0.02, 0.053, 0.080, 0.088, 0.12, 0.16, 0.19, 0.35, 0.36, 0.37, 0.39, 0.44, 0.53, 0.95 and 1.1 mg/kg.

The Meeting estimated a maximum residue level of 2 mg/kg and an STMR value of 0.27 mg/kg for spinosad in Brassica vegetables.

### *Cucurbits*

Spinosad is registered in the USA for use on cucumbers at 0.14 kg ai/ha with a 1-day PHI. In six trials that matched GAP, the concentrations of spinosad residues in cucumbers, in ranked order, were 0.01, 0.024, 0.046, 0.052, 0.053 and 0.059 mg/kg.

Spinosad is registered in the USA for use on cucurbit vegetables other than cucumbers at 0.14 kg ai/ha with a 3-day PHI. In six trials in the USA that matched GAP, the concentrations of spinosad residues in musk melons, in ranked order, were 0.036, 0.045, 0.054, 0.092, 0.12 and 0.16 mg/kg. In three trials in the USA that matched GAP, the concentrations of spinosad residues in summer squash were < 0.01, 0.024 and 0.038 mg/kg.

The Meeting agreed to pool the data to support an MRL for cucurbit vegetables, as follows (ranked order): < 0.01, 0.01, 0.024 (2), 0.036, 0.038, 0.045, 0.046, 0.052, 0.053, 0.054, 0.059, 0.092, 0.12 and 0.16 mg/kg. The Meeting estimated a maximum residue level of 0.2 mg/kg and an STMR value of 0.046 mg/kg for spinosad in cucurbit vegetables.

### *Tomato*

In Argentina, spinosad is registered for use on tomato at 0.11 kg ai/ha with harvesting permitted 3 days after the final application. In six trials in Argentina that matched GAP conditions, the concentrations of spinosad residues were 0.01, 0.02, 0.06 (2), 0.17 and 0.21 mg/kg.

In Australia, spinosad is registered for use on tomato at 0.096 kg ai/ha with harvesting permitted 1 day after the final application. In five trials in Australia that matched GAP conditions, the concentrations of spinosad residues were 0.02, 0.03 (3) and 0.04 mg/kg. In a trial in New Zealand that matched Australian GAP, the concentration of spinosad residues was 0.04 mg/kg.

GAP in New Zealand for use of spinosad on tomato requires a 3-day PHI after application at 0.048 kg ai/ha. The concentration of spinosad residues in a trial that matched GAP in New Zealand was < 0.01 mg/kg.

Spinosad is registered in the USA for use on fruiting vegetables, including tomato, at 0.18 kg ai/ha with harvesting permitted 1 day after the final application. The concentrations of spinosad residues in 18 trials that matched GAP (0.15 kg ai/ha is sufficiently close to 0.18 kg ai/ha), in ranked order, were < 0.01 (2), 0.013, 0.02 (3), 0.023, 0.024, 0.026, 0.03 (3), 0.04 (2), 0.062, 0.086 and 0.11 (2) mg/kg.

As the data on tomato appeared to represent the same population, except for that from New Zealand where the GAP application rate was lower, they may be combined for evaluation. The concentrations of spinosad residues in tomatoes in the 30 trials, in ranked order, were < 0.01 (2), 0.01, 0.013, 0.02 (5), 0.023, 0.024, 0.026, 0.03 (6), 0.04 (4), 0.06 (2), 0.062, 0.086, 0.11(2), 0.17 and 0.21 mg/kg. The Meeting estimated a maximum residue level of 0.3 mg/kg and an STMR value of 0.03 mg/kg for spinosad in tomato.

### *Peppers*

In Australia, spinosad is registered for use on peppers at 0.096 kg ai/ha with harvesting permitted 1 day after the final application. In two trials in Australia that matched GAP conditions, the concentrations of spinosad residues on sweet peppers were 0.04 and 0.12 mg/kg.

Spinosad is registered in the USA for use on fruiting vegetables, including peppers, at 0.18 kg ai/ha with harvesting permitted 1 day after the final application. The concentrations of spinosad residues in eight trials that matched GAP (0.15 kg ai/ha is sufficiently close to 0.18 kg ai/ha) in hot and sweet peppers, in ranked order, were 0.02, 0.03, 0.05 (2), 0.062, 0.073, 0.14 and 0.17 mg/kg.

The Meeting agreed to combine the data on peppers from Australia and the USA, as follows: 0.02, 0.03, 0.04, 0.05 (2) 0.062, 0.073, 0.12, 0.14 and 0.17 mg/kg. The Meeting estimated a maximum residue level of 0.3 mg/kg and an STMR value of 0.056 mg/kg for spinosad in peppers.

### *Sweet corn*

In the USA, spinosad is registered for use on sweet corn at 0.11 kg ai/ha with harvesting permitted 1 day after the final application. In nine trials in USA that matched GAP conditions, the concentrations of spinosad residues on sweet corn were below the LOQ (0.01 mg/kg). The Meeting estimated a maximum residue level of 0.01\* and an STMR value of 0.01 mg/kg for spinosad in sweet corn.

### *Leafy vegetables*

In the USA, spinosad is registered for use on cole crops, including mustard greens, at 0.18 kg ai/ha with a 1-day PHI. In trials in the USA that matched GAP (0.15 kg ai/ha is sufficiently close to 0.18 kg ai/ha) for cole crops, the concentrations of spinosad residues in mustard greens were 0.040, 1.0, 3.5, 4.0, 5.0, 5.5, 5.6 and 5.7 mg/kg.

Australian GAP permits an application rate of 0.096 kg ai/ha and a 3-day PHI for the use of spinosad on lettuce. In three trials in Australia that matched GAP conditions, the concentrations of spinosad residues in head lettuce were 0.21, 1.1 and 1.7 mg/kg.

Spinosad is registered in Australia for use on Chinese cabbage at 0.096 kg ai/ha with a PHI of 3 days. In a trial in Australia that matched GAP conditions, the concentration of spinosad residues in Chinese cabbage was 0.10 mg/kg.

Spinosad is registered in Japan for use on Chinese cabbage at a spray concentration of 0.01 kg ai/hl with a PHI of 3 days. In trials in Japan that matched GAP conditions, the concentration of spinosad residues in Chinese cabbage was 0.09 (2) mg/kg.

In the USA, spinosad is registered for use on leafy vegetables at 0.18 kg ai/ha with a 1-day PHI. In trials in the USA that matched GAP conditions (0.15 kg ai/ha is sufficiently close to 0.18 kg ai/ha), the concentrations of spinosad residues were 0.052, 0.11, 0.12, 0.67, 0.73, 0.77, 0.85, 0.93 and

2.0 mg/kg in head lettuce; 1.4, 1.9, 2.0, 4.7, 4.9 and 5.2 mg/kg in leaf lettuce; and 1.5, 1.9, 2.4, 2.8, 2.9, 3.0, 4.0, 4.4 and 4.5 mg/kg in spinach.

The range of residue concentrations was quite wide, but there was overlap among the different crops. The Meeting decided to pool the data to support an MRL for leafy vegetables, as follows (ranked order): 0.040, 0.052, 0.090 (2), 0.10, 0.11, 0.12, 0.21, 0.67, 0.73, 0.77, 0.85, 0.93, 1.0, 1.1, 1.4, 1.5, 1.7, 1.9 (2), 2.0 (2), 2.4, 2.8, 2.9, 3.0, 3.5, 4.0 (2), 4.4, 4.5, 4.7, 4.9, 5.0, 5.2, 5.5, 5.6 and 5.7 mg/kg. The Meeting estimated a maximum residue level of 10 mg/kg and an STMR value of 1.9 mg/kg for spinosad in leafy vegetables.

#### *Celery*

In the USA, spinosad is registered for use on leafy vegetables, including celery, at 0.18 kg ai/ha with a 1-day PHI. In trials in the USA that matched GAP (0.15 kg ai/ha is sufficiently close to 0.18 kg ai/ha), the concentrations of spinosad residues in celery were 0.40, 0.45, 0.84, 1.1, 1.3 and 1.7 mg/kg.

The Meeting estimated a maximum residue level of 2 mg/kg and an STMR value of 0.97 mg/kg for spinosad in celery.

#### *Legume vegetables*

Spinosad is registered in the USA for use on succulent beans at 0.11 kg ai/ha with harvesting permitted 3 days after the final application. In 11 trials that matched GAP, the concentrations of spinosad residues in snap beans seed and pod, in ranked order, were < 0.01 (2), 0.02 (3), 0.042, 0.077, 0.085, 0.14, 0.15 and 0.20 mg/kg. In seven trials that matched GAP, the concentrations of spinosad residues in snow peas seed and pod, in ranked order, were < 0.01, 0.01, 0.03, 0.039, 0.063, 0.20 and 0.21 mg/kg.

The Meeting agreed to pool the data for snap beans and snow peas to estimate an MRL for legume vegetables. The concentrations of residues, in ranked order, were < 0.01 (3), 0.01, 0.02 (3), 0.03, 0.039, 0.042, 0.063, 0.077, 0.085, 0.14, 0.15, 0.20 (2) and 0.21 mg/kg. The Meeting estimated a maximum residue level of 0.3 mg/kg and an STMR value of 0.041 mg/kg for spinosad in legume vegetables.

#### *Soya bean (dry)*

Spinosad is registered in Brazil for use on soya beans with application at 0.024 kg ai/ha and a 9-day PHI. In two trials in Brazil with a 9-day PHI but with application at 0.048 kg ai/ha, the concentration of spinosad residues was below the LOQ (0.01 mg/kg).

Spinosad is registered in the USA for use on soya beans at 0.070 kg ai/ha with harvesting permitted 28 days after the final application. The concentrations of spinosad residues were below the LOQ (0.01 mg/kg) in seven trials in which the application rate was 0.38 kg ai/ha and the PHI was 28 days.

The Meeting agreed that the residue in soya beans is effectively zero because application rates higher than that of GAP did not produce concentrations of residues exceeding the LOQ. The Meeting estimated a maximum residue level of 0.01\* mg/kg and an STMR value of 0 mg/kg for spinosad in soya bean (dry).

#### *Potato*

In Brazil, spinosad is registered for use on potato at 0.20 kg ai/ha with harvesting permitted 3 days after the final application. In two trials that matched GAP and two trials at 0.40 kg ai/ha, the concentrations of spinosad residues were below the LOQ (0.01 mg/kg).

Spinosad is registered in the USA for use on tuber vegetables including potatoes at 0.11 kg ai/ha with harvesting permitted 7 days after the final application. In 14 trials that matched GAP, the concentrations of spinosad residues were below the LOQ (0.005 mg/kg). In two trials with an application rate of 0.62 kg ai/ha and harvesting 7 and 8 days after the third treatment, the concentrations of residues were also below the LOQ (0.005 mg/kg).

The Meeting agreed that, because higher application rates did not result in measurable residues, the concentration in potatoes was effectively zero. The practical LOQ for enforcement purposes is 0.01 mg/kg. The Meeting estimated a maximum residue level of 0.01\* mg/kg and an STMR value of 0 mg/kg for spinosad in potato.

#### *Radish, Japanese*

GAP in Japan permits three spray applications of spinosad at a concentration of 0.01 kg ai/hl with harvesting 7 days after the final application. In two trials in which the conditions matched GAP, the concentrations of spinosad residues were < 0.01 and 0.01 mg/kg in Japanese radish roots and 0.07 and 0.23 mg/kg in the leaves.

The Meeting noted that the concentrations in the leaves would be included in the recommendations for leafy vegetables. Only two trials were available, and, even though Japanese radish is a minor crop, the Meeting agreed that the number of trials was insufficient to make a recommendation.

#### *Cereals*

Spinosad is registered in Brazil for use on maize at an application rate of 0.048 kg ai/ha and a 7-day PHI. In eight trials in Brazil, all with a 7-day PHI but with application rates of 0.048 kg ai/ha (two trials), 0.060 kg ai/ha (two trials), 0.096 kg ai/ha (two trials) and 0.12 kg ai/ha (two trials), the concentrations of spinosad residues were all below the LOQ (0.01 mg/kg).

Spinosad is registered in the USA for use on maize at 0.11 kg ai/ha with harvesting permitted 28 days after the final application. In five trials in which the application rate was 0.50 kg ai/ha and the PHI 27–30 days, the concentrations of residues were all below the LOQ (0.01 mg/kg).

The Meeting agreed that the concentration of residues in maize is effectively zero because rates higher than that in GAP did not produce concentrations exceeding the LOQ. The Meeting estimated a maximum residue level of 0.01\* mg/kg and an STMR value of 0 mg/kg for spinosad in maize.

Spinosad is registered in the USA for use on sorghum at 0.11 kg ai/ha with harvesting permitted 7 days after the final application. In eight trials that matched GAP, the concentrations of spinosad residues in sorghum, in ranked order, were 0.03, 0.088, 0.12, 0.16, 0.17, 0.18, 0.47 and 0.68 mg/kg.

The Meeting estimated a maximum residue level of 1 mg/kg and an STMR value of 0.165 mg/kg for spinosad in sorghum.

Spinosad is registered in the USA for use on wheat at 0.11 kg ai/ha with harvesting permitted 21 days after the final application. The Meeting was unable to evaluate the trials on wheat in the USA, because spinosad was used at 0.50 kg ai/ha.

#### *Almonds*

Spinosad is registered in the USA for use on almonds at 0.18 kg ai/ha with harvesting permitted 14 days after the final application. The concentrations of spinosad residues in almond kernels were below the LOQ (0.01 mg/kg) in 12 trials that were in line with GAP conditions.

The Meeting estimated a maximum residue level of 0.01\* mg/kg and an STMR value of 0.01 mg/kg for spinosad in almonds.

#### *Cotton seed*

In Australia, spinosad is registered for use on cotton at 0.10 kg ai/ha with harvesting permitted 28 days after the final application. In six trials in Australia that matched GAP conditions, the concentrations of spinosad residues in cotton seed were below the LOQ (0.01 mg/kg). In six trials at higher application rates (two trials at 0.15 kg ai/ha and four trials at 0.20 kg ai/ha) with harvesting 28 days after the final application, the concentrations of residues in cotton seed were all below the LOQ (0.01 mg/kg).

Spinosad is registered in Brazil for use on cotton, with application at 0.072 kg ai/ha and a 7-day PHI. In two trials in Brazil with a 7-day PHI and application rates of 0.072 and 0.14 kg ai/ha, the concentrations of spinosad residues in cotton seed were below the LOQ (0.01 mg/kg).

Spinosad is registered in the USA for use on cotton at 0.10 kg ai/ha with harvesting permitted 28 days after the final application. The concentrations of spinosad residues in cotton seed were below the LOQ (0.01 mg/kg) in 19 trials in which the PHI was 28 days but with various application rates (one trial at 0.10 mg/kg, one at 0.12 kg ai/ha, 14 at 0.125 kg ai/ha, one at 0.14 kg ai/ha and two at 0.20 kg ai/ha).

In summary, the concentrations of spinosad residues in cotton seed were below the LOQ (0.01 mg/kg) in 33 trials on cotton. The Meeting noted that, as residues did reach cotton seed in a processing trial with an exaggerated application rate, the concentration could not be considered to be effectively zero.

The Meeting estimated a maximum residue level of 0.01\* mg/kg and an STMR value of 0.01 mg/kg for spinosad in cotton seed.

#### *Maize forage and fodder*

In the USA, a 7-day PHI is required for use of spinosad on maize (field corn) for maize forage. In 12 trials on sweet corn that matched GAP requirements for maize forage, the concentrations of spinosad residues in sweet corn forage, in ranked order, were 0.12, 0.074, 0.087, 0.098, 0.099, 0.16, 0.17, 0.18, 0.36, 0.44, 0.48 and 0.49 mg/kg (fresh weight) and 0.12, 0.41, 0.49, 0.50, 0.53, 0.67, 0.72, 1.2, 1.3, 2.3, 2.8 and 3.1 mg/kg (dry weight).

The Meeting estimated a maximum residue level of 5 mg/kg and an STMR value of 0.70 mg/kg for spinosad in maize forage.

In the USA, a 28-day PHI is required for use of spinosad on maize (field corn) for maize fodder. In 12 trials on sweet corn that matched GAP requirements for maize fodder, the concentrations of spinosad residues in sweet corn stover, in ranked order, were 0.03, 0.053, 0.074, 0.097, 0.099, 0.11, 0.12, 0.17 (2), 0.23, 0.46 and 0.68 mg/kg (fresh weight) and 0.13, 0.15, 0.21, 0.29, 0.38, 0.41, 0.52, 0.61 (2), 0.81, 0.92 and 2.1 mg/kg (dry weight).

The Meeting estimated a maximum residue level of 5 mg/kg and an STMR value of 0.46 mg/kg for spinosad in maize fodder.

*Sorghum forage*

In the USA, a 7-day PHI is required for use of spinosad on sorghum for fodder and a 14-day PHI for forage. The concentrations of spinosad residues in sorghum forage in four trials that matched GAP were 0.052, 0.078, 0.095 and 0.18 mg/kg (fresh weight) and 0.18, 0.24, 0.28 and 0.47 mg/kg (dry weight).

The Meeting agreed that the number of supervised trials was insufficient to recommend a maximum residue level.

*Wheat forage, hay and straw*

In the USA, an application rate of 0.11 kg ai/ha is required for use of spinosad on wheat, with a 21-day PHI required for grain and straw and 14 days for forage and hay. The concentrations of spinosad residues in wheat forage in six trials that matched GAP were < 0.01 (2), 0.01 (2), 0.05 and 0.054 mg/kg (fresh weight) and 0.04, < 0.05 (2), 0.06, 0.22 and 0.23 mg/kg (dry weight). The Meeting noted that the concentration in forage was lower than that in straw and fodder.

The concentrations of spinosad residues in six trials in the USA that matched GAP were < 0.01, 0.019, 0.05, 0.052, 0.15 and 0.17 mg/kg (fresh weight) and < 0.02, 0.03, 0.06, 0.13, 0.21 and 0.27 mg/kg (dry weight) in wheat hay; and < 0.01, 0.19, 0.37, 0.53, 0.56 and 0.73 (fresh weight) and < 0.02, 0.22, 0.41, 0.59, 0.64 and 0.83 mg/kg (dry weight) in wheat straw. The combined data for wheat straw and hay were < 0.02 (2), 0.03, 0.06, 0.13, 0.21, 0.22, 0.27, 0.41, 0.59, 0.64 and 0.83 mg/kg (dry weight).

The Meeting estimated a maximum residue level of 1 mg/kg and an STMR value of 0.215 mg/kg for spinosad in wheat straw and fodder.

*Almond hulls*

Spinosad is registered in the USA for use on almonds at 0.18 kg ai/ha with harvesting permitted 14 days after the final application. In 12 trials in line with GAP conditions, the concentrations of spinosad residues in almond hulls, in ranked order, were 0.20, 0.27, 0.28, 0.37, 0.45, 0.49, 0.62, 0.67, 0.69, 0.73, 0.82 and 1.1 mg/kg.

The Meeting estimated a maximum residue level of 2 mg/kg and an STMR value of 0.56 mg/kg for spinosad in almond hulls.

*Fate of residues during processing*

The Meeting received information on the fate of incurred residues of spinosad during the processing of apples, oranges, grapes, tomatoes and cotton seed. Processing factors were calculated for commodities derived from these raw agricultural commodities on the basis of the concentrations of residues of spinosyn A or spinosyns A and D measured by HPLC or of spinosad by immunoassay. The results for spinosyn A or the sum of spinosyns A and D were similar, except at low concentrations, where analytical errors and rounding of data influenced the results. When the concentration of residues in a processed commodity was below the LOQ, the processing factor was calculated from the LOQ and was prefixed with a 'less than' symbol (<).

The processing factors for apples were 2.1 and 5.2 (mean, 3.9) for processing to wet pomace, < 0.09 and 0.07 (mean, 0.08) to juice and 0.09 to purée. Application of these factors to the STMR value for apples resulted in STMR-P values of 0.064 mg/kg for wet apple pomace, 0.0013 mg/kg for apple juice and 0.0015 mg/kg for apple purée.



The processing factors for oranges were  $< 0.13$  to juice and 2.2 to dried pulp. Application of these factors to the STMR value for citrus whole fruit resulted in STMR-P values of 0.007 mg/kg for orange juice and 0.12 mg/kg for dried processed citrus pulp.

The processing factors for tomatoes to juice were 0.026 and 0.25; as the two values did not agree, the Meeting agreed to choose the higher value. The processing factors for tomatoes to purée were 0.18 and 0.58, and again the higher value was chosen. The processing factor for tomato to paste was 1.94. Application of these factors to the STMR value for tomatoes resulted in STMR-P values of 0.0075 mg/kg for tomato juice, 0.017 mg/kg for tomato purée and 0.059 mg/kg for tomato paste.

Application of the processing factors for cotton seed to hulls (0.20), meal ( $< 0.17$ ), crude oil (0.18) and refined oil (0.20) to the STMR value for cotton seed resulted in STMR-P values of 0.0020 mg/kg for hulls, 0.0017 mg/kg for meal, 0.0018 mg/kg for crude oil and 0.0020 mg/kg for refined oil.

The Meeting recommended MRLs of 0.01\* mg/kg for crude and edible cotton seed oils on the basis of the LOQ of the available analytical method.

### ***Residues in animal commodities***

#### ***Direct treatment of farm animals***

Spinosad is registered for direct use on sheep in Australia, by jetting and wound dressing. The jetting mixture contains 25 mg ai/l and is applied at a rate of 0.5 l per month of wool growth.

In a trial in Australia that matched label instructions, long-wool sheep were treated with a hand-held jetting applicator delivering 5.1 l in a 21-s application time for each sheep. Five animals were slaughtered 5, 12, 15 and 21 days after treatment, and residues were measured in the tissues. The concentrations of spinosad residues were below the LOQ (0.01 mg/kg) in muscle, kidney, liver, back fat and perirenal fat in all samples.

The Meeting estimated a maximum residue level of 0.01\* mg/kg for spinosad residues in sheep meat (fat) and sheep offal.

The STMR concept is designed for use in supervised field trials on crops to obtain the typical residue value when a pesticide is used at maximum GAP. The method is not directly applicable to a trial of single direct treatment of animals. However, the Meeting agreed that a typical residue value for a pesticide used directly on animals (at maximum label conditions) would be useful in estimating long-term dietary intake. The Meeting estimated a typical concentration of spinosad residues (from direct use at maximum label conditions) of 0.01 mg/kg in sheep meat and sheep offal.

In the USA, beef and dairy cattle may be treated directly with spinosad in an aqueous suspension formulation as a pour-on. The permitted application rate is 2 mg ai/kg bw, and no restrictions on milk or slaughter intervals are imposed. Animals may also be sprayed (at a concentration of 400 mg/l) at a rate of 0.76 g ai/animal. Spinosad is also approved for treatment of animal housing.

In trials in the USA, groups of Holstein dairy cows underwent five cycles of each of three treatments: (1) body spray with 2 l at 400 mg ai/l every 7 days; (2) body spray with 5 l at 400 mg ai/l every 21 days; or (3) pour-on at 2 mg ai/kg bw every 14 days. The housing was sprayed every 7 days. Residues were measured in milk throughout the study. The animals were slaughtered for tissue collection at intervals after a cycle of treatments. Muscle, kidney, liver and milk were analysed by

immunoassay and fat by HPLC; the values reported are for the sum of spinosyns A, D, B and B of D. The concentrations of residues arising from treatments 1 and 2 were similar, but both were much lower than that from treatment 3. The highest concentrations observed after treatment 3 were 0.28 mg/kg in muscle, 0.87 mg/kg, in kidney, 1.2 mg/kg in liver, 2.7 mg/kg in renal fat, 2.2 mg/kg in subcutaneous fat and 0.65 mg/kg in milk.

The Meeting estimated maximum residue levels of 3 mg/kg for cattle meat (fat), 1 mg/kg for cattle kidney, 2 mg/kg for cattle liver and 1 mg/kg for cattle milk.

As for the sheep treatments, the Meeting agreed that a typical residue value for a pesticide used directly on animals (at maximum label conditions) would be useful in estimating long-term dietary intake. In this case, the median concentration of residues in the tissues of the three animals slaughtered at the shortest interval after treatment (or later if the values were higher later) was taken to represent that typical value. For milk, the highest average concentration for the group on the day after treatment (or later if the values were higher later) was taken to represent the typical value.

The Meeting estimated typical concentrations of spinosad residues (from direct use at maximum label conditions) of 0.078 mg/kg for cattle meat, 0.31 mg/kg for kidney, 0.66 mg/kg for liver and 0.65 mg/kg for milk. These values can be used in the same way as STMR values for estimating long-term dietary intake.

#### *Dietary burden of farm animals*

The Meeting estimated the dietary burden of spinosad residues in farm animal on the basis of the diets listed in Appendix IX of the *FAO Manual*. Calculation from MRLs and STMR-P values provides the levels in feed suitable for estimating MRLs for animal commodities, while calculation from STMR and STMR-P values for feed is suitable for estimating STMR values for animal commodities. The percentage of dry matter is taken as 100% when MRLs and STMR values are already expressed as dry weight.

The concentrations of spinosad residues in milk reached a plateau after about 6 days, i.e. relatively rapidly. The maximum residue levels in animal commodities were derived from the MRLs, as stated by the 1997 JMPR.

#### *Estimated maximum dietary burden of farm animals*

Commodity	Group	Residue (mg/kg)	Basis	Dry matter (%)	Residue, dry weight (mg/kg)	Choose diets (%)			Residue contribution (mg/kg)		
						Beef cattle	Dairy cows	Poultry	Beef cattle	Dairy cows	Poultry
<hr/>											
Apple pomace wet	AB	0.064	STMR-P	40	0.16	10			0.016		
Citrus pulp	AB	0.12	STMR-P	91	0.13						
Maize forage	AF	5	MRL	100	5.0	40	50		2.0	2.5	
Maize fodder	AS	5	MRL	100	5.0						
Wheat straw and fodder, dry	AS	1	MRL	100	1.0						

Commodity	Group	Residue (mg/kg)	Basis	Dry matter (%)	Residue, dry weight (mg/kg)	Choose diets (%)			Residue contribution (mg/kg)		
						Beef cattle	Dairy cows	Poultry	Beef cattle	Dairy cows	Poultry
Sorghum	GC	1	MRL	86	1.2	40	40	80	0.47	0.47	0.93
Almond hulls	AM	2	MRL	90	2.2	10	10		0.22	0.22	
Cotton seed hulls		0.0020	STMR-P	90	0.0022						
Cotton seed meal		0.0017	STMR-P	88	0.0019			20			0.0004
Total						100	100	100	2.7	3.2	0.93

*Estimated STMR dietary burden of farm animals*

Commodity	Group	Residue (mg/kg)	Basis	Dry matter (%)	Residue, dry weight (mg/kg)	Choose diets (%)			Residue contribution (mg/kg)		
						Beef cattle	Dairy cows	Poultry	Beef cattle	Dairy cows	Poultry
Apple pomace wet	AB	0.064	STMR-P	40	0.16	10			0.016		
Citrus pulp	AB	0.12	STMR-P	91	0.13						
Maize forage	AF	0.70	STMR	100	0.70	40	50		0.28	0.35	
Maize fodder	AS	0.46	STMR	100	0.46						
Wheat straw and fodder, dry	AS	0.215	STMR	100	0.22						
Sorghum	GC	0.165	STMR	86	0.19	40	40	80	0.08	0.08	0.15
Almond hulls	AM	0.56	STMR	90	0.62	10	10		0.062	0.062	
Cotton seed hulls	SO	0.0020	STMR-P	90	0.0022						
Cotton seed meal	SO	0.0017	STMR-P	88	0.0019			20			0.00039
Total						100	100	100	0.43	0.49	0.15

The dietary burdens of spinosad for estimating MRLs and STMR values for animal commodities (residue concentrations in animal feeds expressed as dry weight) are: 2.7 and 0.43 mg/kg for beef cattle, 3.2 and 0.49 mg/kg for dairy cattle and 0.93 and 0.15 mg/kg for poultry.

### Feeding studies

The Meeting received information on the concentrations of residues arising in tissues and milk when dairy cows were dosed with spinosad in capsules at the equivalent of 1, 3 or 10 ppm in the diet for 28 days. The concentrations in fat were higher than those in other tissues. The transfer factors (concentration of residue in tissue ÷ concentration in feed) for tissues and milk were reasonably consistent at the three dietary levels: fat, 0.65, 0.37, 0.57, mean 0.53; muscle, 0.026, 0.018, 0.028, mean 0.024; kidney, 0.073, 0.095, 0.087, mean 0.085; liver, 0.16, 0.16, 0.18, mean 0.17, milk 28 days, 0.044, 0.048, 0.049, mean 0.047; cream 28 days, 0.18, 0.20, 0.19, mean 0.19.

The average concentration in milk (day 14, HPLC analysis) from the three animals at 1 ppm was 0.044 mg/kg, and that in milk from cows at 3 ppm was 0.13 mg/kg. The highest individual concentrations (HPLC analysis) at 3 ppm in the diet were 1.7 mg/kg in fat, 0.069 mg/kg in muscle, 0.44 mg/kg in liver and 0.26 mg/kg in kidney. The mean concentrations (HPLC analysis) in the three animals at 1 ppm were 0.65 mg/kg in fat, 0.020 mg/kg in muscle, 0.13 mg/kg in liver and 0.065 mg/kg in kidney.

The Meeting received information on the concentrations of residues in tissues and eggs after laying hens were dosed with spinosad at the equivalent of 0.1, 0.3, 1 or 5 ppm in the diet for 41 days. At the lower feeding levels, the concentrations of residues were often below the LOQ of the analytical method. The values in fat were substantially higher than those in other tissues and eggs. The concentrations in fat from hens at 5 ppm were 8.7 and 7.0 times higher than those in hens at 1 ppm in abdominal and subcutaneous fat, respectively, slightly more than the five times that was expected. The concentrations of residues in eggs from hens at 5 ppm reached a plateau by day 13, but the values in eggs were generally below the LOQ (0.01 mg/kg) at lower dietary concentrations.

### Maximum residue levels

As the maximum dietary burdens of beef and dairy cattle were 2.7 and 3.2 mg/kg, respectively, the concentrations of residues in tissues and milk were taken as those seen at the dietary concentration of 3 ppm, without interpolation. As the STMR dietary burdens (0.43 and 0.49 mg/kg) were lower than the lowest dietary concentration, 1 ppm, the resulting residues in tissues and milk were calculated by applying the transfer factors (concentration of residue in tissue or milk ÷ concentration in feed) found at the lowest dietary concentration to the STMR dietary burdens.

The highest individual tissue concentration of residue at the relevant dietary concentration was used in conjunction with the highest dietary burden of residue to calculate the likely highest residue in animal commodities. The mean concentration of residue in tissues from animals at the relevant dietary concentration was used in conjunction with the STMR dietary burden to estimate the STMR values for animal commodities. For milk, the mean concentration of residue at the plateau for the relevant dietary concentration was used to estimate both the highest residue and the STMR values. As the STMR burden of dairy cows exceeds that of beef cattle, it was used to estimate the STMR value in fat, muscle, liver and kidney.

Feeding level (ppm) <i>Interpolated / actual</i>	Residue concentration (mg/kg)									
	Milk (mean)		Fat		Muscle		Liver		Kidney	
			Highest	Mean	Highest	Mean	Highest	Mean	Highest	Mean
MRL beef cattle 2.7 / 3										
MRL dairy cows	0.13 /		1.7	/ 1.7	0.069 /		0.44 /		0.26 /	

3.2 / 3	0.13	0.069	0.44	0.26	
STMR beef cattle					
0.43 / 1					
STMR dairy cows	0.022 /	0.32 /	0.010 /	0.064 /	0.032 /
0.49 / 1	0.044	0.65	0.020	0.13	0.065

The maximum concentrations of residues expected in tissues are: 1.7 mg/kg in fat, 0.069 mg/kg in muscle, 0.26 mg/kg in kidney, 0.44 mg/kg in liver and 0.13 mg/kg in milk.

The Meeting estimated maximum residue levels of 2 mg/kg for cattle meat (fat), 0.5 mg/kg for cattle kidney, 0.5 mg/kg for cattle liver and 0.2 mg/kg for milk.

The STMR dietary burden for beef and dairy cattle is 0.5 mg/kg (the higher of the two values). As the transfer factors were reasonably consistent across dietary levels, the Meeting agreed that extrapolation below the lowest concentration (1 ppm) was appropriate. The Meeting estimated STMR values of 0.32 mg/kg for cattle fat, 0.010 mg/kg for cattle meat, 0.032 mg/kg for cattle kidney, 0.064 mg/kg for cattle liver and 0.022 mg/kg for cattle milk.

The concentrations of residues arising from direct treatment of animals were higher than those resulting from feed intake. The recommended MRLs are therefore based on the direct treatments. Similarly, the estimates for typical concentrations of spinosad residues (from direct use at maximum label conditions) should be used for estimating long-term intake in place of STMR values derived from the dietary burden of farm animals and animal feeding studies.

As the maximum dietary burden of poultry was 0.93 mg/kg, the concentrations of residues in tissues and eggs can be taken directly from the study in which hens were fed a diet containing 1 ppm, without interpolation, where the highest concentrations of residues were < 0.01 mg/kg in muscle, 0.16 mg/kg in fat, 0.01 mg/kg in liver and 0.01 mg/kg in eggs.

The Meeting estimated maximum residue levels of 0.2 mg/kg for poultry meat (fat) and 0.01 mg/kg for eggs. As the STMR dietary burden for poultry was 0.24 mg/kg, the concentrations of residues in tissues and eggs can be taken directly from the study in which hens were fed a diet containing 0.3 ppm, without interpolation. The Meeting estimated STMR values of 0.01 mg/kg for poultry meat, 0.05 mg/kg for poultry fat, 0.01 mg/kg for poultry liver and 0.01 mg/kg for eggs.

## Recommendations

On the basis of the data from supervised trials, the Meeting concluded that the residue concentrations listed below are suitable for establishing MRLs and for assessing the IEDIs.

*Definition of the residue (for compliance with MRL and for estimation of dietary intake): sum of spinosyn A and spinosyn D. The residue is fat-soluble, but residues in milk should be measured in whole milk.*

Commodity		MRL (mg/kg)	STMR or STMR-P (mg/kg)
CCN	Name		
AM 0660	Almond hulls	2	0.56
TN 0660	Almonds	0.01*	0.01
JF 0226	Apple juice		0.0013
	Apple pomace, wet		0.064

Commodity		MRL (mg/kg)	STMR or STMR-P (mg/kg)
CCN	Name		
	Apple puree		0.0015
FP 0226	Apple	0.1	0.0165
VB 0040	Brassica vegetables, head cabbages, flowerhead	2	0.27
	Brassicas		
MO 1280	Cattle kidney	1 <sup>a</sup>	0.31 <sup>b</sup>
MO 1281	Cattle liver	2 <sup>a</sup>	0.66 <sup>b</sup>
MM 0812	Cattle meat	3 (fat) <sup>a</sup>	0.078 <sup>b</sup>
ML 0812	Cattle milk	1 <sup>a</sup>	0.65
VS 0624	Celery	2	0.97
FC 0001	Citrus fruits	0.3	0.01
AB 0001	Citrus, dried processing pulp		0.12
SO 0691	Cotton seed	0.01*	0.01
	Cotton seed hulls		0.0020
	Cotton seed meal		0.0017
OC 0691	Cotton seed oil, crude	0.01*	0.0018
OR 0691	Cotton seed oil, edible	0.01*	0.0020
PE 0112	Eggs	0.01	0.01
VC 0045	Fruiting vegetables, cucurbits	0.2	0.046
FI 0341	Kiwifruit	0.05	0.02
VL 0053	Leafy vegetables	10	1.9
VP 0060	Legume vegetables	0.3	0.041
GC 0645	Maize	0.01*	0
AS 0645	Maize fodder (dry)	5	0.46
AF 0645	Maize forage (dry)	5	0.70
JF 0004	Orange juice		0.0072
VO 0051	Peppers	0.3	0.056
VR 0589	Potato	0.01*	0
PM 0110	Poultry meat	0.2 (fat)	0.01
MO 0822	Sheep, edible offal of	0.01* <sup>a</sup>	0.01
MM 0822	Sheep meat	0.01* (fat) <sup>a</sup>	0.01
GC 0651	Sorghum	1	0.165
VD 0541	Soya bean (dry)	0.01*	0
FS 0012	Stone fruits	0.2	0.0265
VO 0447	Sweet corn (corn-on-the-cob)	0.01*	0.01
VO 0448	Tomato	0.3	0.03
JF 0448	Tomato juice		0.0075
	Tomato paste		0.059
	Tomato puree		0.017
AS 0654	Wheat straw and fodder, dry	1	0.215

\* The MRL is estimated at or about the LOQ.

<sup>a</sup> The MRL accommodates external animal treatment.

<sup>b</sup> Residues from direct animal treatment, not an STMR, but median concentration of residues from animals in a treatment group.

### **Dietary risk assessment**

#### *Chronic intake*

The evaluation of spinosad resulted in recommendations for new MRLs and STMR values for raw and processed commodities. Data on consumption were available for 29 food commodities and were used to calculate dietary intake. The results are shown in Annex 3 (Report 2001).

The IEDIs in the five GEMS/Food regional diets, based on estimated STMrs were 2–30% of the ADI (0–0.02 mg/kg bw). The Meeting concluded that long-term intake of residues of spinosad from uses that have been considered by the JMPR is unlikely to present a public health concern.

*Short-term intake*

The 2001 JMPR concluded that it was unnecessary to establish an acute RfD for spinosad. The Meeting therefore concluded that short-term dietary intake of spinosad residues is unlikely to present a risk to consumers.

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