SPINOSAD (203)

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EXPLANATION

Spinosad was first evaluated by the JMPR in 2001. An ADI of 0-0.02 mg/kg bw was estimated and an acute reference dose was determined to be unnecessary. MRLs were recommended for many crops and animal commodities.

The CCPR (35th Session, 2003, paragraph 135, ALINORM 03/24A) advanced the draft MRL for spinosad for cattle milk to Step 5 while noting the concerns of some delegations regarding the high MRL in milk (equivalent to 25 mg/kg in the fat).

The CCPR (36th Session, 2004, paragraph 168, ALINORM 04/27/24) recalled earlier discussions on the expression of MRLs for milk of partially fat-soluble pesticides and decided that the MRL for spinosad in milk should be returned to Step 6, and requested the JMPR to consider further how MRLs should be expressed in milk and milk fat.

Information on registered uses and data from supervised residue trials on grapes and stored grain, and the direct uses of spinosad on sheep for blowfly and lice control with supporting residue data were reported to the Meeting.

RESIDUE ANALYSIS

Analytical methods

Analytical method GRM 00.04 was used for the analysis of cereal grains and processed products in the storage and processing trials. Its description and validation are summarized below.

HPLC analytical methods for spinosad in animal commodities and their validation were provided in conjunction with the direct animal treatment trials on sheep. They are in the revised procedure, an HPLC-MS finish replaces an HPLC-UV finish with the remainder of the method essentially unchanged.

***Cereal grains*, wheat, maize, rice, barley, oats and processed commodities (GRM 00.04, Hastings and Clements, 2000)**

Analytes:	spinosyns A and D	HPLC-MSD	GRM 00.04
LOQ:	0.01 mg/kg		
Precision	Repeatability S_r: 0.075-0.10 for individual spinosyns A, D, K, B and N-demethyl D in cereal grain, pulse and forage samples at 0.01, 0.1 and 1 mg/kg (n = 33) for two analysts on three batches of samples. (S_r is relative standard deviation.)		
Recoveries	Cereal grains and pulses: 85-114% (n = 15) for individual spinosyns A, D, K, B and N-demethyl D at 0.01, 0.1 and 1 mg/kg.		
Description	Based on GRM 95.03, evaluated by the 2001 JMPR. Samples were extracted with acetonitrile + water (80% + 20%). An aliquot from the extract was diluted with acetonitrile and cleaned up on a strong cation exchange solid-phase extraction column. The spinosyns (A, D, K, B and *N*-demethyl D) were eluted with dilute ammonium acetate in acetonitrile + methanol, the eluate evaporated and the residue re-dissolved for analysis by HPLC with a mass-selective detector.		

Fat, muscle, liver, kidney **(ELS1303, Hacket *et al*., 2003).**

Analytes:	spinosyns A and D	HPLC-MS	STMCR 428
LOQ:	0.01 mg/kg		
Precision	Repeatability S_r : 0.058 for recovery tests at 0.01 mg/kg		
Recoveries	Fat, muscle, liver and kidney: 76-110% (n = 30) for spinosyn A + spinosyn D at 0.01 mg/kg; 80-107% (n = 13) at 0.1 mg/kg,		
Description	Muscle, liver and kidney were extracted with acetonitrile + water (80% + 20%), and fat with hexane + dichloromethane (60% + 40%). An aliquot from the extract was purified by liquid-liquid partition and silica clean-up. After the eluate was taken to dryness, the residue was taken up in methanol + acetonitrile + 2% ammonium acetate for HPLC-MS analysis.		

Bovine milk **(STMCR 428, Mooney, 2002)**

Analytes:	spinosyns A and D	HPLC-MS	STMCR 428
LOQ:	0.005 mg/kg		
Precision	Repeatability S_r : 0.057 for recovery tests at 0.005 mg/kg (Spinosyns A and D).		
Recoveries	Spinosyn A at 0.005 mg/kg: 96-107% (n=6) Spinosyn D at 0.005 mg/kg: 79-94% (n=6) Spinosyn A at 0.01-0.02 mg/kg: 88-109% (n=18) Spinosyn D at 0.01-0.02 mg/kg: 84-113% (n=18) Data were also available for spinosyns B and B of D.		
Description	Milk or cream were shaken with acetonitrile and centrifuged, and an aliquot of the supernatant liquid purified and analysed as above for muscle.		

USE PATTERN

Spinosad is registered in a number of countries for use on grapes for the control of light brown apple and grape berry moths, western flower and grape thrips, Helicoverpa armigera, grape leafroller and skeletonizer, omnivorous leafroller and orange tortix. Registered uses on grapes are summarized in Table 1.

Spinosad is used in the USA for the control of grain-storage pests in barley, corn, millet, oats, rice, sorghum and wheat (Table 2).

It is also registered for external animal treatment for the control of flystrike and lice on sheep in Australia (Table 3). Spinosad may be applied by jetting, in a plunge- or shower-dip, as a flystrike dressing, or by aerosol application to a flystrike wound.

Table 1. Registered uses of spinosad on grapes. All foliar applications.

Country	Formulation	Application		No.	PHI, days
		Rate, kg ai/ha	Spray conc. kg ai/hl		
Australia L	SC 120		0.0048		14
Chile	SC 480	0.060-0.072			14
Croatia	SC 240	0.029-0.072			21
Cyprus L	SC 480	0.036-0.072	0.0072		7
Cyprus	DP 0.15%	0.006		3	N/A
Greece L	SC 480		0.0048-0.012	4	14
Israel L	SC 480		0.0048-0.0096	3	7
Italy L	SC120	0.12	0.012	5	15
Italy L	SC 480		0.0048-0.012	5	15
Lebanon	SC 480		0.0036-0.0048		7
Romania	SC 240	0.036-0.048		3	21
South Africa L	SC 480		0.0072-0.0096	2	42
South Africa L	WP 800		0.0072-0.0096	2	42
Turkey	SC 480	0.024			14
USA L	SC 240	0.07-0.14		*	7

L: Label or label copy provided.

* Maximum seasonal application: 0.49 kg ai/ha

Table 2. Registered post-harvest uses of spinosad on stored grain[1] in the USA. Label or label copy provided.

Form	Application			Storage interval
	Method	Rate, g ai/t grain	Contact time	
SC 240	Coarse spray	1	Coarse spray to moving stream of grain	No minimum interval
SC 240	Top dressing	1	Apply ½ of mixture across surface and rake to a depth of ~10 cm, and rest to surface and leave undisturbed.	

[1] Stored grain: barley, bird seed, corn, cotton, millet, oats, peanuts (in hull), rice, sorghum, soybeans, sunflowers and wheat.

Table 3. Registered uses of spinosad for the external treatment of sheep for lice and blowfly in Australia (label or label copy provided). For listed uses withholding period before slaughter is 0 days. Label restraints include: do not use on sheep producing milk.

Form	Application	
	Method and rate	Concn
SC 25 g/l	Jetting, for long-wool treatment with jetting apparatus, apply 0.5 l of fluid for each month of wool growth (up to 5 l per sheep) from poll to tail base, around neck and along each side, ensuring saturation to skin.	25 mg ai/l
SC 25 g/l	Plunge dip (2-6 weeks after shearing). Sheep should swim 9 m, head of each sheep to be dunked twice after entering the dip. Inspect sheep to ensure wetting.	Initial 10 mg ai/l, 15 mg ai/l constant replenishment
SC 25 g/l	Shower dip (2-6 weeks after shearing). Dip should be filled to the recommended capacity and run for 12 min to ensure wetting. Sump should not fall by more than 25%. Inspect sheep to ensure wetting.	Initial 20 mg ai/l, 40 mg ai/l constant replenishment
SC 25 g/l	Flystrike dressing. Remove wool from around wound using clippers or shears. Apply 1-2 litres onto wound.	25 mg ai/l
SC 25 g/l	Mulesing[1] wound dressing. Spray 40-80 ml onto wound and 25 mm into surrounding wool	125 mg ai/l
AE 2.8 g/l[2]	Aerosol can spray. A typical clipped flystrike wound of 200 cm^2 area should take 6 seconds to treat.	

[1] Surgical removal of folds of skin in the breech of sheep as measure against flystrike. Named after JHW Mules, 1876-1946, South Australian sheep-raiser who first practised this procedure. Macquarie Dictionary.

[2] Registered product 56734 - Australian Pesticides and Veterinary Medicines Authority, 10-Aug-04. NB Label copy not provided.

RESIDUES RESULTING FROM SUPERVISED TRIALS

The Meeting received information on supervised trials for the uses of spinosad on grapes and cereal grains (see 'Fate of residues in storage and processing' below).

Trials were well documented with laboratory and field reports. The former included method validation including procedural recoveries with spiking at residue levels similar to those occurring in samples from the supervised trials. Dates of analyses or duration of sample storage were also provided. Although trials included control plots, no control data are recorded in the Tables because no residues in control samples exceeded the LOQ. Residues are unadjusted for recoveries.

When residues were not detected they are shown as below the LOQ (e.g. <0.01 mg/kg). Residues, application rates and spray concentrations have generally been rounded to two significant figures or, for residues near the LOQ, to one significant figure. Residues from the trials conducted

according to maximum GAP have been used for the estimation of maximum residue levels. These results are double underlined.

Periods of freezer storage between sampling and analysis were recorded for all trials and were covered by the periods of the freezer storage stability studies.

Grapes. Twelve US supervised residue trials conducted in five States on the use of spinosad on grapes were reported to the Meeting. Spinosad was applied by tractor-mounted sprayers and backpacks to single unreplicated plots of 33 to 176 square m. The results are shown in Table 4. Samples were analysed by immunoassay method GRM 96.11 which was evaluated by the 2001 JMPR. The relevant Table from the 2001 Evaluations is included for convenience as

Table **5**.

Table 4. Spinosad residues in grapes and grape products after four applications of SC 240 formulation in supervised trials in the USA.

Location , year, (variety)	Application		PHI days	Commodity	Spinosyns, mg/kg Immunoassay	Ref
	kg ai/ha	water, l/ha				
CA 1999 (Thompson Seedless)	0.14	1340	7	grapes juice raisins	0.17 0.57 0.27	06851.99-CA85
CA 1999 (Thompson Seedless)	0.14	1320	7	grapes	0.22	06851.99-CA86
CA 1999 (Perlette)	0.14	1330	8	grapes	0.03	06851.99-CA117
ID 1999 (Chardonnay)	0.14	1120	7	grapes	0.23	06851.99-ID17
NJ 1998 (Concord)	0.10	410	6	grapes	<0.01	06851.98-NJ22
NY 1998 (Chardonnay)	0.10	1020	7	grapes juice	0.05 0.069	06851.98-NY19
WA 1999 (Concord)	0.14	1050	6	grapes	0.13	06851.99-WA35
CA 2000 (Sauvignon Blanc Musque)	0.14	470	7	grapes	0.39	A6851.00-CA134
CA 2000 (Thompson Seedless)	0.14	760	7	grapes	0.082	A6851.00-CA135
CA 2000 (Flame Seedless)	0.14	750	7	grapes	0.077	A6851.00-CA136
CA 2000 (Thompson Seedless)	0.14	720	7	grapes	0.086	A6851.00-CA137
CA 2000 (Sauvignon Blanc)	0.14	480	7	grapes	0.02	A6851.00-CA138

Table 5. Spinosad residues in grapes from supervised trials in France, Italy and Spain (Table 39, JMPR Evaluations, 2001). References are not listed at end.

Country, year (variety)	Application [1]					PHI, days	Spinosyn residues, mg/kg				Ref.
	Form	kg ai/ha	kg ai/hl	water, l/ha	no.		A HPLC	D HPLC	Total [2] HPLC	IA [3]	
France, 1997 (Chenin, Riparia Gloire)	SC 480	0.060	0.029	210	4 5	16 0 5 10 14	<0.01 0.03 0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01 <0.01	<0.01 0.05 0.01 <0.01 <0.01	<0.01 0.08 0.02 <0.01 <0.01	GHE-P-7575
France, 1997 (Gamay)	SC 480	0.060	0.032	190	4 5	17 0 5 10 15	<0.01 0.08 0.02 0.02 0.01	<0.01 0.02 <0.01 <0.01 <0.01	<0.01 0.10 0.03 0.03 0.02	<0.01 0.09 0.04 0.02 0.02	GHE-P-7575

Country, year (variety)	Application [1]					PHI, days	Spinosyn residues, mg/kg				Ref.
	Form	kg ai/ha	kg ai/hl	water, l/ha	no.		A HPLC	D HPLC	Total [2] HPLC	IA [3]	
France, 1997 (Negrette)	SC 480	0.060	0.024	260	5	15[4]	<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 5 10 16	<0.01 0.03 <0.01 <0.01 <0.01	<0.01 0.02 <0.01 <0.01 <0.01	<0.01 0.05 0.02 0.02 <0.01		GHE-P-7850
France, 1998 (Gamay)	SC 480	2×0.096 +3×0.048	2×0.042 +3×0.021	230	4 5	15 0 6 10 15	0.02 0.04 0.03 0.03 0.01	0.02 0.03 0.02 0.02 <0.01	0.04 0.07 0.05 0.05 0.02		GHE-P-7851
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
Italy, 1997 (Tocai Verde)	SC 480	0.060	0.015	400	4 5	22 0 5 10 16	0.01 0.18 0.07 0.04 0.03	<0.01 0.04 0.02 0.01 0.01	0.02 0.24 0.09 0.06 0.04[5]	0.02 0.45 0.15 0.08 0.05[5]	GHE-P-7579
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 5 9 14	0.02 0.18 0.10 0.06 0.03	0.03 0.14 0.08 0.05 0.03	0.05 0.32 0.18 0.11 0.06		GHE-P-7852
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[6]	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 5 10 15	0.08 0.23 0.14 0.12 0.07	0.02 0.06 0.05 0.03 0.02	0.11 0.30 0.19 0.16 0.09[5]	0.12 0.40 0.24 0.17 0.12[5]	GHE-P-7578

Country, year (variety)	Application [1]					PHI, days	Spinosyn residues, mg/kg				Ref.
	Form	kg ai/ha	kg ai/hl	water, l/ha	no.		A HPLC	D HPLC	Total [2] HPLC	IA [3]	
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	0.03 c <0.01	0.07 c 0.02		GHE-P-7854

[1] Application rate, spray concentration and volume per ha were not identical for each application (variation generally within 20%). Single values are for the last application.
[2] Total includes spinosyns A, D, B, N-demethyl-D and K
[3] Immunoassay
[4] Samples stored frozen for approx. 17 months.
[5] Samples stored frozen for approx. 18 months.
[6] Samples analysed by IA method Nov-Dec 97 and by HPLC method Jan-Feb 99
[7] control plot sample

FATE OF RESIDUES IN STORAGE AND PROCESSING

Storage

In a series of storage and processing trials in the USA, spinosad was applied to grain at a target rate of 1 g ai/t (020061, McCormick and Dolder, 2004). In most of the trials 18-23 kg of grain was sprayed in a mixer with a pressurised spray syringe or similar. In two larger scale trials on wheat and maize the moving grain was spray-treated as it entered the augur, and the amounts treated were 9.9 t of maize and 30.9 t of wheat. Maximum and minimum ambient storage temperatures were recorded. Grain moisture in maize at the IN site was 17% when deposited in the storage bin and was reduced to below 15% by aeration or heated air as required, and in the wheat at the KS site was about 15% at the beginning of storage and was maintained below that level by aeration. Grain moisture was not measured in the smaller trials, but as the grain was purchased commercially it would have been 12-15% for storage without spoilage.

Spinosad residue data for stored barley, maize, oats, rice and wheat are shown in Table 6. Samples were analysed by method GRM 00.04 for spinosyns A and D.

Residues in the grain immediately after treatment ranged from 43% to 91% of the target application rate, reflecting the efficiency of application, and in the two larger trials the initial residues were 77% and 87%. Residue levels declined very little during the storage period.

Table 6. Spinosad residues in stored cereal grains (barley, maize, oats, rice and wheat) from supervised trials with post-harvest treatments in the USA.

Location, year (variety)	Application		Wt treated	Storage period, months	Residue, mg/kg			Ref
	Form	g ai/t			Spinosyn A	Spinosyn D	Total	
BARLEY								
CA 2003	SC 240	1	18 kg	0 3	0.59 0.43	0.096 0.068	0.69 0.50	020061-3B2
IN 2003 (Robust)	SC 240	1	18 kg	0 3	0.79 0.70	0.13 0.11	0.91 0.81	020061-3B1
TX 2003	SC 240	1	18 kg	0 3	0.62 0.74	0.10 0.12	0.72 0.86	020061-3B3

Location, year (variety)	Application		Wt treated	Storage period, months	Residue, mg/kg			Ref
	Form	g ai/t			Spinosyn A	Spinosyn D	Total	
MAIZE								
CA 2003	SC 240	1	18 kg	0	0.49	0.078	0.57	020061-3C2
				3	0.51	0.084	0.59	
IN 2003 (Diener 9111)	SC 240	1	18 kg	0	0.39	0.062	0.45	020061-3C1
				3	0.35	0.057	0.41	
IN 2003 (DeKalb 57-01)	SC 240	1.6	9.9 t	0	1.2	0.18	1.4	020061-C
				3	0.67	0.11	0.77	
				6	0.67	0.11	0.77	
				11	0.43	0.070	0.50	
MS 2003	SC 240	1	23 kg	0	0.47	0.074	0.54	020061-C3
				3	0.50	0.082	0.58	
				6	0.37	0.059	0.42	
				11	0.43	0.069	0.50	
TX 2003	SC 240	1	23 kg	13 days	0.51	0.079	0.59	020061-C2
				3	0.78	0.12	0.90	
				6	0.56	0.094	0.66	
				11	0.50	0.080	0.58	
TX 2003 (WMCO)	SC 240	1	18 kg	0	0.54	0.089	0.63	020061-3C3
				3	0.46	0.073	0.54	
OATS								
CA 2003	SC 240	1	18 kg	0	0.41	0.064	0.47	020061-3O2
				3	0.28	0.045	0.33	
IN 2003 (INO 9021)	SC 240	1	18 kg	0	0.58	0.091	0.67	020061-3O1
				3	0.59	0.097	0.69	
TX 2003 (INO 9021)	SC 240	1	18 kg	0	0.59	0.098	0.69	020061-3O3
				3	0.54	0.087	0.63	
RICE								
CA 2003	SC 240	1	18 kg	0	0.42	0.068	0.48	020061-3R1
				3	0.58	0.091	0.67	
MS 2003	SC 240	1	23 kg	0	0.63	0.10	0.73	020061-R2
				3	0.54	0.090	0.63	
				6	0.59	0.095	0.68	
				11	0.80	0.13	0.93	
TX 2003	SC 240	1	14 kg	13 days	0.64	0.11	0.75	020061-R1
				3	0.67	0.11	0.78	
				6	0.64	0.11	0.75	
				11	0.78	0.13	0.91	
WHEAT								
CA 2003	SC 240	1	18 kg	0	0.37	0.062	0.43	020061-3W2
				3	0.29	0.047	0.34	
IN 2003 (D500W-WU5)	SC 240	1	18 kg	0	0.69	0.12	0.81	020061-3W1
				3	0.54	0.090	0.63	
KS 2003	SC 240	1.2	30.9 t	0	0.79	0.13	0.92	020061-W
				3	0.64	0.10	0.74	
				6	0.82	0.13	0.95	
				11	0.61	0.10	0.71	
MS 2003	SC 240	1	23 kg	0	0.63	0.10	0.73	020061-W3
				3	0.64	0.11	0.75	
				6	0.48	0.078	0.56	
				11	0.50	0.083	0.59	
TX 2003	SC 240	1	23 kg	13 days	0.52	0.083	0.61	020061-W2
				3	0.62	0.099	0.72	
				6	0.68	0.11	0.79	
				11	0.42	0.067	0.48	
TX 2003	SC 240	1	18 kg	0	0.60	0.098	0.70	020061-3W3
				3	0.53	0.090	0.62	

Processing

Information on the fate of incurred spinosad residues during the processing of grapes, maize, rice and wheat was reported to the present Meeting. Data on grapes were evaluated by the 2001 Meeting.

Three trials in France and one in Italy on the processing of grapes to pomace and wine were reported to the 2001 JMPR (Residues Evaluations pp.823-824). In all four trials residues of spinosad were below the LOQ (0.01 mg/kg) in all wine samples, but in the grapes were <0.01-0.03 mg/kg, so the best estimated processing factor for wine is <0.33.

Juice and raisins were produced from grapes in two US trials, 06851.99-CA85 and 06851.98-NY19 (Table 4). Calculated processing factors are juice 3.3 and 1.4, and raisins 1.6.

The residues resulting from the milling of maize, rice and wheat and calculated processing factors are shown in Table 7. Residues of spinosad were almost exclusively on the outside of the grain, and were strongly concentrated in the aspirated grain fraction from the milling of maize and wheat. Residue levels in grits and flour were much lower, and the level in wheat bran was essentially the same as in the grain.

Table 7. Spinosad residues in stored cereal grains (maize, rice and wheat) and their processed fractions from milling trials in the USA. Processing factors are calculated as residue levels in processed commodities ÷ residue levels in grain.

Location, year (variety)	Application		Wt treated	Storage, months	Commodity	Residue, mg/kg			Processing factor	Ref
	Form	g ai/t				Spinosyn A	Spinosyn D	Total		
MAIZE										
IN 2003 (DeKalb 57-01)	SC 240	1.6	9.9 t	0	grain	1.2	0.18	1.4		020061-C
					AGF [1]	135	23	158	116	
					grits	0.079	0.013	0.091	0.067	
					meal	0.21	0.034	0.24	0.18	
					flour	0.27	0.050	0.31	0.23	
					oil (dry mill)	0.32	0.055	0.37	0.27	
					starch	0.0019	[2] ndr	0.0019	0.0014	
					oil (wet mill)	1.09	0.14	1.2	0.91	
IN 2003 (DeKalb 57-01)	SC 240	1.6	9.9 t	6	grain	0.670	0.11	0.77		020061-C
					AGF	68	11.5	80	104	
					grits	0.064	0.010	0.074	0.096	
					meal	0.097	0.017	0.11	0.15	
					flour	0.092	0.017	0.11	0.14	
					oil (dry mill)	0.19	0.037	0.22	0.29	
					starch	0.0013	[2] ndr	0.0013	0.0017	
					oil (wet mill)	0.85	0.11	0.96	1.2	
RICE										
TX 2003	SC 240	1	14 kg	13 days	grain	0.64	0.11	0.75		020061-R1
					hulls	1.8	0.31	2.1	2.8	
					bran	0.61	0.099	0.71	0.95	
					brown rice	0.095	0.015	0.11	0.15	
					white rice	0.022	0.0039	0.026	0.035	
TX 2003	SC 240	1	14 kg	6	grain	0.64	0.11	0.75		020061-R1
					hulls	1.8	0.31	2.1	2.8	
					bran	0.40	0.065	0.46	0.62	
					brown rice	0.047	0.0072	0.054	0.072	
					white rice	0.0057	0.0012	0.007	0.0094	

Location, year (variety)	Application		Wt treated	Storage, months	Commodity	Residue, mg/kg			Processing factor	Ref
	Form	g ai/t				Spinosyn A	Spinosyn D	Total		
WHEAT										
KS 2003	SC 240	1.2	30.9 t	6	grain	0.82	0.13	0.95		020061-W
					AGF	260	44	302	317	
					bran	0.80	0.12	0.92	0.97	
					middlings	0.25	0.040	0.29	0.30	
					shorts	0.91	0.15	1.05	1.2	
					germ	0.59	0.098	0.68	0.72	
					flour	0.28	0.048	0.33	0.34	
					gluten	1.04	0.19	1.2	1.3	
					starch	0.0058	0.0008	0.007	0.0074	

[1] aspirated grain fractions.
[2] no detectable residue, expected to be approximately 20% of the spinosyn A concentration.

In three wheat milling and baking trials in the USA spinosad was sprayed onto the moving grain stream before it was carried by augur into storage at a target rate of 1 g ai/t (GH-C 5718, McCormick and Dolder, 2004). The wheat was stored for 6 months before it was taken for milling and baking. Spinosad residues were measured on the wheat, milled fractions and baked bread (Table 8) using analytical method GRM 00.04.

The wheat was milled in a Buhler mill to produce bran, shorts and flour. The flour was baked into bread (flour 150 g, yeast 1.5 g, non-fat dry milk 4.5 g, shortening 4.5 g, ammonium chloride 0.075 g, potassium bromate 0.43 g, water 93 g, sugar 9 g, salt 3 g) at 220ºC for 15-20 minutes.

Table 8. Spinosad residues in hard red winter wheat stored for 6 months, milled products and bread from milling and baking trials in the USA (GH-C 5718, McCormick and Dolder, 2004).

Location, year	Application		Wt treated	Commodity	Residue, mg/kg			Processing factor	Ref
	Form	g ai/t			Spinosyn A	Spinosyn D	Total		
KS 2000	SC 240	1.0	183 t	grain	0.65	0.14	0.79		GH-C 5718
			(6500 bu)	bran	1.3	0.27	1.6	2.0	trial SH
				shorts	0.67	0.13	0.80	1.0	
				flour	0.18	0.042	0.22	0.28	
				baked bread	0.12	0.026	0.14	0.18	
KS 2000	SC 240	1.0	225 t	grain	0.60	0.13	0.73		GH-C 5718
			(8000 bu)	bran	1.2	0.24	1.5	2.0	trial BL
				shorts	0.57	0.11	0.68	0.93	
				flour	0.11	0.025	0.13	0.18	
				baked bread	0.066	0.016	0.082	0.11	
KS 2000	SC 240	1.0	135 t	grain	0.43	0.090	0.52		GH-C 5718
			(4800 bu)	bran	1.4	0.29	1.6	3.2	trial RC
				shorts	0.70	0.13	0.83	1.6	
				flour	0.095	0.022	0.12	0.23	
				baked bread	0.054	0.013	0.067	0.13	

Table 9. Summary of processing factors for spinosad residues in grapes and cereals.

Crop	Residue, mg/kg	Processed commodity	Residues, mg/kg	Processing factors	
					Mean
Grapes	0.02	wine	<0.01 <0.01		
Grapes	0.03	wine	<0.01 <0.01 <0.01 <0.01		<0.33
Grapes	0.17	juice	0.57	3.3	
Grapes	0.05	juice	0.069	1.4	2.4

Crop	Residue, mg/kg	Processed commodity	Residues, mg/kg	Processing factors	
					Mean
Grapes	0.17	raisins	0.27	1.6	1.6
Maize		grits	see Table 7	0.067, 0.096	0.082
Maize		flour	see Table 7	0.23, 0.14	0.19
Maize		oil, dry mill	see Table 7	0.27, 0.29	0.28
Maize		oil, wet mill	see Table 7	0.91, 1.2	1.1
Rice		hulls	see Table 7	2.8, 2.8	2.8
Rice		bran	see Table 7	0.95, 0.62	0.79
Rice		brown rice	see Table 7	0.15, 0.072	0.11
Rice		white rice	see Table 7	0.035, 0.0094	0.022
Wheat		bran	see Table 7 and Table 8	0.97, 2.0, 2.0, 3.2	2.0
Wheat		shorts	see Table 7 and Table 8	1.2, 1.0, 0.93, 1.6	1.2
Wheat		flour	see Table 7 and Table 8	0.34, 0.28, 0.18, 0.23	0.26
Wheat		baked bread	see Table 8	0.18, 0.11, 0.13	0.14

RESIDUES IN ANIMAL COMMODITIES

Direct animal treatments

Information on residue levels occurring in the tissues of sheep treated with spinosad in a plunge dip, by application of a pour-on formulation and by application of an aerosol to fly-strike wounds was reported.

The Australian guideline for ectoparasiticide residues in sheep tissues (APVMA. Residue guideline no. 27) recognises the various possible use patterns on off-shears, short- or long-wool sheep, application as plunge dip, spray dip, jetting solution, or pour-on, and differences between breeds. It allows residue trials with only plunge or shower dipping if both are proposed and plunge dipping is preferred. It also requires treatment at the highest rate on the label. The plunge dip trial (ES012, Rothwell *et al*., 2000) used the higher concentration (20 mg ai/hl) specified for the shower dip and so is in accordance with the guideline.

In a supervised trial in Australia in 2000 25 Merino hoggets[1] weighing between 22 and 39.5 kg were plunge dipped in 20 mg ai/l spinosad prepared from a 25 g/l SC (ES012, Rothwell *et al.*, 2000). Animals were ear-tagged for identification and run in a single paddock with control animals at Armidale, NSW, grazing on native and improved pasture with access to clean drinking water. On days 5, 15, 35, 49 and 63 after treatment five treated and one control animal were slaughtered (Table 10).

Samples were analysed within 5 months of the first date of tissue collection by HPLC-MS after conventional extraction and clean-up (GRM 95-03, West *et al.*, 1995), previously evaluated by the 2001 JMPR.

Two plunge dip trials on sheep in Australia were evaluated by the 2001 JMPR (Evaluations, Table 73, pp.813-814). Dip concentrations were 10 mg ai/l. The highest concentrations of residues of spinosad were <0.01 mg/kg in the liver, 0.014 mg/kg in the kidneys, <0.01 mg/kg in muscle, 0.033 mg/kg in back fat and 0.042 mg/kg in perirenal fat.

[1] Hogget: a young sheep of either sex, aged from 10 months to cutting two adult teeth.

Table 10. Spinosad residues in the tissues of sheep after being plunged in a dip containing 20 mg ai/l spinosad (ES012, Rothwell *et al*., 2000) in a trial in Australia.

Sample	Days after treatment	Residues, mg/kg, Spinosyn A + spinosyn D [1]	Mean, mg/kg
Liver	5	<0.002, <0.01, <0.01, 0.012, 0.014	0.01
Liver	15	<0.002, <0.002, <0.002, <0.002, <0.002	<0.002
Kidney	5	0.011, <0.01, 0.01, 0.011, 0.011	0.01
Kidney	15	<0.002, <0.01, <0.002, <0.01, <0.002	<0.01
Muscle	5	<0.01, <0.01, <0.01, <0.01, 0.011	0.01
Muscle	15	<0.002, <0.002, <0.002, <0.002, <0.002	<0.002
Back fat	5	0.021, 0.012, 0.020, 0.014, 0.032	0.020
Back fat	15	0.022, 0.013, 0.019, 0.032, 0.026	0.022
Back fat	35	0.010, 0.027, 0.012, <0.01	0.015
Back fat	49	<0.01, 0.016, <0.01, <0.01, <0.01	0.011
Back fat	63	<0.01, <0.01, <0.01, <0.002, <0.01	<0.01
Perirenal fat	5	0.068, <0.01, 0.091, 0.094, 0.083	0.069
Perirenal fat	15	0.030, 0.033, 0.028, 0.049, 0.042	0.036
Perirenal fat	35	0.011, 0.017, 0.017, 0.012, <0.01	0.013
Perirenal fat	49	0.015, <0.01, <0.002, <0.002, <0.01	0.01
Perirenal fat	63	<0.01, <0.01, <0.002, <0.002, <0.01	<0.01

[1] Limit of detection 0.002 mg/kg. Limit of quantification 0.01 mg/kg.

In a trial in Australia in 2002 six groups of 3 adult Merino wethers[2] (18-19 micron wool, 2-5 years old, weighing 33.5-57 kg) and 3 second-cross wether lambs (27-28 micron wool, 4-5 months old, weighing 29.5-46 kg) were treated with a 20 g/l spinosad pour-on formulation, 12.5 mg ai/kg live body weight, immediately after shearing (ELANCO/GLP/0202, Ridley, 2003). The animals were ear-tagged for identification, run together on a commercial farm at Orange, NSW, grazing on native and improved pastures and fed sheep pellets and hay because of the dry conditions. Control animals were located separately to avoid cross-contamination. 2, 7, 14, 28, 56 and 112 days after treatment six animals were slaughtered (Table 11).

Samples were analysed within 6 months of the pour-on application for spinosyns A and D by the HPLC-UV procedure (GRM 95-03, West *et al.*, 1995) previously evaluated by the 2001 JMPR.

Table 11. Spinosad residues in the tissues of sheep after an off-shears pour-on spinosad treatment at 12.5 mg ai/kg live body weight (ELANCO/GLP/0202, Ridley, 2003) in a trial in Australia.

Sample	Days after treatment	Spinosad residues, mg/kg. Spinosyn A + spinosyn D [1]		Mean, mg/kg
		3 Merino sheep	3 cross-bred lambs	
Muscle	2	0.07, 0.04, 0.03	0.03, 0.04, 0.09	0.05
Muscle	7	0.02, 0.04, 0.03	0.03, 0.03, 0.04	0.06
Muscle	14	0.01, 0.01, 0.01	0.03, <0.01, 0.02	0.01
Muscle	28	<0.005, <0.01, <0.005	<0.01, 0.01, <0.005	0.01
Muscle	56	<0.005, <0.01, <0.005	<0.005, <0.005, <0.005	<0.01
Muscle	112	<0.005, <0.005, <0.005	<0.005, <0.005, <0.005	<0.005
Liver	2	0.16, 0.20, 0.11	0.16, 0.10, 0.30	0.17
Liver	7	0.06, 0.08, 0.07	0.12, 0.12, 0.08	0.09
Liver	14	0.03, 0.04, 0.02	0.06, 0.03, 0.05	0.04
Liver	28	<0.01, <0.01, <0.005	0.01, <0.01, <0.005	0.01
Liver	56	<0.005, <0.01, <0.005	<0.005, <0.005, <0.005	<0.01
Liver	112	<0.005, <0.005, <0.005	<0.005, <0.005, <0.005	<0.005

[2] Wether: a male sheep castrated young.

Sample	Days after treatment	Spinosad residues, mg/kg. Spinosyn A + spinosyn D [1]		Mean, mg/kg
		3 Merino sheep	3 cross-bred lambs	
Kidney	2	0.25, 0.10, 0.16	0.10, 0.09, 0.25	0.16
Kidney	7	0.08, 0.13, 0.07	0.08, 0.13, 0.10	0.10
Kidney	14	0.02, 0.04, 0.04	0.05, 0.03, 0.04	0.04
Kidney	28	<0.01, <0.01, <0.005	<0.01, 0.01, <0.01	0.01
Kidney	56	<0.005, <0.005, <0.005	<0.005, <0.005, <0.005	<0.005
Kidney	112	<0.005, <0.005, <0.005	<0.005, <0.005, <0.005	<0.005
Perirenal fat	2	1.33, 0.87, 0.48	0.87, 0.43, 2.01	1.0
Perirenal fat	7	0.62, 0.92, 0.51	0.80, 1.07, 0.82	0.79
Perirenal fat	14	0.17, 0.28, 0.19	0.66, 0.20, 0.28	0.30
Perirenal fat	28	0.12, 0.06, 0.02	0.18, 0.11, 0.03	0.09
Perirenal fat	56	0.02, 0.04, <0.01	<0.01, 0.02, <0.005	0.02
Perirenal fat	112	<0.005, <0.005, <0.005	<0.01, <0.005, <0.005	<0.01
Back fat	2	0.47, 0.20, 0.29	0.28, 0.19, 0.71	0.36
Back fat	7	0.45, 0.57, 0.62	0.51, 0.73, 0.79	0.61
Back fat	14	0.13, 0.14, 0.19	0.55, 0.33, 0.43	0.30
Back fat	28	0.13, 0.04, 0.01	0.27, 0.18, 0.03	0.11
Back fat	56	0.01, 0.05, <0.005	<0.005, 0.01, <0.005	0.01
Back fat	112	<0.005, <0.005, <0.005	<0.01, <0.005, <0.005	<0.01

[1] Limit of detection 0.005 mg/kg. Limit of quantification 0.01 mg/kg.

In another trial in Australia in 2003 six groups of sheep, each consisting of 3 adult Merino wethers (19.5 micron wool, weighing 38.5-537 kg) and 3 second-cross Dorset wether lambs (weighing 28.5-34.5 kg), were treated with a 20 g/l spinosad pour-on formulation the day after shearing (ELS1303, Hacket *et al.,* 2003) at a rate of 12.5 mg ai/kg live body weight. Animals ear-tagged for identification were run together on a research farm at Armidale, NSW, grazing on native and improved pastures with access to water in PVC troughs. Control animals were located in a separate paddock to avoid cross-contamination. At 2, 7, 14, 28, 56, 84 and 112 days after treatment one of the groups of six animals was slaughtered (Table 12).

Samples were analysed within 5 months of the pour-on application for spinosyns A and D by HPLC-MS procedure STM CR 428, based on mass-spectrometric detection after the extraction and clean-up of the HPLC-UV procedure GRM 95-03 (West *et al.*, 1995) previously evaluated by the 2001 JMPR.

Table 12. Spinosad residues in the tissues of sheep after an off-shears pour-on spinosad treatment at 12.5 mg ai/kg live body weight (ELS1303, Hacket *et al*., 2003) in a trial in Australia in 2003.

Sample	Days after treatment	Spinosad residues, mg/kg. Spinosyn A + spinosyn D [1]		Mean, mg/kg
		3 Merino sheep	3 cross-bred Dorset lambs	
Muscle	2	0.036, 0.056, 0.023	0.12, 0.073, 0.089	0.066
Muscle	7	0.013, 0.019, 0.012	0.013, 0.016, <0.01	0.014
Muscle	14	<0.01, <0.005, <0.005	0.024, <0.005, 0.025	0.01
Muscle	28	<0.005, <0.005, <0.005	<0.005, <0.005, <0.005	<0.005
Muscle	56	<0.005, <0.005, <0.01	<0.005, <0.005, <0.005	<0.01
Muscle	84	<0.005, <0.005, <0.005	<0.005, <0.005, <0.005	<0.005
Muscle	112	<0.005, <0.005, <0.005	<0.005, <0.005, <0.005	<0.005
Liver	2	0.086, 0.19, 0.096	0.31, 0.14, 0.31	0.19
Liver	7	0.033, 0.032, 0.027	0.013, 0.032, 0.022	0.027
Liver	14	0.017, 0.012, 0.013	0.019, 0.015, 0.013	0.015
Liver	28	<0.005, <0.01, <0.01	<0.01, <0.005, <0.005	<0.01
Liver	56	<0.005, <0.005, <0.005	<0.005, <0.005, <0.005	<0.005
Liver	84	<0.005, <0.005, <0.005	<0.005, <0.005, <0.005	<0.005
Liver	112	<0.005, <0.005, <0.005	<0.005, <0.005, <0.005	<0.005
Kidney	2	0.079, 0.19, 0.090	0.39, 0.32, 0.25	0.22

Sample	Days after treatment	Spinosad residues, mg/kg. Spinosyn A + spinosyn D [1]		Mean, mg/kg
		3 Merino sheep	3 cross-bred Dorset lambs	
Kidney	7	0.041, 0.033, 0.014	0.017, 0.036, 0.044	0.031
Kidney	14	0.025, 0.015, 0.019	0.028, 0.019, 0.014	0.020
Kidney	28	<0.005, <0.01, <0.01	<0.01, <0.005, <0.005	<0.01
Kidney	56	<0.005, <0.005, <0.005	<0.005, <0.005, <0.005	<0.005
Kidney	84	<0.005, <0.005, <0.005	<0.005, <0.005, <0.005	<0.005
Kidney	112	<0.005, <0.005, <0.005	<0.005, <0.005, <0.005	<0.005
Perirenal fat	2	0.37, 0.70, 0.33	1.8, 0.85, 1.1	0.86
Perirenal fat	7	0.36, 0.44, 0.19	0.19, 0.64, 1.3	0.52
Perirenal fat	14	0.085, 0.35, 0.35	0.60, 0.52, 0.37	0.38
Perirenal fat	28	0.052, 0.094, 0.052	0.025, 0.029, 0.11	0.061
Perirenal fat	56	0.020, <0.005, 0.011	0.013, <0.005, 0.023	0.013
Perirenal fat	84	<0.005, <0.01, <0.005	<0.005, <0.01, <0.01	<0.01
Perirenal fat	112	<0.005, <0.005, <0.005	<0.005, <0.005, <0.005	<0.005
Back fat	2	0.075, 0.060, 0.032	0.11, 0.078, 0.060	0.069
Back fat	7	0.080, 0.13, 0.064	<0.01, 0.035, 0.20	0.087
Back fat	14	0.087, 0.11, 0.16	0.12, 0.10, 0.10	0.11
Back fat	28	0.020, 0.053, 0.026	0.14, <0.005, 0.019	0.044
Back fat	56	0.011, <0.005, <0.01	<0.005, <0.005, <0.005	0.010
Back fat	84	<0.01, <0.005, <0.005	<0.01, <0.01, <0.005	<0.01
Back fat	112	<0.005, <0.005, <0.005	<0.005, <0.005, <0.005	<0.005

[1] Limit of detection 0.005 mg/kg. Limit of quantification 0.01 mg/kg.

In an aerosol formulation trial in Australia in 2002 flystruck sheep were treated with spinosad according to the proposed label instructions and slaughtered 2 and 7 days after treatment (ELS1164, Chick, 2002). The sheep in the trial should have been 16 adult Merinos weighing between 25 and 50 kg with a flystrike lesion of a minimum of 200 cm^2. In fact the 2 control and 14 treated sheep, including some crossbred, obtained from commercial properties, weighed between 22.5 and 45.5 kg, were an equal mixture of males and females and had existing flystrike lesions between 108 and 1600 cm^2. The aerosol formulation contained 4 mg/g spinosad and 0.8 mg/g chlorhexidine digluconate (registered product 2.8 mg/g spinosad and 0.39 mg/g chlorhexidine digluconate) and delivered 1.54 g formulation per second. Animals were clipped around the flystruck area, the area was measured and the dose calculated at a rate of 1 second of aerosol spray per 40 cm^2 of affected area. The aerosol can was weighed before and after treatment. The sheep identified by eartags grazed native and improved pastures in the holding paddocks near Armidale in NSW. The results are shown in Table 13.

Table 13. Spinosad residues in the tissues of sheep treated for flystrike lesions with an aerosol formulation and slaughtered 2 or 7 days later in a trial in Australia in 2002 (ELS1164, Chick, 2002).

Animal bw, kg[2]	Spinosad, mg	Days post-treatment	Spinosad residues (spinosyn A + spinosyn D), mg/kg [1]				
			Muscle	Perirenal fat	Liver	Kidney	Back fat
x 36	20.7	2	<0.005	<0.005	<0.005	<0.005	<0.005
x 28.5	26.6	2	<0.005	<0.005	<0.005	<0.005	<0.01
x 29	113	2	<0.005	<0.005	0.01	<0.01	0.03
x 33.5	18.9	2	<0.005	<0.005	<0.005	<0.005	<0.005
m 34.5	97.4	2	<0.005	0.02	0.01	<0.01	0.01
m 25	61.3	2	0.02	0.04	0.02	0.02	0.04
m 37	210	2	0.03	0.12	0.04	0.03	0.11
m 41.5	84.9	2	0.02	0.07	0.02	0.02	0.04
m 45.5	148	2	0.02	0.06	0.02	0.02	0.05
m 24	71.1	7	<0.01	0.10	<0.01	<0.005	0.02
m 31	16.8	7	0.01	0.20	0.02	<0.005	0.14
m 27.5	159	7	0.01	0.03	0.01	<0.01	<0.01

Animal bw, kg[2]	Spinosad, mg	Days post-treatment	Spinosad residues (spinosyn A + spinosyn D), mg/kg [1]				
			Muscle	Perirenal fat	Liver	Kidney	Back fat
m 22	98.6	7	0.01	<0.01	<0.01	<0.005	0.03
m 25	43.1	7	<0.01	<0.01	<0.005	<0.005	<0.01

[1] Limit of detection 0.005 mg/kg. Limit of quantification 0.01 mg/kg.
[2] x: crossbred sheep. m: Merino sheep.

NATIONAL MAXIMUM RESIDUE LIMITS

The following MRLs and tolerances for the commodities evaluated were reported to the Meeting.

Country	Commodity	MRL or tolerance, mg/kg
Australia	Edible offal (mammalian) Grapes Meat (mammalian) [in the fat]	T 0.2 0.5 T 1
Israel	Grapes	0.2
Italy	Grapes	0.2
USA	Grapes Stored grain	0.5 1

T: temporary MRL

APPRAISAL

Spinosad was first evaluated by the 2001 JMPR, which established an ADI of 0–0.02 mg/kg bw. An ARfD was judged to be unnecessary. MRLs were recommended for fruits, vegetables, nuts, oil seeds, cereal grains, animal feeds and animal commodities. Questions about the MRL for milk were raised by the CCPR at its Thirty-fifth and Thirty-sixth Sessions, and the JMPR was requested to consider further how MRLs for milk and milk fat should be expressed.

The Meeting received information on registered uses and data from supervised residue trials on grapes and stored grain. Information on direct uses of spinosad on sheep for control of blowfly and lice and supporting residue data were also received.

Methods of analysis

An immunoassay method previously evaluated by the 2001 JMPR was used in the supervised trials on grapes.

The analytical method used for analysis of spinosad residues in cereal grains and processed products was based on previously evaluated methods. Samples were extracted with acetonitrile and water and the extracts cleaned up on a strong cation-exchange column. Spinosyns A, D, K, B and *N*-demethyl D were eluted with dilute ammonium acetate in acetonitrile and methanol, ready for analysis by HPLC with mass selective detection. The LOQ was 0.01 mg/kg.

The analytical methods used for fat, muscle, liver, kidney and bovine milk were similar in principle to the above method, but with variations in clean-up depending on the substrate. The LOQ for milk was 0.005 mg/kg, and that for the other substrates was 0.01 mg/kg.

Results of supervised trials on crops

Grape

The Meeting received the results of supervised trials for use of spinosad on grapes in the USA. The samples were analysed by immunoassay method GRM 96.11, which was evaluated by the JMPR previously.

In the USA, spinosad may be applied to grapes at 0.14 kg ai/ha with a maximum seasonal application of 0.49 kg ai/ha and harvesting 7 days after the final application. In 12 trials in the USA that conformed substantially to the registered use, the residue levels were: < 0.01, 0.02, 0.03, 0.05, 0.077, 0.082, 0.086, 0.13, 0.17, 0.22, 0.23 and 0.39 mg/kg.

The residue levels of spinosad in grapes in supervised trials in France, Italy and Spain are recorded in Table 39 (p. 761) of the JMPR Residue Evaluations for 2001. Spinosad may be used on grapes in Cyprus at 0.072 kg ai/ha with a PHI of 7 days. The conditions used in trials in France, Italy and Spain, where the application rate was 0.060 kg with a PHI of 5 days, were considered sufficiently similar to those of Cyprus GAP. The residue levels, determined by an HPLC method, in grapes in two trials in France (0.01 and 0.03 mg/kg), one trial in Italy (0.09 mg/kg) and one trial in Spain (0.19 mg/kg) were, in ranked order: 0.01, 0.03, 0.09 and 0.19 mg/kg. The levels in the same samples determined by the immunoassay method were: 0.02, 0.04, 0.15 and 0.24 mg/kg.

The Meeting combined the data from Europe and the USA obtained by the immunoassay method. The residue levels in the 16 trials, in ranked order, median underlined, were: < 0.01, 0.02 (two), 0.03, 0.04, 0.05, 0.077, <u>0.082</u>, <u>0.086</u>, 0.13, 0.15, 0.17, 0.22, 0.23, 0.24 and 0.39 mg/kg.

The Meeting estimated a maximum residue level of 0.5 mg/kg and an STMR value for spinosad in grapes of 0.084 mg/kg.

Fate of residues during storage

In a series of trials with cereal grain (barley, maize, oats, rice and wheat) in the USA, spinosad was applied to grain at a target rate of 1 g ai/t. Most of the trials were small-scale, only 18–23 kg grain being treated; in two larger trials, 9.9 t of maize and 30.9 t of wheat were treated. The duration of storage was 3–11 months at ambient temperatures. Samples were analysed by HPLC with mass spectromtery detection.

The residue levels in grain immediately after treatment represented 43–91% of the target application rate, reflecting the efficiency of application in the experiments. In the two larger trials, the initial residue levels were 77% and 87% of the target rates. The residue levels declined very slowly, if at all. The highest residue level in each trial was taken, whether at day 0 or after 11 months' storage. The trial in which a dose rate of 1.6 g ai/t was used was excluded as being outside GAP. The residue levels in the 20 trials were: 0.43, 0.45, 0.47, 0.58, 0.59, 0.63, 0.67, 0.69 (three), 0.70, 0.75, 0.79, 0.81, 0.86, 0.90, 0.91 (two), 0.93 and 0.95 mg/kg.

In three further trials in the USA in which wheat in storage batches of 135–225 t was treated at 1 g ai/t for storage and processing, the spinosad residue levels after 6 months' storage were 0.52, 0.73 and 0.79 mg/kg.

The residue levels in the 23 trials, in ranked order, were: 0.43, 0.45, 0.47, 0.52, 0.58, 0.59, 0.63, 0.67, 0.69 (three), <u>0.70</u>, 0.73, 0.75, 0.79 (two), 0.81, 0.86, 0.90, 0.91 (two), 0.93 and 0.95 mg/kg.

The Meeting estimated a maximum residue level of 1 mg/kg and an STMR for spinosad on cereal grains of 0.70 mg/kg on the basis of post-harvest use. The Meeting withdrew its previous recommendations for maize (0.01* mg/kg) and sorghum (1 mg/kg), to be replaced by the recommendation for cereal grains.

Fate of residues during processing

Three trials in France and one in Italy on processing of grapes to pomace and wine are summarized on pp. 823–824 of the JMPR Residue Evaluations of 2001.

In these trials, the levels of spinosad residues were below the LOQ (0.01 mg/kg) in all wine samples. As the residue levels in grapes were low (< 0.01–0.03 mg/kg), the best estimate of the processing factor for wine is < 0.33. Processing factors of 3.3 and 1.4 for juice and 1.6 for raisins produced from grapes were calculated in two trials in the USA. Juice was produced on a very small scale, with manual crushing, pressing and straining of about 1 kg of grapes, and this was not considered representative of a commercial process.

Processing studies on cereals were provided from the USA, comprising milling of maize (two trials), rice (two trials) and wheat (one trial) and three trials of wheat milling and baking. Spinosad residues were found essentially on the outside of the grain and were strongly concentrated in the aspirated grain fraction from the milling of maize and wheat. The residue levels in grits and flour were much lower than those in the grain. Most of the residues on rice remained with the husk and bran, with little occurring on white rice.

The following processing factors were calculated from the results of the trials. The factors are mean values, excluding those calculated from undetectable results, except for wine in which no residues were detected.

Commodity	Product	Processing factor	No. of trials
Grapes	Wine	< 0.33	4
	Raisins	1.6	1
Maize	Grits	0.082	2
	Flour	0.19	2
	Oil, dry milling	0.28	2
	Oil, wet milling	1.1	2
Rice	Hulls	2.8	2
	Bran	0.79	2
	Brown rice	0.11	2
	White rice	0.022	2
Wheat	Bran	2.0	4
	Shorts	1.2	4
	Flour	0.26	4
	Baked bread	0.14	3

The Meeting used the processing factors for wine, raisins and cereals to estimate STMR-Ps for processed commodities.

The processing factor for wine (< 0.33) was applied to the STMR for grape (0.084 mg/kg) to calculate an STMR-P of 0.028 mg/kg for wine.

The processing factor for raisins (1.6) was applied to the highest residue level in grapes (0.39 mg/kg) and the STMR for grape (0.084 mg/kg) to calculate a highest residue level of 0.62 mg/kg and an STMR-P of 0.13 mg/kg for raisins.

The Meeting estimated a maximum residue level of 1 mg/kg and an STMR-P value of 0.13 mg/kg for spinosad on dried grapes (currants, raisins and sultanas).

The processing factors for processed cereal fractions were applied to the STMR for cereal grains (0.70 mg/kg) to calculate the following STMR-P values: grits, 0.057 mg/kg; maize flour, 0.13 mg/kg; maize oil, 0.77 mg/kg; rice hulls, 2.0 mg/kg; rice bran, 0.55 mg/kg; brown rice, 0.077 mg/kg; white rice, 0.015 mg/kg; wheat bran, 1.4 mg/kg; wheat flour, 0.18 mg/kg; and white bread, 0.098 mg/kg.

The processing factor for wheat bran (2.0) was applied to the highest residue level in cereals grain (0.95 mg/kg) to calculate a highest residue level of 1.9 mg/kg for wheat bran.

The Meeting estimated a maximum residue level for spinosad on wheat bran of 2 mg/kg.

Residues in animal commodities

Direct treatment of animals

The Meeting received information on residue levels occurring in the tissues of sheep treated with spinosad in a plunge dip, by application of a pour-on formulation and by application of an aerosol to fly-strike wounds.

Sheep (25) were treated in a plunge dip containing 20 mg ai/l spinosad prepared from a 25 g/l suspension concentrate in a supervised trial in line with Australian guidelines and registered uses in Australia in 2000. Groups of animals were slaughtered for tissue collection on days 5, 15, 35, 49 and 63 after treatment. Samples were analysed by HPLC-MS after a conventional extraction and clean-up procedure evaluated by the 2001 JMPR. The highest levels of residues of spinosad in tissues 5 or 15 days after treatment were: 0.014 mg/kg in liver, 0.011 mg/kg in kidney, 0.011 mg/kg in muscle, 0.032 mg/kg in back fat and 0.094 mg/kg in perirenal fat.

Two plunge dip trials on sheep in Australia were reported by the 2001 JMPR (Residue Evaluations, Table 73, pp. 813–814). The dip concentration was 10 mg ai/l. The highest tissue concentrations of spinosad residues were: < 0.01 mg/kg in liver, 0.014 mg/kg in kidney, < 0.01 mg/kg in muscle, 0.033 mg/kg in back fat and 0.042 mg/kg in perirenal fat.

The data on residues from pour-on trials on sheep could not be evaluated because spinosad pour-on uses on sheep are not registered.

A spinosad aerosol spray is registered in Australia for treating fly-strike wounds on sheep. A typical wound of 200 cm^2 should take 6 s to treat. In a trial of the aerosol formulation in Australia in 2002, 14 sheep with fly-strike lesions measuring 108–1600 cm^2 were treated with spinosad according to the proposed label instructions and were slaughtered 2 and 7 days after treatment for tissue collection. The aerosol product contained 4 mg/g spinosad and 0.8 mg/g chlorhexidine digluconate (The registered product has 2.8 mg/g spinosad and 0.39 mg/g chlorhexidine digluconate.) and was delivered at a rate of 1.54 g of formulation per second. The animals were clipped around the fly-strike area, the area was measured and the dose was calculated at a rate of 1 s of aerosol spray per 40 cm^2 of affected area. The highest residue levels were: 0.04 mg/kg in liver, 0.03 mg/kg in kidney, 0.03 mg/kg in muscle, 0.14 mg/kg in back fat and 0.20 mg/kg in perirenal fat.

The Meeting noted that the aerosol treatment resulted in higher residue levels in tissues than the plunge dip or the previously evaluated jetting treatment.

The Meeting estimated maximum residue levels for spinosad of 0.3 (fat) mg/kg in sheep meat and 0.1 mg/kg in edible offal of sheep.

Maximum residue levels

Spinosad residues can occur in meat and milk after direct use on animals or from residues in animal feeds.

The 2001 JMPR evaluated a feeding study with dairy cows, compiled a dietary burden for farm animals and estimated maximum residue levels of 2 mg/kg in cattle meat (fat), 0.5 mg/kg in cattle kidneyand 0.5 mg/kg in cattle liver It estimated STMRs of 0.32 mg/kg in cattle fat, 0.010 mg/kg in cattle meat, 0.032 mg/kg in cattle kidney and 0.064 mg/kg in cattle liver. These estimates for cattle commodities were superseded by estimates derived from direct treatment of cattle, which resulted in higher residue levels. The MRL recommendations associated with direct treatment of cattle were: 3 mg/kg (fat) in cattle meat, 1 mg/kg in cattle kidney and 2 mg/kg in cattle liver.

The 2002 JMPR[3] introduced a policy of recommending maximum residue levels for mammalian meat and offal rather than MRLs for cattle meat and offal when residues occurred in feed and a suitable study of cattle feeding was available. In the light of this policy, the Meeting recommended that the 2001 recommendations be reviewed.

The current Meeting proposed maximum residue levels for mammalian meat and offal based on the results of the feeding study in dairy cows and the corresponding dietary burden. None of the recommendations for MRLs by the current Meeting change the previously estimated dietary burden of spinosad residue in cattle. The MRLs for cattle meat, liver and kidney were retained because they are related to direct treatment, which produces higher residue levels than occur from feed. Therefore, the MRLs for mammalian meat and offal should have the qualification 'except cattle'.

The Meeting estimated a maximum residue level of 2 (fat) mg/kg for 'Meat (from mammals other than marine mammals) [except cattle]' and associated STMRs of 0.01 mg/kg for meat and 0.32 mg/kg for fat.

The Meeting estimated a maximum residue level of 0.5 mg/kg for 'Edible offal (mammalian) [except cattle]' and associated STMRs of 0.064 mg/kg for liver and 0.032 mg/kg for kidney.

The Meeting withdrew the current recommendations for sheep meat (0.01* (fat) mg/kg) and edible offal of sheep (0.01* mg/kg), which are superseded by the recommendations for mammalian meat and offal. The Meeting also noted that residue levels resulting from direct treatment of sheep by jetting, plunge dipping and aerosol treatment of wounds did not exceed the maximum residue levels resulting from feed residues. There is no separate MRL recommendation for sheep related to these direct uses.

The CCPR expressed concern about the MRL for spinosad in milk, the levels of spinosad in milk fat and how MRLs might best be expressed for partially fat-soluble compounds in milk. (See also general report item 2.7 on fat-soluble pesticide residues in milk.)

The 2001 JMPR reported that, after direct treatment of dairy cows with spinosad, residues were measured in 119 samples of milk and cream and that the mean quotient of the concentration in cream divided by the concentration in milk was 4.2. A plot of the same residue levels in whole milk against those in cream showed that the residue level in milk was approximately 24% of that in cream (line of best fit through the origin). (See figure in section 2.7.)

The levels of spinosad residues in milk and cream from a feeding study in dairy cows are summarized in Table 79 of the JMPR Residue Evaluations of 2001. The mean quotient of the concentration in cream divided by the concentration in milk from cows at feeding levels of 1, 3 and 10 ppm was 4.0, in good agreement with the results for direct treatment.

The MRL for milk (1 mg/kg) was estimated on the basis of the highest residue level in milk, 0.65 mg/kg, after direct treatment. The calculated concentration in cream would then be $0.65 \times 4.2 = 2.7$ mg/kg. On the assumption that cream is approximately 50% fat, the concentration in fat would be about 5 mg/kg.

The Meeting estimated a maximum residue level for spinosad residues in cattle milk fat of 5 mg/kg.

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 IEDIs.

Definition of the residue (for compliance with the MRL and for estimation of dietary intake: sum of spinosyn A and spinosyn D.

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[3] JMPR Report. 2002. 2.11. Maximum residue levels for animal commodities—group MRLs.

CCN	Commodity	MRL, mg/kg		STMR or STMR-P, mg/kg
		New	Previous	
FM 0812	Cattle milk fat	5		
GC 0080	Cereal grains	1 (Po)		0.70
DF 0269	Dried grapes (= Currants, Raisins and Sultanas)	1		0.13
MO 0105	Edible offal (Mammalian) [except cattle] Note 3	0.5		liver 0.064 kidney 0.032
FB 0269	Grape	0.5		0.084
GC 0645	Maize	W [1]	0.01*	
MM 0095	Meat (from mammals other than marine mammals) [except cattle] [4]	2 (fat)		meat 0.01 fat 0.32
MM 0822	Sheep meat	W [1]	0.01* (fat)	
MO 0822	Sheep, Edible offal of	W [2]	0.01*	
GC 0651	Sorghum	W [1]	1	
CM 0654	Wheat bran, unprocessed	2		1.4
	Grits			0.057
CF 1255	Maize flour			0.13
OC 0645	Maize oil, crude			0.77
CM 1206	Rice bran, unprocessed			0.55
	Rice hulls			2.0
CM 0649	Rice, husked (=Brown rice)			0.077
CM 1205	Rice, polished (=White rice)			0.015
CF 1211	Wheat flour			0.18
CP 1211	White bread			0.098
	Wine			0.027

* The MRL is estimated at or about the LOQ.

[1] Replaced by recommendation for wider group of commodities.

[2] The MRL for 'Edible offal (Mammalian) [except cattle]' is derived from a dairy cow feeding study and the corresponding animal dietary burden. The recommendation is extended to 'Edible offal (Mammalian)' following the policy of the 2002 JMPR[3]. MRL proposals for cattle kidney (1 mg/kg) and cattle liver (2 mg/kg), arising from direct use of spinosad on cattle, should remain. They exceed the recommended 0.5 mg/kg for mammalian edible offal and so require the words 'except cattle' to be added to the commodity description.

[4] The MRL for 'Meat (from mammals other than marine mammals) [except cattle]' is derived from a dairy cow feeding study and the corresponding animal dietary burden. The recommendation is extended to 'Meat (from mammals other than marine mammals)' following the policy of the 2002 JMPR[3]. The MRL proposal for cattle meat (3 mg/kg, fat), arising from direct use of spinosad on cattle, should remain. It exceeds the recommended 2 mg/kg (fat) for mammalian meat and so requires the words 'except cattle' to be added to the commodity description.

DIETARY RISK ASSESSMENT

Long-term 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 42 food commodities from this and previous evaluations and were used to calculate dietary intake. The results are shown in Annex 3.

The IEDIs in the five GEMS/Food regional diets, based on estimated STMRs were 9-30% 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 ARfD 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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