## SPINETORAM (233)

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### EXPLANATION

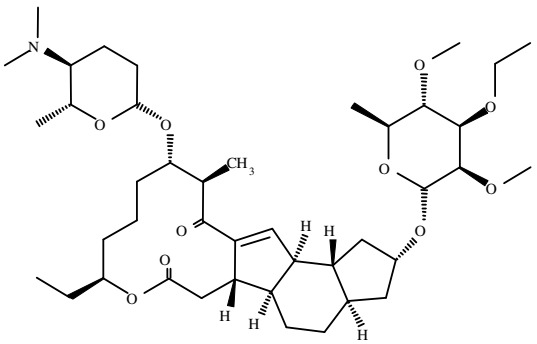
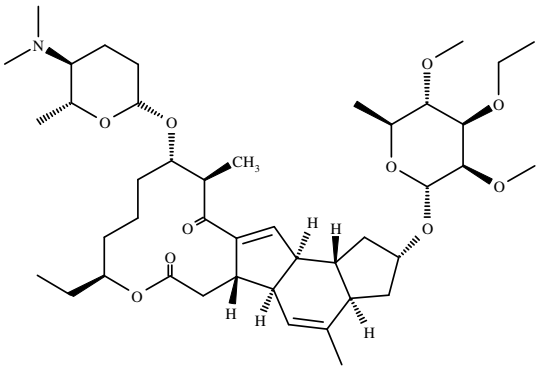
Spinetoram, a multi-component tetracyclic macrolide in the class of spinosyn insecticides, was developed for the control of lepidopterous larvae, leafminers, and thrips on a variety of crops. Its mode of action is disruption of nicotinic/gamma amino butyric acid-gated chloride channels.

It was identified as a priority new compound at the 39th Session of the CCPR in 2007 (ALINORM 07/30/24-Rev.1) for evaluation by the 2008 JMPR. The Meeting received information on physical and chemical properties, animal and plant metabolism, environmental fate, analytical methods, storage stability, use patterns, supervised trials, processing and farm animal feeding.

### IDENTITY

Spinetoram consists of two closely related active ingredients, as shown below, present approximately in a three to one ratio. The term spinetoram is used interchangeably for the combination of these two compounds. Spinetoram is a chemically-modified fermentation product of *Saccharopolyspora spinosa.*

| | | |
|---|---|---|
| ISO common name: | Spinetoram<br>(Mixture of two main components as shown below) | |
| Chemical name | | |
| IUPAC: | Major component<br>(XDE-175-J) | (2*R*,3a*R*,5a*R*,5b*S*,9*S*,13*S*,14*R*,16a*S*, 16b*R*)-2-(6-deoxy-3-*O*-ethyl-2,4-di-*O*-methyl-α-L-mannopyranosyloxy)-13-[(2*R*,5*S*,6*R*)-5-(dimethylamino)tetrahydro-6-methylpyran-2-yloxy]-9-ethyl-2,3,3a,4,5,5a,5b,6,9,10,11,12,13,14,16a,16b-hexadecahydro-14-methyl-1*H-as*-indaceno[3,2-*d*]oxacyclododecine-7,15-dione |
| | Minor component<br>(XDE-175-L) | (2*R*,3a*R*,5a*S*,5b*S*,9*S*,13*S*,14*R*,16a*S*, 16b*S*)-2-(6-deoxy-3-*O*-ethyl-2,4-di-*O*-methyl-α-L-mannopyranosyloxy)-13-[(2*R*,5*S*,6*R*)-5-(dimethylamino)tetrahydro-6-methylpyran-2-yloxy]-9-ethyl-2,3,3a,5a,5b,6,9,10,11,12,13,14,16a,16b-tetradecahydro-4,14-dimethyl-1*H-as*-indaceno[3,2-*d*]oxacyclododecine-7,15-dione |
| CAS: | Major component<br>(XDE-175-J) | (2*R*,3a*R*,5a*R*,5b*S*,9*S*,13*S*,14*R*,16a*S*, 16b*R*)-2-[(6-deoxy-3-*O*-ethyl-2,4-di-*O*-methyl-α-L-mannopyranosyl)oxy]-13-[[(2*R*,5*S*,6*R*)-5-(dimethylamino)tetrahydro-6-methyl-2*H*-pyran-2-yl]oxy]-9-ethyl-2,3,3a,4,5,5a,5b,6,9,10,11,12,13,14,16a,16b-hexadecahydro-14-methyl-1*H-as*-indaceno[3,2-*d*]oxacyclododecine-7,15-dione |
| | Minor component<br>(XDE-175-L) | (2*R*,3a*R*,5a*S*,5b*S*,9*S*,13*S*,14*R*,16a*S*, 16b*S*)-2-[(6-deoxy-3-*O*-ethyl-2,4-di-*O*-methyl-α-L-mannopyranosyl)oxy]-13-[(2*R*,5*S*,6*R*)-5-(dimethylamino)tetrahydro-6-methyl-2*H*-pyran-2-yl]oxy]-9-ethyl-2,3,3a,5a,5b,6,9,10,11,12,13,14,16a,16b-tetradecahydro-4,14-dimethyl-1*H-as*-indaceno[3,2-*d*]oxacyclododecine-7,15-dione |

| | | |
|---|---|---|
| CAS Registry No.: | XDE-175-J | 187166-40-1 |
| | XDE-175-L | 187166-15-0 |
| CIPAC No.: | 802 | |
| Synonyms for active substance: | X574175 (2003-2004) | |
| | XDE-175 (2004-2007) | |
| | DE-175 (2007-) | |
| Structural formula: | XDE-175-J |  |
| | XDE-175-L |  |
| Molecular formula: | XDE-175-J | $C_{42}H_{69}NO_{10}$ |
| | XDE-175-L | $C_{43}H_{69}NO_{10}$ |
| Molecular weight: | XDE-175-J | 748.02 |
| | XDE-175-L | 760.03 |

## PHYSICAL AND CHEMICAL PROPERTIES

### *Pure active ingredient*

| Property | Results | | Reference |
|---|---|---|---|
| | XDE-175-J | XDE-175-L | |
| Appearance: | White powder | White to yellow crystals | Jennings, 2007, FAPC 73363 & FAPC 73364 |
| Odour: | Odourless | Almond odour | Jennings, 2007, FAPC 73363 & FAPC 73364 |
| Melting point: | 143.4 °C | 70.8 °C | Madsen & Jennings, 2005 FAPC-052-002 & FAPC-052-003 |
| Relative density: | 1.1495 g/cm$^3$ at 19.5 °C | 1.1807 g/cm$^3$ at 20.1 °C | Tunink, 2006 NAFST-06-134 |

| Property | Results | | Reference |
|---|---|---|---|
| | XDE-175-J | XDE-175-L | |
| Solubility in water at 20 °C: | | | |
| Unbuffered: | 10.0 mg/L | 31.9 mg/L | Comb, 2005 NAFST-05-071 & NAFST-05-072 |
| pH 5: | 423 mg/L | 1.63 g/L | |
| pH 7: | 11.3 mg/L | 46.7 mg/L | |
| pH 9: | - | 1.98 mg/L | |
| pH 10: | 6.27 mg/L | 0.706 mg/L | |
| Vapour pressure: | $5.3 \times 10^{-5}$ Pa at 20 °C | $2.1 \times 10^{-5}$ Pa at 20 °C | Comb, 2005 NAFST-05-073 & NAFST-05-074 |
| Volatility, Henry's Law Constant at 20 °C (calculation): | | | |
| Unbuffered: | $4.0 \times 10^{-3}$ Pa·m$^3$/mol | $5.0 \times 10^{-4}$ Pa·m$^3$/mol | Huntley, 2005 NAFST-05-129 |
| pH 5: | $9.4 \times 10^{-5}$ Pa·m$^3$/mol | $9.8 \times 10^{-3}$ Pa·m$^3$/mol | |
| pH 7: | $3.5 \times 10^{-3}$ Pa·m$^3$/mol | $3.4 \times 10^{-4}$ Pa·m$^3$/mol | |
| pH 10: | $6.3 \times 10^{-3}$ Pa·m$^3$/mol | $2.3 \times 10^{-2}$ Pa·m$^3$/mol | |
| Octanol-water partition coefficient at 20 °C (logPow): | | | |
| pH 5: | 2.44 | 2.94 | Comb, 2005 NAFST-05-075 & NAFST-05-076 |
| pH 7: | 4.09 | 4.49 | |
| pH 9: | 4.22 | 4.82 | |
| Dissociation constant at 25 °C (pKa): | | | |
| | 7.86 | 7.59 | Madsen & Jennings, 2005 FOR-05-043 & FOR-05-044 |
| Hydrolysis at 20 °C: | | | |
| pH 5: | Stable against hydrolysis<br>Stable | Stable | Laughlin *et al*., 2005 040108 |
| pH 7: | Stable | Stable | |
| pH 9: | | DT50=154 days | |
| Photolysis: | Numerous (> 70) minor breakdown products<br>DT50: 0.5 days<br>Mass balance:<br>101.1 ± 3.7% | N-demethyl-175-L and numerous (>70) numerous minor breakdown products<br>DT50: 0.3 days<br>Mass balance: 94.2 ± 2.0% | Yoder, 2005 040079 |
| Quantum yield at 25 °C: | | | |
| | 0.042 | 0.066 | Yoder, 2005 040079 |
| Theoretical lifetime in aqueous systems at 25°C (calculation): | | | |
| | 0.5 days | 0.3 days | Yoder, 2005 040079 |

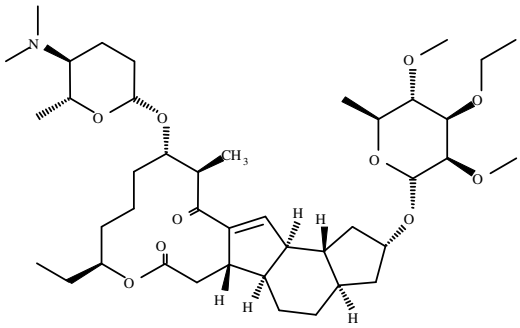
### *Technical material*

| Property | Results | | Reference |
|---|---|---|---|
| Appearance: | Off-white solid | | Madsen & Jennings, 2005 FAPC-052-004 |
| Odour | Musty odour | | Madsen & Jennings, 2005 FAPC-052-004 |
| Solvent solubility at 20 ºC | Methanol | > 250 g/L | Comb, 2005 NAFST-05-078 |
| | Acetone | > 250 g/L | |
| | Xylene | > 250 g/L | |
| | 1,2-Dichloroethane | > 250 g/L | |
| | Ethyl acetate | > 250 g/L | |
| | n-Octanol | 132 g/L | |
| Formulations: | Water dispersible granule (WG) formulation containing 250 g ai/kg<br>Suspension concentrate (SC) formulation containing 120 g ai/L | | |

## METABOLISM AND ENVIRONMENTAL FATE

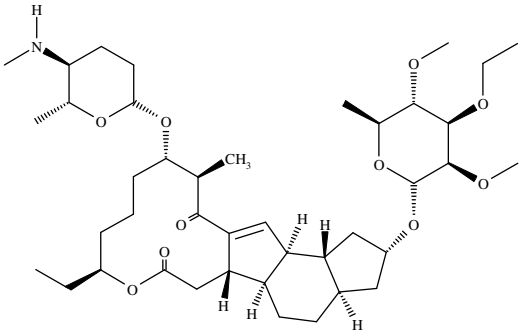
The following links manufacturer code number and structure or description of the compounds appearing in the various metabolism and environmental fate studies.

Structure of compounds appearing in metabolism and environmental fate studies



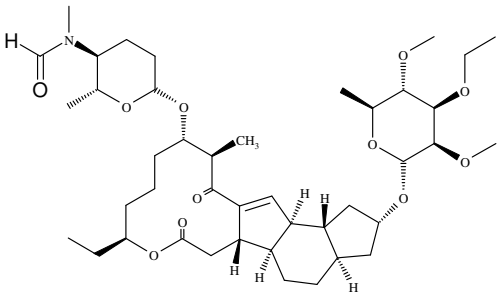
XDE-175-J

Plants, animals, soil, water, rotational crops

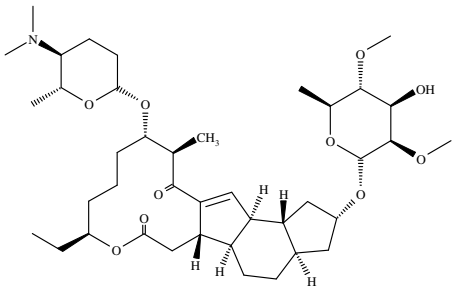


N-demethyl-175-J

Plants, animals, soil, water, rotational crops

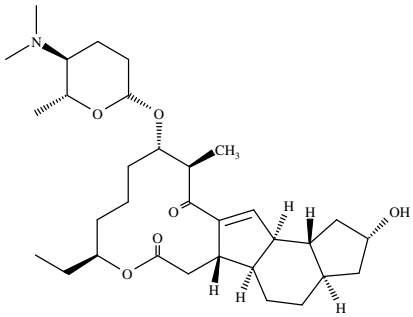


N-formyl-175-J
Plants



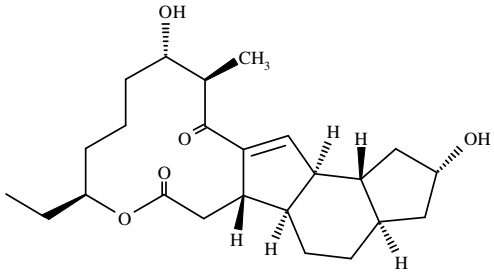
3-O-deethyl-175-J

Plants



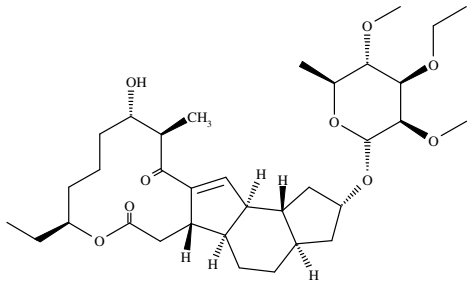
C9-pseudoaglycone-175-J

Plants



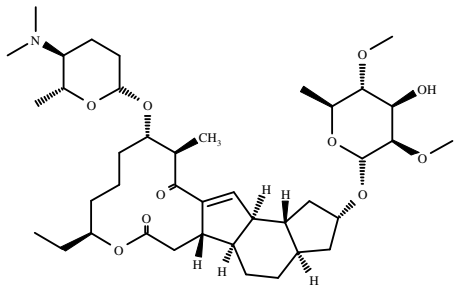
Aglycone-175-J

Plants



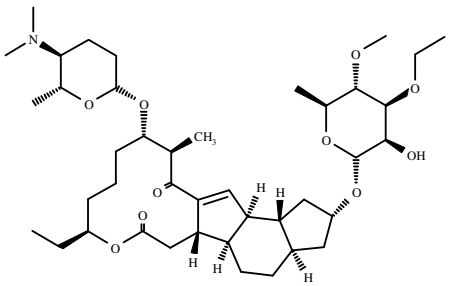
C17-pseudoaglycone-175-J

Soil, plants



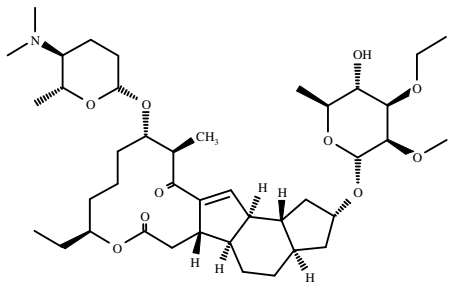
3’-O-deethyl-175-J

Animals



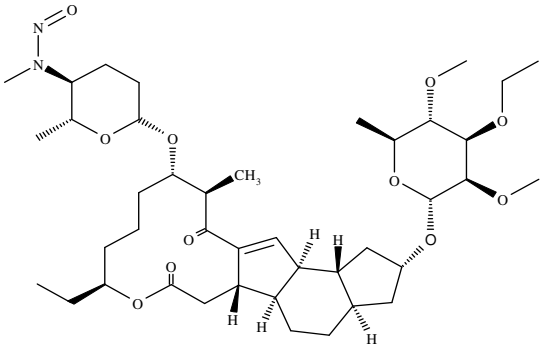
2’-O-demethyl-175-J

Animals



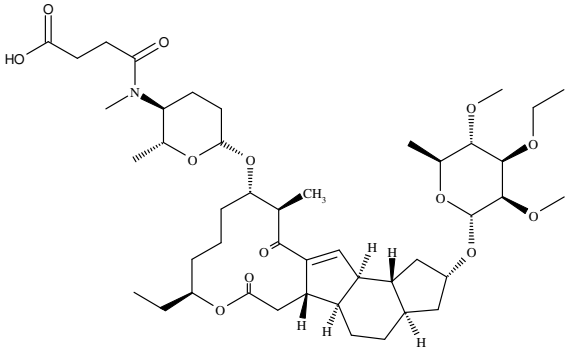
4’-O-demethyl-175-J

Animals



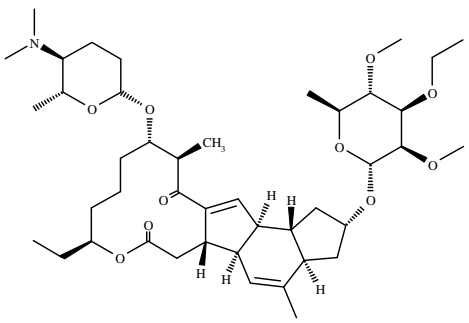
N-demethyl-N-nitroso-175-J

Soil



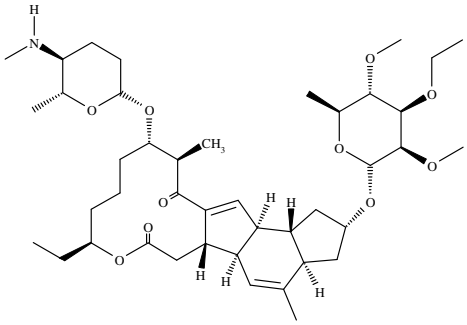
N-succinyl-175-J

Soil



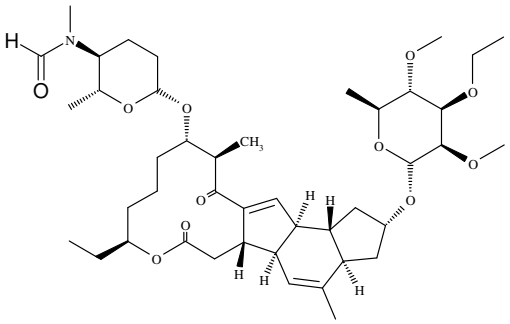
XDE-175-L

Plants, animals, soil, water, rotational crops



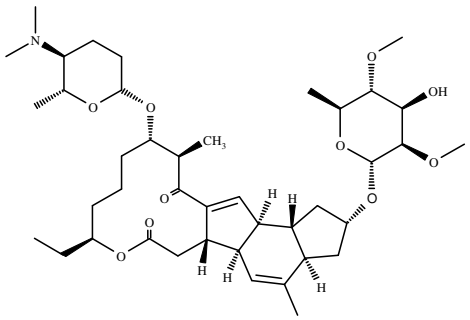
N-demethyl-175-L

Plants, animals, soil, water, rotational crops



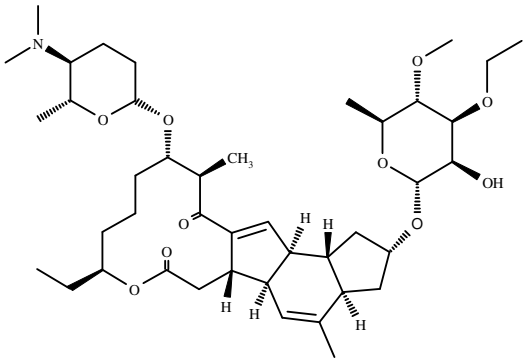
N-formyl -175-L

Plants



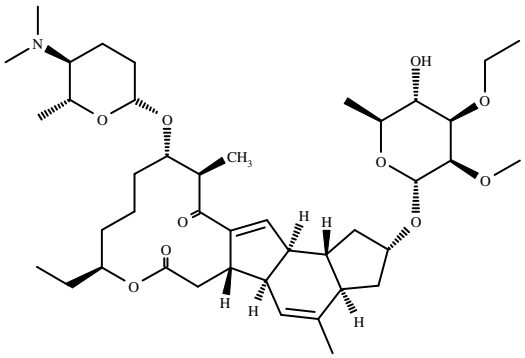
3'-O-deethyl-175-L

Animals



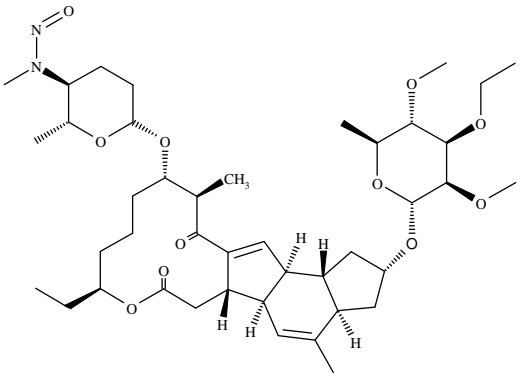
2'-O-demethyl-175-L

Animals



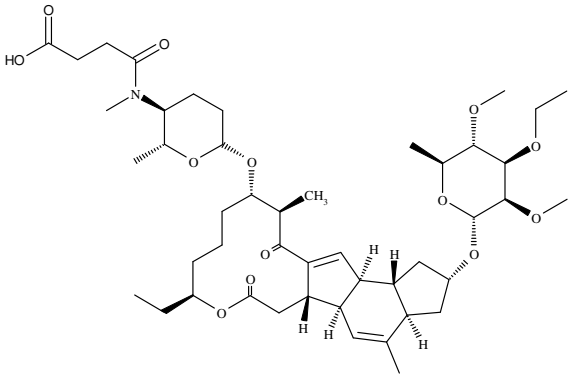
4'-O-demethyl-175-L

Animals



N-demethyl-N-nitroso-175-L

Soil



N-succinyl-175-L

Soil

Radio-labelled XDE-175-J and XDE-175-J Used in Metabolism Studies

In plant and animal metabolism studies, XDE-175-J or XDE-175-L which were uniformly labelled with $^{14}C$ in the macrolide ring was used. No radioactive carbon was incorporated into either sugar group.

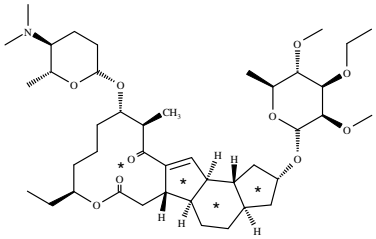
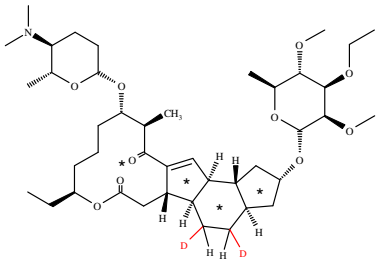
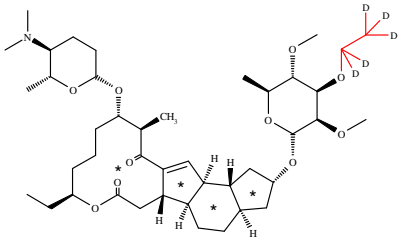
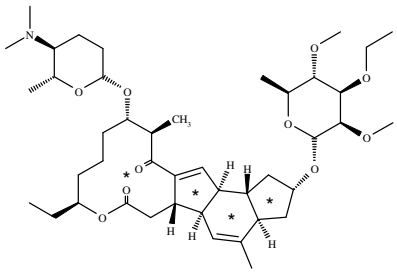
Three forms of $^{14}C$-XDE-175-J produced through fermentation in the presence of $^{14}C$-acetate were combined to give a mixture of 1:1:1 ratio (w/w/w) with nominal specific activity of 11.62 µCi/mg (8.72 mCi/mmol or 429,940 Bq/mg. One form was ethylated at the 3'-position of the rhamnose and the 5,6-position of the macrolide ring then reduced to give $^{14}C$ -XDE-175-J (INV1947). A second form was likewise reduced but was ethylated using a deuterated alkylating agent to give $^{14}C$-XDE-175-J (D5). A third form was ethylated in the same manner as the first but was reduced in

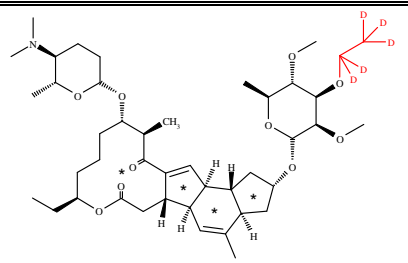
the presence of deuterium to give $^{14}$C-XDE-175-J (D2). These three forms aided in the mass spectrometric analyses of metabolites in confirming that peaks of interest were related to the test materials.

Two forms of $^{14}$C-XDE-175-L were combined to give a mixture of approximately one to one ratio (w/w) with nominal specific activity of 7.57 µCi/mg (5.77 mCi/mmol or 280,090 Bq/mg). One form was ethylated at the 3' position of the rhamnose to give $^{14}$C-XDE-175-L. A second form was ethylated using a deuterated alkylating agent to give $^{14}$C-XDE-175-L (D5). Since the 5,6-positions of the material cannot be reduced, there is no comparable D2 material formed. These two forms aided in the mass spectrometric analyses of metabolites confirming that peaks of interest were related to the test materials.

The following table shows the position of radioactive carbon in the test materials used.

Table 1 Radio-labelled compounds used in the metabolism studies

| Name | Structure |
|---|---|
| $^{14}$C-XDE-175-J |  |
| $^{14}$C-XDE-175-J (D2) |  |
| $^{14}$C-XDE-175-J (D5) |  |
| $^{14}$C-XDE-175-L |  |

| Name | Structure |
|---|---|
| $^{14}$C-XDE-175-L (D5) |  |

***Animal metabolism***

The Meeting received information on the results of studies on lactating goats and laying hens.

Lactating goats: Two lactating goats (Alpine × Nubian Cross breed, 4 years old, 47-52 kg) were given oral doses of either $^{14}$C-XDE-175-J (14.9 mg/animal/day) or $^{14}$C-XDE-175-L (14.8 mg/animal/day) equivalent to 10–11 ppm in the total diet (on a dry weight basis) once daily via balling gun for five consecutive days (Magnussen, *et. al*., 2005; Report 040088). Milk was collected twice a day throughout the dosing period and urine and faeces at 24-h intervals. The mean daily milk production of the control, XDE-175-J treated or XDE-175-L treated goat was 1.01, 1.70 or 1.44 L respectively. Twenty-one hours (±1 h) after administering the final dose, the animals were sacrificed and liver, kidney, fat and muscle samples were collected for analysis.

Among excreta, a total of 51.1% of the administered radioactivity was recovered in faeces in the case of $^{14}$C-XDE-175-J and 78.3% in the case of $^{14}$C-XDE-175-L. A total radioactivity recovered in urine was not significant: 0.17% for $^{14}$C-XDE-175-J; and 0.03% for $^{14}$C-XDE-175-L. The cage rinse contained less than 0.01% of the administered dose.

Radioactivity started to appear in milk within 24 h after the first administration and seemed to reach a plateau by the fifth administration. The maximum total radioactive residues expressed in mg-equivalents of respective compound/kg were 0.047 mg/kg in the second milking of day 3 and the first milking of day 4 for $^{14}$C-XDE-175-L and 0.039 mg/kg in the first milking of day 5 for $^{14}$C-XDE-175-L. In the course of the study, no milk sample contained more than 0.07% of the administered dose. Only about 0.3% of the total administered dose of $^{14}$C-XDE-175-J and 0.2% of that of $^{14}$C-XDE-175-L was recovered from all the collected milk samples.

Radioactive residues were relatively low in edible tissues after sacrifice: the concentrations in fat were the highest at 0.235 and 0.119 mg/kg and in other tissues they were, in decreasing order, in liver at 0.116 and 0.099 mg/kg, in kidney at 0.065 and 0.047 mg/kg and in muscle at 0.017 and 0.015 mg/kg for $^{14}$C-XDE-175-J and $^{14}$C-XDE-175-L, respectively (Table 2). For both compounds, only about 0.3% and 0.5% of the total dose was accounted for in the edible tissues for XDE-175-L and XDE-175-J, respectively. The results showed that radioactivity of these compounds were not readily transferred into these edible tissues and that the residues accumulate more in tissues with higher fat content.

Table 2 Total radioactive residues (TRR) in the milk and edible tissues of lactating goats given either $^{14}$C-XDE-175-J or $^{14}$C-XDE-175-L

| Tissue | | Total Radioactive Residues (mg/kg) | |
|---|---|---|---|
| | | XDE-175-J | XDE-175-L |
| Milk | Day 1 1st | - | - |
| | Day 1 2nd | 0.001 | 0.011 |
| | Day 2 1st | 0.012 | 0.015 |
| | Day 2 2nd | 0.023 | 0.014 |
| | Day 3 1st | 0.019 | 0.021 |

| Tissue | | Total Radioactive Residues (mg/kg) | |
|---|---|---|---|
| | | XDE-175-J | XDE-175-L |
| | Day3 2nd | 0.047 | 0.014 |
| | Day 4 1st | 0.047 | 0.029 |
| | Day 4 2nd | 0.037 | 0.028 |
| | Day 5 1st | 0.029 | 0.039 |
| | Day 5 2nd | 0.037 | 0.029 |
| Fat | | 0.235 | 0.119 |
| Liver | | 0.116 | 0.099 |
| Kidney | | 0.065 | 0.047 |
| Muscle | | 0.017 | 0.015 |

Radioactive residues in tissues and milk were extracted under mild condition: fat was extracted with hexane; liver, kidney and muscle with acetonitrile/water; and milk with acetonitrile. The organic extract was subjected to SPE cleanup prior to analysis. In tissues and milk, radioactive residues extracted in organic solvents were ≥ 85% of TRR, except for liver where residues in organic solvent were 57–71% of TRR. Radioactive residues extracted in aqueous layer were ≤ 5% of TRR for all tissues and milk, except for $^{14}$C-XDE-175-J in liver where the aqueous extract contained 18% of TRR. Less than 15% of TRR or 0.025 mg/kg were unextractable from all tissues except liver. From liver, 23–35% of TRR were unextractable.

Characterization and identification of the organo-soluble residues from the muscle, milk and excreta samples showed the primary residue to be XDE-175-J or XDE-175-L, indicating that minimal metabolism had occurred. No residue components other than the unchanged parent compounds were identified in milk, kidney or fat. Radioactive residues in liver consisted of XDE-175-J or XDE-175-L, a metabolite that was tentatively identified as N-demethyl-175-J or –L at very low levels (< 2% of the TRR), and an unidentified metabolite (Metabolite J1 or Metabolite L1) that was more polar than most of the available reference standards. Radioactive residues in muscle also consisted primarily of XDE-175-J or XDE-175-L and much lesser amounts of what seemed to be the same unidentified metabolite as found in liver. Table 3 shows summaries of the characterised radioactive residues. Overall, for both compounds 77–99% of TRR were extracted.

Table 3 Characterization and identification of radioactive residues in the tissues and milk of lactating goats following oral doses of $^{14}$C-XDE-175-J or $^{14}$C-XDE-175-L

| Compound | Muscle | | Fat | | Kidney | | Liver | | Day 5 Milk [a] | |
|---|---|---|---|---|---|---|---|---|---|---|
| | mg/kg | % TRR | mg/kg | % TRR | mg/kg | % TRR | mg/kg | % TRR | mg/kg | % TRR |
| Oral Dose of $^{14}$C-XDE-175-J | | | | | | | | | | |
| XDE-175-J | 0.007 | 42.3 | 0.190 | 80.8 | 0.035 | 53.9 | 0.035 | 29.8 | 0.029 | 84.4 |
| N-Demethyl-175-J | - | - | - | - | - | - | 0.002 | 1.8 | - | - |
| Metabolite J1 | 0.001 | 5.5 | - | - | < 0.001 | - | 0.012 | 10.0 | - | - |
| Total identified | 0.007 | 42.3 | 0.190 | 80.8 | 0.035 | 53.9 | 0.037 | 31.6 | 0.029 | 84.4 |
| Total characterised [b] | 0.009 | 47.2 | 0.021 | 8.8 | 0.021 | 31.8 | 0.050 | 43.3 | 0.005 | 14.7 |
| Total extracted | 0.016 | 89.5 | 0.211 | 89.6 | 0.056 | 85.7 | 0.087 | 74.9 | 0.034 | 99.1 |
| Total unextractable | 0.002 | 10.7 | 0.025 | 10.5 | 0.009 | 14.5 | 0.029 | 25.2 | < 0.001 | 1.0 |
| Total measured (TRR in ( )) | (0.017) | 100 | (0.235) | 100 | (0.065) | 100 | (0.116) | 100 | (0.034) | 100 |

| Compound | Muscle | | Fat | | Kidney | | Liver | | Day 5 Milk [a] | |
|---|---|---|---|---|---|---|---|---|---|---|
| | mg/kg | % TRR | mg/kg | % TRR | mg/kg | % TRR | mg/kg | % TRR | mg/kg | % TRR |
| Oral Dose of $^{14}$C-XDE-175-L | | | | | | | | | | |
| XDE-175-L | 0.007 | 45.8 | 0.086 | 72.3 | 0.030 | 64.3 | 0.026 | 25.9 | 0.042 | 84.2 |
| N-Demethyl-175-L | - | - | - | - | - | - | 0.001 | 1.2 | - | - |
| Metabolite L1 | 0.003 | 17.7 | - | - | - | - | 0.027 | 26.9 | - | - |
| Total identified | 0.007 | 45.8 | 0.086 | 72.3 | 0.030 | 64.3 | 0.027 | 27.1 | 0.042 | 84.2 |
| Total characterised | 0.008 | 47.3 | 0.018 | 14.8 | 0.012 | 21.8 | 0.049 | 49.5 | 0.007 | 13.6 |
| Total extracted | 0.015 | 93.1 | 0.104 | 87.1 | 0.042 | 86.1 | 0.076 | 76.6 | 0.049 | 97.8 |
| Total unextractable | 0.001 | 6.9 | 0.016 | 12.9 | 0.007 | 13.9 | 0.023 | 23.4 | 0.002 | 2.2 |
| Total measured (TRR in ( )) | (0.015) | 100 | (0.119) | 100 | (0.047) | 100 | (0.099) | 100 | (0.049) | 100 |

[a] Prior to extraction and analysis, aliquots of the Day 5 PM milk sample were re-assayed by liquid scintillation counter to confirm the TRR levels.

[b] Characterised but not identified.

Laying hens: Laying hens (Bovan White Leghorn; 24 week old; 1.3–1.6 kg) were given either $^{14}$C-XDE-175-J or $^{14}$C-XDE-175-L, each at 1.25 or 1.75 mg/bird/day, equivalent to10 ppm in the diet, once daily via balling gun for seven consecutive days with (Smith-Drake, 2005; Report 040087). Each dosing group consisted of ten birds. Eggs were collected twice daily and excreta once daily. The hens were sacrificed within 22 ± 3 h of the final dose and the liver, muscle (breast and thigh), fat (abdominal), and skin with subcutaneous fat was collected for analysis.

The overall recovery of the dose was 95 and 94% for $^{14}$C-XDE-175-J and $^{14}$C-XDE-175-L, respectively. Approximately 93 and 91% of the administered dose of $^{14}$C-XDE-175-J and $^{14}$C-XDE-175-L, respectively, was recovered in the excreta.

Recovery of the administered dose in eggs and tissues was low for both $^{14}$C-XDE-175-J (0.40+0.98%) and $^{14}$C-XDE-175-L (0.93 + 2.1%). TRR in eggs increased over the experimental period and reached a maximum of 0.204 and 0.488 mg/kg for $^{14}$C-XDE-175-J and $^{14}$C-XDE-175-L, respectively, on Day 7. TRR in the tissues were highest in abdominal fat (1.04–2.46 mg/kg), followed by skin with subcutaneous fat (0.66–1.41 mg/kg), liver (0.53–0.90 mg/kg), eggs (0.2–0.49 mg/kg in Day 7) and muscle (0.05–0.11 mg/kg) (Table 4). TRR found in eggs and tissues of hens dosed with $^{14}$C-XDE-175-L were approximately twice as high as those from hens dosed with $^{14}$C-XDE-175-J. There is a tendency for these test compounds to accumulate more in tissues with higher fat content.

Table 4 Total radioactive residues (TRR) in the eggs and edible tissues of hens given either $^{14}$C-XDE-175-J or $^{14}$C-XDE-175-L

| Tissue | | Total Radioactive Residues (mg/kg) | |
|---|---|---|---|
| | | XDE-175-J | XDE-175-L |
| Egg | Day 1 | 0.000 | 0.000 |
| | Day 2 | 0.032 | 0.036 |
| | Day 3 | 0.067 | 0.123 |
| | Day 4 | 0.114 | 0.225 |
| | Day 5 | 0.161 | 0.340 |
| | Day 6 | 0.185 | 0.420 |
| | Day 7 | 0.204 | 0.488 |
| Abdominal fat | | 1.04 | 2.46 |
| Skin/Fat | | 0.661 | 1.41 |

| Tissue | Total Radioactive Residues (mg/kg) | |
|---|---|---|
| | XDE-175-J | XDE-175-L |
| Muscle | 0.050 | 0.108 |
| Liver | 0.535 | 0.902 |

Radioactive residues were readily extracted under the mild condition. Following hexane extraction of abdominal fat and skin with fat, 94–97% of TRR were extracted in hexane layer and 3–6% of TRR were unextractable. Partition of radioactive residues in muscle, liver, and egg samples between acetonitrile/water and dichloromethane resulted in 73–88% of TRR in the organic phase, < 1% of TRR in the aqueous phase, and 12–20% of TRR unextractable. Acid extraction of the liver tissue remaining after the above-mentioned extraction further released 7–8% of TRR as organo-soluble residues with 4–7% of TRR still not extracted. Table 5 shows summaries of the identified/characterised residues. Unextracted radioactivity was less than 7% of the TRR in all samples except muscle and eggs in which it was 12–20%.

Neither of these two test compound was extensively or rapidly metabolized in hens. Unchanged XDE-175-J or XDE-175-L remained as the primary residue in the egg and tissues.

Table 5 Characterization of radioactive residues in hen tissues following oral doses of $^{14}$C-XDE-175-J or $^{14}$C-XDE-175-L

| Compound | Abdominal fat | | Skin with fat | | Muscle | | Liver | | Day 4 Egg | |
|---|---|---|---|---|---|---|---|---|---|---|
| | mg/kg | % TRR | mg/kg | % TRR | mg/kg | % TRR | mg/kg | % TRR | mg/kg | % TRR |
| Oral Dose of $^{14}$C-XDE-175-J | | | | | | | | | | |
| XDE-175-J | 0.723 | 69.6 | 0.531 | 80.2 | 0.034 | 67.8 | 0.069 | 13.0 | 0.068 | 58.4 |
| 3'-O-deethyl-175-J | 0.019 | 1.8 | - | - | - | - | 0.093 | 17.7 | - | - |
| O-demethyl-175-J | 0.059 | 5.7 | 0.022 | 3.3 | 0.002 | 3.2 | 0.034 | 6.5 | - | - |
| Total identified | 0.801 | 77.1 | 0.553 | 83.5 | 0.036 | 71.0 | 0.196 | 37.2 | 0.068 | 58.4 |
| Total characterised [a] | 0.205 | 19.9 | 0.086 | 13.0 | 0.005 | 10.5 | 0.294 | 55.8 | 0.028 | 25.6 |
| Total extracted | 1.006 | 97.0 | 0.639 | 96.5 | 0.041 | 80.5 | 0.490 | 93.0 | 0.096 | 84.0 |
| Total unextractable | 0.032 | 3.0 | 0.023 | 3.5 | 0.010 | 19.5 | 0.037 | 7.0 | 0.019 | 16.0 |
| Total measured (TRR in ( )) | (1.04) | 100 | (0.662) | 100 | (0.050) | 100 | (0.526) | 100 | (0.115) | 100 |
| Oral Dose of $^{14}$C-XDE-175-L | | | | | | | | | | |
| XDE-175-L | 1.366 | 55.5 | 0.784 | 55.6 | 0.048 | 44.5 | 0.105 | 11.7 | 0.111 | 48.9 |
| 3'-O-deethyl-175-L | 0.128 | 5.2 | 0.079 | 5.6 | 0.006 | 5.4 | 0.115 | 12.8 | 0.029 | 12.5 |
| O-demethyl-175-L | 0.479 | 19.5 | 0.239 | 16.9 | 0.020 | 17.8 | 0.135 | 15 | 0.030 | 13.4 |
| N-demethyl-175-L | - | - | - | - | - | - | 0.015 | 1.7 | - | - |
| Total identified | 1.973 | 80.2 | 1.102 | 78.1 | 0.074 | 67.7 | 0.370 | 41.2 | 0.170 | 74.8 |
| Total characterised [a] | 0.414 | 16.8 | 0.226 | 15.9 | 0.016 | 14.8 | 0.493 | 54.5 | 0.029 | 13.2 |
| Total extracted | 2.387 | 97.0 | 1.328 | 94.0 | 0.090 | 82.5 | 0.863 | 95.7 | 0.199 | 88 |
| Total unextractable | 0.075 | 3.0 | 0.084 | 6.0 | 0.019 | 17.5 | 0.039 | 4.4 | 0.027 | 12 |
| Total measured (TRR in ( )) | (2.46) | 100 | (1.41) | 100 | (0.108) | 100 | (0.902) | 100 | (0.226) | 100 |

[a] Characterised but not identified.

Unchanged parent compound was the primary residue component in milk and all ruminant tissues as well as eggs and all avian tissues except liver for both XDE-175-J and XDE-175-L. The liver and muscle of the goat treated with XDE-175-J or XDE-175-L contained one unidentified metabolite present at less than 10 or 27% of TRR, respectively. The ruminant liver also contained a

minor amount of N-demethyl-175-J or N-demethyl-175-L (≤ 2% TRR). In hen liver, the O-deethyl- and O-demethyl metabolites were also found.

Metabolism of spinetoram appears to be primarily through dealkylation of the rhamnose sugar to give the O-deethyl and/or O-demethyl (two possible isomers) metabolites. Many other minor metabolites were also observed with each being present at levels lower than 10% of the TRR. Proposed metabolic pathways for spinetoram in livestock are shown in Figure 1 (XDE-175-J) and Figure 2 (XDE-175-L). The metabolism spinetoram in ruminant paralleled that in hens.

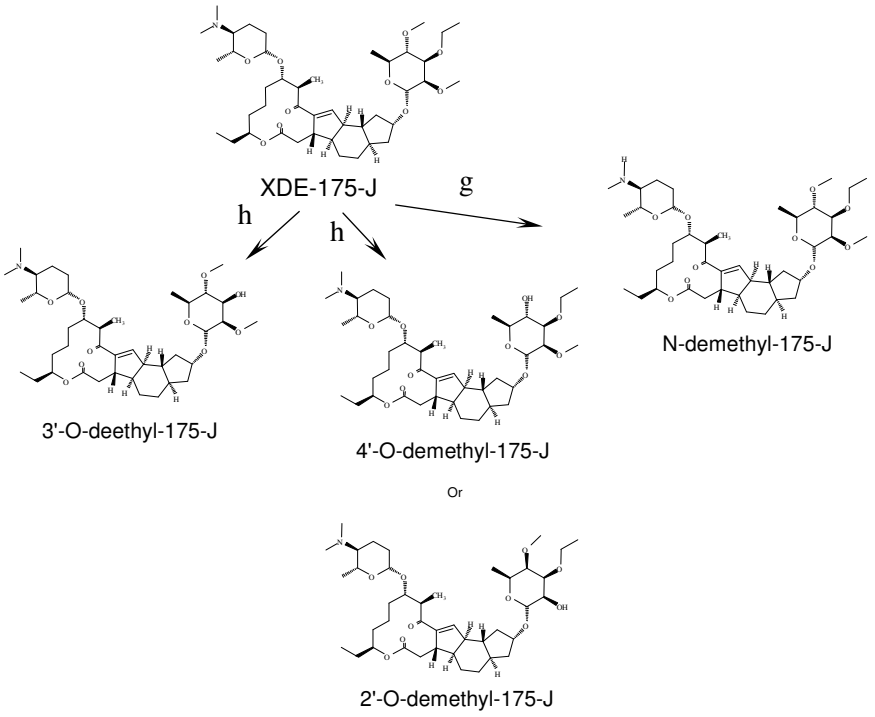



Figure 1 Proposed metabolic pathway of XDE-175-J in livestock

g, goat; h, hen.

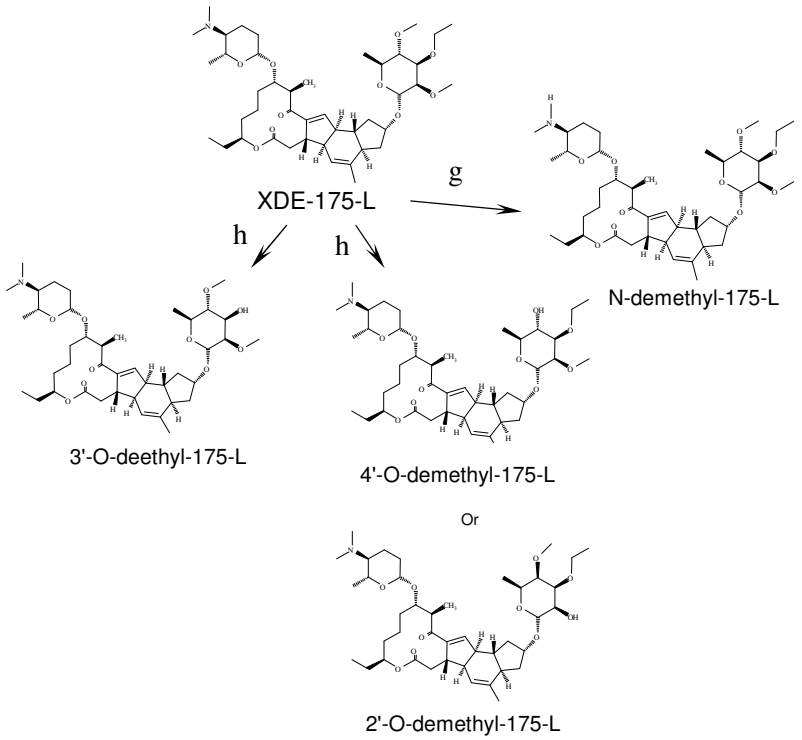



Figure 2 Proposed Metabolic Pathway of XDE-175-L in Livestock

g, goat; h, hen.

### *Plant Metabolism*

The Meeting received information on the fate of spinetoram after foliar applications in apples, lettuce and turnips representing the fruits, leafy crops and root crops, respectively.

*Apple*

A branch with immature fruits of individual Granny Smith apple tree was treated with single foliar application of either $^{14}$C-XDE-175-J or $^{14}$C-XDE-175-L at the rate of 1.8 kg ai/ha (4.8×) or 1.1 kg ai/ha (8.9×), respectively (Byrne, *et. al*., 2005; Report 040050). Immature apples and leaves were collected 0, 1, 3, 7, and 14 days after treatment (DAT). Covered apples, as dark samples, were also collected 3 DAT and 7 DAT (covered from 3–7 DAT). Mature apples were collected 30 DAT, as well as leaf samples. In addition, one branch on each treated tree was covered with plastic prior to application for testing translocation to an untreated branch. Apple fruits and leaves of the untreated branches were collected 30 DAT.

The collected samples were washed with acetonitrile and dichloromethane. The total radioactive residues (TRR) were determined as the sum of the residues in the washings and the washed fruit or leaves. In the fruit obtained from the $^{14}$C-XDE-175-J treated branch (0–30 DAT), 78–93% of TRR were found in the acetonitrile washings and 4–11% in the dichloromethane washings. Peels contained 2–11% of TRR while pulp contained less than 1% (≤ 0.007 mg/kg). In fruits from $^{14}$C-XDE-175-L treated branch (0-30 DAT), 46–85% of TRR were found in acetonitrile washings and 10–21% in dichloromethane washings. Peel contained 6–33% of TRR while pulp contained less than 4%.

In leaves from the $^{14}$C-XDE-175-J treated branch (0–30 DAT), 67–88% of TRR were recovered in acetonitrile washings and 6–11% of TRR in dichloromethane washings. Washed leaves contained 5–23% of TRR. In leaves from $^{14}$C-XDE-175-L treated branch (0–30 DAT), 55–79% of

TRR were recovered in acetonitrile washings and 10–17% of TRR in dichloromethane washings. Washed leaves contained 11–31% of TRR.

The leaves and peel from both treatments, and pulp from the $^{14}$C-XDE-175-L treatment were extracted with acetonitrile:water (80:20, v/v). Only pulp from the $^{14}$C-XDE-175-L treated trees was extracted since TRR in pulp from the $^{14}$C-XDE-175-J treated trees were ≤ 1% of TRR (≤ 0.007 mg/kg). Radioactive residues unextractable were < 10% of TRR in or on all samples. Pooled acetonitrile washing, dichloromethane washing, and extracts were analysed. Tables 6 and 7 summarize the identified residues. Several minor metabolites assumed to be structurally similar to the parent compound were also detected in the treated apples and leaves, each at ≤ 7.5% of TRR. A multi-component mixture of extensively degraded compounds represented up to 39–77% of TRR but each component was less than 1% of TRR.

Radioactive residues in the apple fruits from $^{14}$C-XDE-175-L treated tree shows a tendency to be higher when exposed than those in covered (dark) apple samples.

In the translocation experiment, TRR were below the limit of quantification of 0.001 mg/kg in apple fruits and 0.053 mg/kg and 0.009 mg/kg in leaves collected from the $^{14}$C-XDE-175-J and $^{14}$C-XDE-175-L treated trees, respectively indicating that translocation is negligible. As the concentrations in tissues were very low, no further analysis was conducted.

Table 6 Characterization and identification of radioactive residues in apple fruit washings and extracts [a] following application of $^{14}$C-XDE-175-J at 1.8 kg ai/ha or $^{14}$C-XDE-175-L at 1.1 kg ai/ha

| Compound | %TRR | mg/kg | %TRR | mg/kg | %TRR | mg/kg | %TRR | mg/kg |
|---|---|---|---|---|---|---|---|---|
| Application of $^{14}$C-XDE-175-J at 1.8 kg ai/ha | | | | | | | | |
| | 0 DAT | | 1 DAT | | 3 DAT (dark) | | 3 DAT | |
| XDE-175-J | 82.2 | 0.715 | 77.2 | 1.286 | 61.4 | 0.625 | 54.8 | 0.729 |
| N-demethyl-175-J | 5.0 | 0.044 | 7.0 | 0.116 | 10.2 | 0.104 | 8.4 | 0.112 |
| N-formyl-175-J | 1.7 | 0.015 | 4.1 | 0.068 | 3.1 | 0.031 | 3.6 | 0.048 |
| C9-pseudoaglycone-175-J | 0.5 | 0.004 | 0.1 | 0.002 | 0.8 | 0.008 | 1.3 | 0.018 |
| 3-O-deethyl-175-J | 1.2 | 0.010 | 3.9 | 0.065 | - | - | - | - |
| Total identified | 90.6 | 0.788 | 92.3 | 1.534 | 75.5 | 0.768 | 68.1 | 0.907 |
| Total characterised [b] | 9.2 | 0.081 | 7.2 | 0.130 | 24.4 | 0.250 | 31.5 | 0.418 |
| Total extractable | 99.8 | 0.869 | 99.5 | 1.664 | 99.9 | 1.018 | 99.6 | 1.325 |
| Total unextractable | 0.4 | 0.003 | 0.4 | 0.006 | 0.3 | 0.003 | 0.9 | 0.011 |
| Total measured (TRR in ( )) | 100 | (0.870) | 100 | (1.67) | 100 | (1.02) | 101 | (1.33) |
| | 7 DAT (dark) | | 7 DAT | | 14 DAT | | 30 DAT | |
| XDE-175-J | 44.4 | 0.352 | 43.3 | 0.501 | 44.3 | 0.485 | 18.9 | 0.135 |
| N-demethyl-175-J | 8.3 | 0.066 | 9.5 | 0.110 | 9.1 | 0.100 | 6.3 | 0.044 |
| N-formyl-175-J | 5.9 | 0.047 | 5.1 | 0.059 | 3.0 | 0.033 | 3.5 | 0.024 |
| C9-pseudoaglycone-175-J | 1.5 | 0.011 | 2.9 | 0.033 | 3.8 | 0.043 | 1.5 | 0.010 |
| 3-O-deethyl-175-J | 0.1 | 0.001 | 0.1 | 0.001 | 3.2 | 0.035 | 2.0 | 0.014 |
| Total identified | 60.2 | 0.477 | 60.9 | 0.704 | 63.4 | 0.696 | 32.2 | 0.227 |
| Total characterised [b] | 37.7 | 0.299 | 38.3 | 0.445 | 35.0 | 0.381 | 64.3 | 0.462 |
| Total extracted | 97.9 | 0.776 | 99.2 | 1.149 | 98.4 | 1.077 | 96.5 | 0.689 |
| Total unextractable | 1.6 | 0.013 | 1.6 | 0.02 | 1.3 | 0.014 | 3.8 | 0.027 |
| Total measured (TRR in ( )) | 100 | (0.793) | 101 | (1.16) | 100 | (1.09) | 100 | (0.713) |
| Application of $^{14}$C-XDE-175-L at 1.1 kg ai/ha | | | | | | | | |
| | 0 DAT | | 1 DAT | | 3 DAT (dark) | | 3 DAT | |
| XDE-175-L | 42.7 | 0.184 | 18.4 | 0.115 | 17.7 | 0.237 | 12.9 | 0.115 |
| N-demethyl-175-L | 7.9 | 0.034 | 4.0 | 0.025 | 3.4 | 0.046 | 3.6 | 0.032 |

| Compound | %TRR | mg/kg | %TRR | mg/kg | %TRR | mg/kg | %TRR | mg/kg |
|---|---|---|---|---|---|---|---|---|
| N-formyl-175-L | 1.6 | 0.007 | 2.3 | 0.014 | 3.5 | 0.047 | 0.9 | 0.008 |
| Total identified | 52.2 | 0.225 | 24.7 | 0.154 | 24.6 | 0.330 | 17.4 | 0.155 |
| Total characterised [b] | 45.1 | 0.194 | 74.5 | 0.466 | 71.6 | 0.958 | 75.9 | 0.672 |
| Total extracted | 97.3 | 0.419 | 99.2 | 0.620 | 96.2 | 1.288 | 93.3 | 0.827 |
| Total unextractable | 2.0 | 0.009 | 1.2 | 0.007 | 3.0 | 0.041 | 7.0 | 0.063 |
| Total measured (TRR in ( )) | 99 | (0.431) | 100 | (0.626) | 99 | (1.34) | 100 | (0.888) |
| | 7 DAT (dark) | | 7 DAT | | 14 DAT | | 30 DAT | |
| XDE-175-L | 8.5 | 0.088 | 1.3 | 0.005 | - | - | - | - |
| N-demethyl-175-L | 2.7 | 0.027 | 1.0 | 0.004 | 0.3 | 0.002 | 0.4 | 0.003 |
| N-formyl-175-L | 1.0 | 0.010 | 1.0 | 0.004 | 0.2 | 0.001 | 0.4 | 0.003 |
| Total identified | 12.2 | 0.125 | 3.3 | 0.013 | 0.5 | 0.003 | 0.8 | 0.006 |
| Total characterised | 82.6 | 0.858 | 86.6 | 0.307 | 90.6 | 0.477 | 89.5 | 0.650 |
| Total extracted | 94.8 | 0.983 | 89.9 | 0.320 | 91.1 | 0.48 | 90.3 | 0.656 |
| Total unextractable | 4.0 | 0.042 | 7.2 | 0.025 | 6.9 | 0.036 | 9.0 | 0.066 |
| Total measured (TRR in ( )) | 99 | (1.04) | 97 | (0.356) | 98 | (0.528) | 99 | (0.728) |

[a] Sum of residues of pooled fruit washings and peel/pulp extracts by HPLC. Only the pulp from XDE-175-L treated tree was extracted.

[b] Characterised but not identified

Table 7 Characterization and identification of radioactive residues in apple leaf washings [a] and extracts following application of $^{14}$C-XDE-175-J at 1.8 kg ai/ha or $^{14}$C-XDE-175-L at 1.1 kg ai/ha (Byrne, *et. al.*, 2005)

| Compound | %TRR | mg/kg | %TRR | mg/kg | %TRR | mg/kg |
|---|---|---|---|---|---|---|
| Application of $^{14}$C-XDE-175-J at 1.8 kg ai/ha | | | | | | |
| | 0 DAT | | 1 DAT | | 3 DAT | |
| XDE-175-J | 80.3 | 105 | 78.6 | 100.0 | 42.4 | 71.27 |
| N-demethyl-175-J | 9.1 | 11.9 | 10.4 | 13.35 | 14.8 | 24.83 |
| N-formyl-175-J | 2.4 | 3.20 | 2.4 | 3.07 | 0.7 | 1.15 |
| C9-pseudoaglycone-175-J | 0.9 | 1.11 | 0.2 | 0.21 | 2.3 | 3.86 |
| 3-O-deethyl-175-J | 0.1 | 0.10 | 0.9 | 1.07 | 4.2 | 6.95 |
| Total identified | 92.8 | 122 | 92.5 | 117.69 | 64.4 | 108.06 |
| Total characterised [b] | 9.6 | 12.8 | 8.5 | 10.92 | 35.1 | 59.23 |
| Total extracted | 102.4 | 134 | 101.0 | 128.61 | 99.5 | 167.29 |
| Total unextractable | 0.3 | 0.33 | 0.2 | 0.29 | 0.6 | 1.03 |
| Total measured (TRR in ( )) | 103 | (131) | 101 | (127) | 100 | (168) |
| | 7 DAT | | 14 DAT | | 30 DAT | |
| XDE-175-J | 37.6 | 58.40 | 24.0 | 34.36 | 19.9 | 27.77 |
| N-demethyl-175-J | 12.0 | 18.70 | 9.4 | 12.45 | 7.5 | 10.50 |
| N-formyl-175-J | 3.4 | 5.39 | 3.0 | 4.34 | 2.2 | 3.07 |
| C9-pseudoaglycone-175-J | 1.7 | 2.63 | 3.6 | 5.11 | 3.1 | 4.37 |
| 3-O-deethyl-175-J | 2.2 | 3.40 | 4.3 | 6.15 | 2.5 | 3.45 |
| Total identified | 56.9 | 88.53 | 44.3 | 63.40 | 35.2 | 49.15 |
| Total characterised [b] | 41.9 | 65.06 | 54.1 | 77.51 | 61.0 | 84.98 |
| Total extracted | 98.8 | 153.59 | 98.4 | 140.92 | 96.2 | 134.13 |
| Total unextractable | 0.5 | 0.78 | 1.0 | 1.48 | 3.3 | 4.54 |

| Compound | %TRR | mg/kg | %TRR | mg/kg | %TRR | mg/kg |
|---|---|---|---|---|---|---|
| Total measured (TRR in ( )) | 99 | (155) | 99 | (143) | 100 | (140) |
| Application of $^{14}$C-XDE-175-L at 1.1 kg ai/ha | | | | | | |
| | 0 DAT | | 1 DAT | | 3 DAT | |
| XDE-175-L | 26.8 | 18.56 | 19.6 | 9.29 | 2.0 | 1.15 |
| N-demethyl-175-L | 1.8 | 1.36 | 3.2 | 1.53 | 0.6 | 0.37 |
| N-formyl-175-L | 2.0 | 1.50 | 1.3 | 0.58 | 0.7 | 0.38 |
| Total identified | 30.6 | 21.42 | 24.1 | 11.40 | 3.3 | 1.90 |
| Total characterised [b] | 69.3 | 42.93 | 73.8 | 35.08 | 95 | 55.17 |
| Total extracted | 99.9 | 64.35 | 97.9 | 46.48 | 98.3 | 57.07 |
| Total unextractable | 1.2 | 0.77 | 1.3 | 0.60 | 2.2 | 1.29 |
| Total measured (TRR in ( )) | 101 | (64.4) | 99 | (47.4) | 101 | (58.1) |
| | 7 DAT | | 14 DAT | | 30 DAT | |
| XDE-175-L | 1.3 | 0.59 | 0.7 | 0.24 | 0.2 | 0.12 |
| N-demethyl-175-L | 0.9 | 0.41 | 0.6 | 0.25 | 0.2 | 0.12 |
| N-formyl-175-L | 1.2 | 0.55 | 1 | 0.39 | 0.3 | 0.18 |
| Total identified | 3.4 | 1.55 | 2.3 | 0.89 | 0.7 | 0.42 |
| Total characterised [b] | 94.6 | 49.44 | 94.4 | 37.26 | 90.4 | 50.34 |
| Total extracted | 98.0 | 50.99 | 96.7 | 38.14 | 91.1 | 50.76 |
| Total unextractable | 2.7 | 1.38 | 2.4 | 0.94 | 6.8 | 3.77 |
| Total measured (TRR in ( )) | 101 | (52.1) | 99 | (39.4) | 98 | (55.4) |

[a] Sum of residues of pooled washings and extracts by HPLC.

[b] Characterised but not identified

*Lettuce*

Individual pots of red leaf lettuce (variety New Fire Red) were treated with either single or multiple foliar spray applications of $^{14}$C-XDE-175-J or $^{14}$C-XDE-175-L (Magnussen, *et. al*., 2005; Report 040048). For $^{14}$C-XDE-175-J, single applications were made at rates equivalent to 0.90 kg ai/ha (single application treatment) while the same amount of the test compound was sprayed in three separate applications with the equal rate at weekly intervals (multiple application treatment). For $^{14}$C-XDE-175-L, plants were treated in a similar fashion but at a rate equivalent to 0.30 kg ai/ha. For both compounds, the amounts applied approximately correspond to four times the maximum seasonal rate described on label.

Lettuce receiving a single application was harvested at 0, 0.25, 1, 3, and 7 DAT. Lettuce treated with multiple applications was harvested at 3 and 7 DAT. Identification of residues was not conducted for some of the 7 DAT samples as they were damaged by heat stress.

Collected samples were washed first with dichloromethane followed by acetonitrile in order to remove surface residues. Total radioactive residues (TRR) were determined as sum of radioactive residues in washings and washed lettuce. In both $^{14}$C-XDE-175-J and $^{14}$C-XDE-175-L treated lettuce samples, 76–96% of TRR were found in the solvent washings. From lettuce samples collected 0, 0.25 and 1 day after the last application, approximately 3.4–21% of TRR were found in acetonitrile washings. The radioactive residues remaining after washing in lettuce samples taken 3 DAT were extracted by homogenizing and refluxing with acetonitrile/water (75/25, v/v) followed by partitioning with acetonitrile/dichloromethane (50/50, v/v) at neutral and acidic pH. About 12–19% of TRR were in the organic phase and 0.3–3.4% of TRR in the aqueous phase. Overall, only 0.2–5.2% of TRR remained unextractable in all treated lettuce samples.

The solvent washings and extracts were pooled and analysed. Table 8 summaries the identified residues. Several minor metabolites assumed to be structurally similar to the parent

compound were also observed in the $^{14}$C-XDE-175-J and $^{14}$C-XDE-175-L treated lettuce at or less than 6% of TRR. A multi-component mixture of extensively degraded compounds represented up to 13–78% of TRR, each component at less than 3% of TRR.

Table 8 Characterization and identification of radioactive residues in lettuce following application of $^{14}$C-XDE-175-J at 0.90 kg ai/ha or $^{14}$C-XDE-175-L at 0.30 kg ai/ha

| Compound | 0 DAT | | 0.25 DAT | | 1 DAT | | 3 DAT | | 3 DAT (multiple)[a] | |
|---|---|---|---|---|---|---|---|---|---|---|
| | %TRR | mg/kg | %TRR | mg/kg | %TRR | mg/kg | %TRR | mg/kg | %TRR | mg/kg |
| Application of $^{14}$C-XDE-175-J at 0.90 kg ai/ha | | | | | | | | | | |
| XDE-175-J | 63.6 | 31.74 | 42.1 | 27.23 | 31.1 | 10.71 | 17.6 | 6.41 | 8.5 | 0.52 |
| N-demethyl-175-J | 8.9 | 4.44 | 18.0 | 11.64 | 19.6 | 6.75 | 15.5 | 5.64 | 7.2 | 0.44 |
| N-formyl-175-J | 6.6 | 3.29 | 7.8 | 5.05 | 10.6 | 3.65 | 11.2 | 4.08 | 14.8 | 0.90 |
| Total identified | 79.1 | 39.48 | 67.9 | 43.92 | 61.3 | 21.11 | 44.3 | 16.12 | 30.5 | 1.86 |
| Total characterised | 20.7 | 10.32 | 31.6 | 20.45 | 37.5 | 12.90 | 55.1 | 20.07 | 66.6 | 4.06 |
| Total extracted | 99.8 | 49.80 | 99.5 | 64.37 | 98.8 | 34.01 | 88.4 | 36.19 | 97.1 | 5.93 |
| Total unextractable | 0.2 | 0.10 | 0.5 | 0.32 | 1.2 | 0.41 | 0.6 | 0.22 | 3.0 | 0.18 |
| Total measured (TRR in ( )) | 100 | (49.9) | 100 | (64.7) | 100 | (34.4) | 100 | (36.3) | 100 | (6.11) |
| Application of $^{14}$C-XDE-175-L at 0.30 kg ai/ha | | | | | | | | | | |
| XDE-175-L | 62.4 | 6.18 | 8.2 | 1.47 | 11.9 | 0.91 | 5.1 | 0.55 | 2.8 | 0.10 |
| N-demethyl-175-L | 17.6 | 2.08 | 6.7 | 0.54 | 7.2 | 0.55 | 3.5 | 0.38 | 1.5 | 0.05 |
| N-formyl-175-L | 5.9 | 0.70 | 5.3 | 0.43 | 4.0 | 0.30 | 2.0 | 0.22 | 1.1 | 0.04 |
| Total identified | 75.9 | 8.95 | 30.2 | 2.45 | 23.1 | 1.76 | 10.6 | 1.14 | 5.4 | 0.18 |
| Total characterised | 22.5 | 2.66 | 65.8 | 5.33 | 71.7 | 8.97 | 87.7 | 9.46 | 91.2 | 3.08 |
| Total extracted | 98.4 | 11.61 | 96 | 7.77 | 94.8 | 7.21 | 98.3 | 10.60 | 96.6 | 3.28 |
| Total unextractable | 1.6 | 0.19 | 4.0 | 0.32 | 5.2 | 0.40 | 1.8 | 0.19 | 3.4 | 0.12 |
| Total measured (TRR in ( )) | 100 | (11.8) | 100 | (8.10) | 100 | (7.61) | 100 | (10.8) | 100 | (3.38) |

[a] Samples received multiple applications of $^{14}$C-XDE-175-J or $^{14}$C-XDE-175-L.

*Turnips*

Individual pots of Purple Top White Globe turnips were treated with either a single or multiple foliar applications of $^{14}$C-XDE-175-J or $^{14}$C-XDE-175-L (Graper, *et. al*., 2005; Report 040049). Single applications of XDE-175-J were made at rates equivalent to 0.90 kg ai/ha (single application treatment) while the same amount of the test compound was sprayed in three separate applications with the equal rate at weekly intervals (multiple application treatment). For XDE-175-L, plants were treated in a similar fashion but at a rate equivalent to 0.30 kg ai/ha. For both test compounds, applied approximately correspond to four times the maximum seasonal rate described on label.

Following application, plants treated with a single application were harvested at 0, 0.25, 1, 3 and 7 DAT. Plants that received the multiple applications were harvested at 3 and 7 DAT of the final application (17 and 21 days after the initial application).

Collected turnip tops were washed first with dichloromethane followed by acetonitrile in order to remove surface residues. Total radioactive residues (TRR) were determined as the sum of the residue in the washing and washed tissue. In $^{14}$C-XDE-175-J treated turnip tops, 59–91% of TRR were surface residues found in the solvent washings. In $^{14}$C-XDE-175-L treated turnip tops, surface residues were 39–80% of TRR, slightly smaller percentage than in XDE-175-J treated tops. In treatment with both compounds, the percentage of TRR as surface residues of turnip tops was lower in samples receiving multiple applications than for those receiving a single application.

Washed turnip tops were extracted by homogenizing and refluxing with acetonitrile/water (80/20, v/v) followed by partitioning with dichloromethane at neutral and acidic pH. Approximately 8–35% of TRR were found in organic phase and < 0.1–9% of TRR were in the aqueous phase, but < 1–18% of TRR were unextractable. The remaining residues in 3 DAT turnip top samples receiving multiple applications were extracted with acid and 2–3% of TRR were released as organo-soluble residues and 1–4% of TRR as aqueous residues, leaving 4–5% of TRR still not extracted.

Turnip root samples were extracted by homogenizing and refluxing with acetonitrile/water (80/20, v/v) followed by dichloromethane partitioning at neutral and acidic pH. In turnip root samples from both $^{14}$C-XDE-175-J and $^{14}$C-XDE-175-L treatments, 38–93% of TRR were in organic phase and 2–28% of TRR were in the aqueous phase, but 6–34% of TRR were unextractable.

For metabolite characterization and identification, the washings and residues in organic solvent and aqueous extracts of turnip tops or turnip roots were pooled as composite extracts. For tops, samples harvested at 0.25 DAT and 3 DAT after a single application and those at 3 DAT after multiple applications were used. For roots, samples taken at 3 DAT after a single application were used. Table 9 summarizes residues characterised and identified in turnip tops and roots. Several minor metabolites assumed to be structurally similar to the parent compound were also observed in treated turnip tops, each less than 4% of TRR. A multi-component mixture of extensively degraded compounds represented 10–74% of TRR, each compound at less than 1% TRR.

Table 9 Characterization and identification of radioactive residues in turnip tops and roots following application of $^{14}$C-XDE-175-J at 0.90 kg ai/ha or $^{14}$C-XDE-175-L at 0.30 kg ai/ha

| Compound | TOPS | | | | | | ROOTS | |
|---|---|---|---|---|---|---|---|---|
| | 0.25 DAT | | 3 DAT | | 3 DAT (multiple)[a] | | 3 DAT | |
| | % TRR | mg/kg | % TRR | mg/kg | % TRR | mg/kg | % TRR | mg/kg |
| Application of $^{14}$C-XDE-175-J at 0.90 kg ai/ha | | | | | | | | |
| XDE-175-J | 35.7 | 4.20 | 9.4 | 1.11 | 4.9 | 0.35 | 22.3 | 0.03 |
| N-demethyl-175-J | 14.4 | 1.69 | 8.5 | 1.01 | 4.1 | 0.30 | 10.0 | 0.01 |
| N-formyl-175-J | 10.4 | 1.23 | 11.2 | 1.32 | 11.4 | 0.82 | 16.6 | 0.02 |
| C17-pseudoaglycone-175-J | - | - | - | - | 0.3 | 0.02 | - | - |
| Aglycone-175-J | - | - | - | - | 0.5 | 0.03 | - | - |
| Total identified | 60.5 | 7.12 | 29.1 | 3.43 | 21.2 | 1.53 | 48.9 | 0.06 |
| Total characterised | 38.9 | 4.58 | 68.7 | 8.08 | 74.8 | 5.41 | 40.8 | 0.05 |
| Total extracted | 99.4 | 11.71 | 97.8 | 11.51 | 96.0 | 6.93 | 89.0 | 0.11 |
| Total unextractable | 0.6 | 0.08 | 2.2 | 0.26 | 4.0 | 0.29 | 10.3 | 0.01 |
| Total measured (TRR in ( )) | 100 | (11.8) | 100 | (11.8) | 100 | (7.22) | 100 | (0.12) |
| Application of $^{14}$C-XDE-175-L at 0.30 kg ai/ha | | | | | | | | |
| XDE-175-L | 17.1 | 0.91 | 2.9 | 0.06 | 3.0 | 0.07 | 14.8 | 0.005 |
| N-demethyl-175-L | 7.4 | 0.40 | 0.6 | 0.01 | 1.1 | 0.02 | - | - |
| N-formyl-175-L | 3.0 | 0.16 | 1.0 | 0.2 | 0.5 | 0.01 | 3.0 | 0.001 |
| Total identified | 27.5 | 1.47 | 4.5 | 0.10 | 4.6 | 0.10 | 17.8 | 0.006 |
| Total characterised | 69.8 | 3.73 | 88.8 | 1.89 | 89.2 | 1.92 | 62.4 | 0.019 |
| Total extracted | 97.3 | 5.20 | 93.3 | 1.99 | 93.8 | 2.03 | 80.2 | 0.025 |
| Total unextractable | 2.7 | 0.14 | 6.6 | 0.14 | 6.2 | 0.13 | 19.8 | 0.006 |
| Total measured (TRR in ( )) | 100 | (5.34) | 100 | (2.13) | 100 | (2.16) | 100 | (0.031) |

[a] Samples received multiple applications of $^{14}$C-XDE-175-J or $^{14}$C-XDE-175-L.

A consistent metabolism of spinetoram was observed in the three crops studied–apple, lettuce and turnip–indicating that a common metabolism is expected for not only fruits, leafy vegetables and root vegetables but also other plants.

Based on the results of the submitted metabolism studies, it appears that three metabolic pathways are responsible for the breakdown of spinetoram in plants. The first one involves changes to the N-dimethyl moiety on the forosamine sugar to give the N-demethyl and N-formyl metabolites. Due to the presence of these metabolites at the 0-day PHI, it is thought that their formation may be the result of photolysis. The second involves cleavage of the macrolide ring system at one or more positions, ultimately resulting in a complex residue mixture consisting of numerous components. The third, applicable only to XDE-175-J, involves changes to the rhamnose sugar producing the 3-O-deethyl and C9-pseudoaglycone-175-J metabolites. All the metabolites occurring as a result of changes in forosamine and rhamnose are further degraded via the second pathway. XDE-175-L might also undergo degradation through the third pathway no relevant metabolites were detected perhaps because degradation through the second pathway might be too fast. The proposed metabolic pathways for spinetoram in plants are shown in Figures 3 and 4 for XDE-175-J and XDE-175-L, respectively.

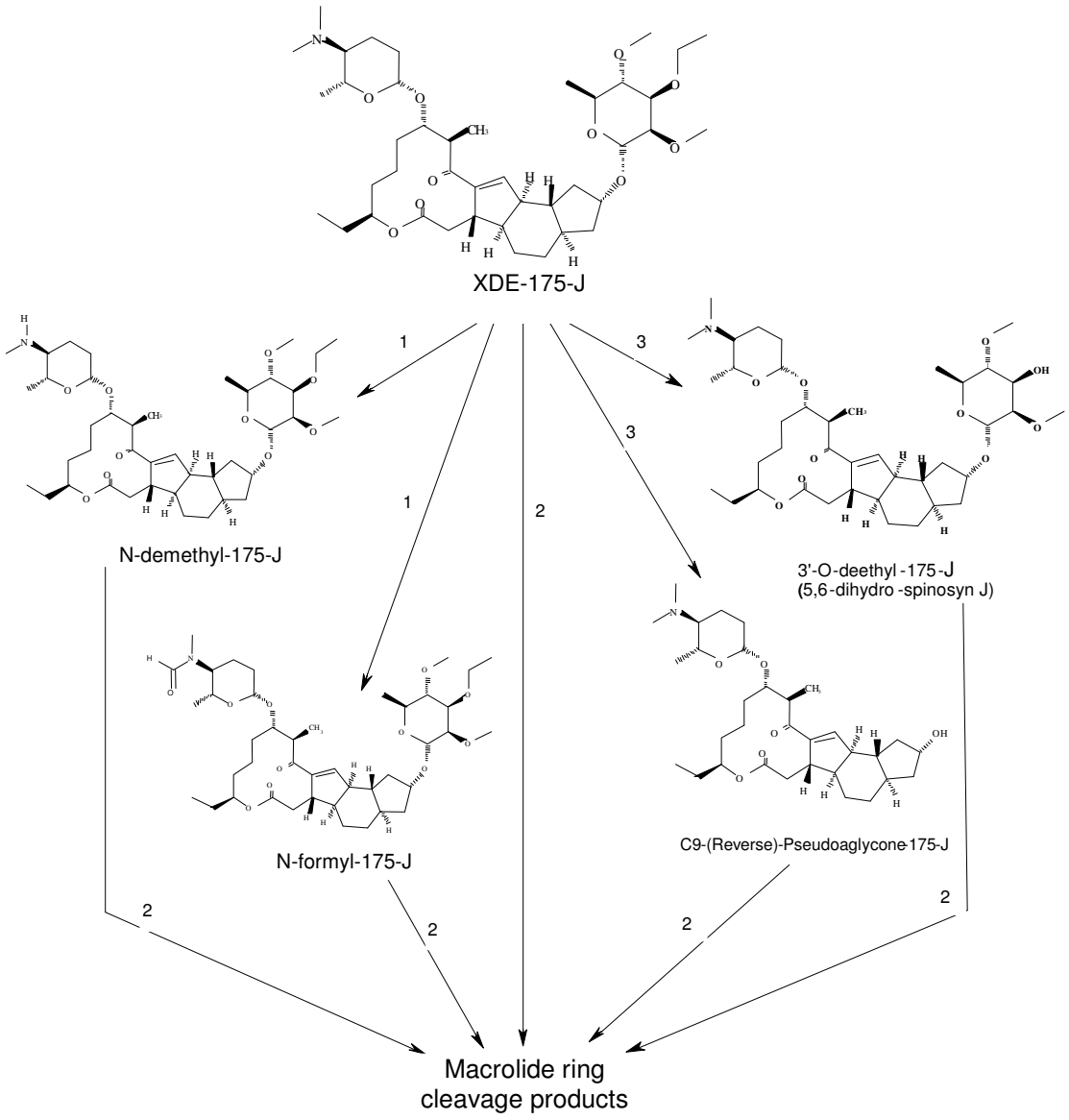



Figure 3 Proposed Metabolic Pathway of XDE-175-J in Plants

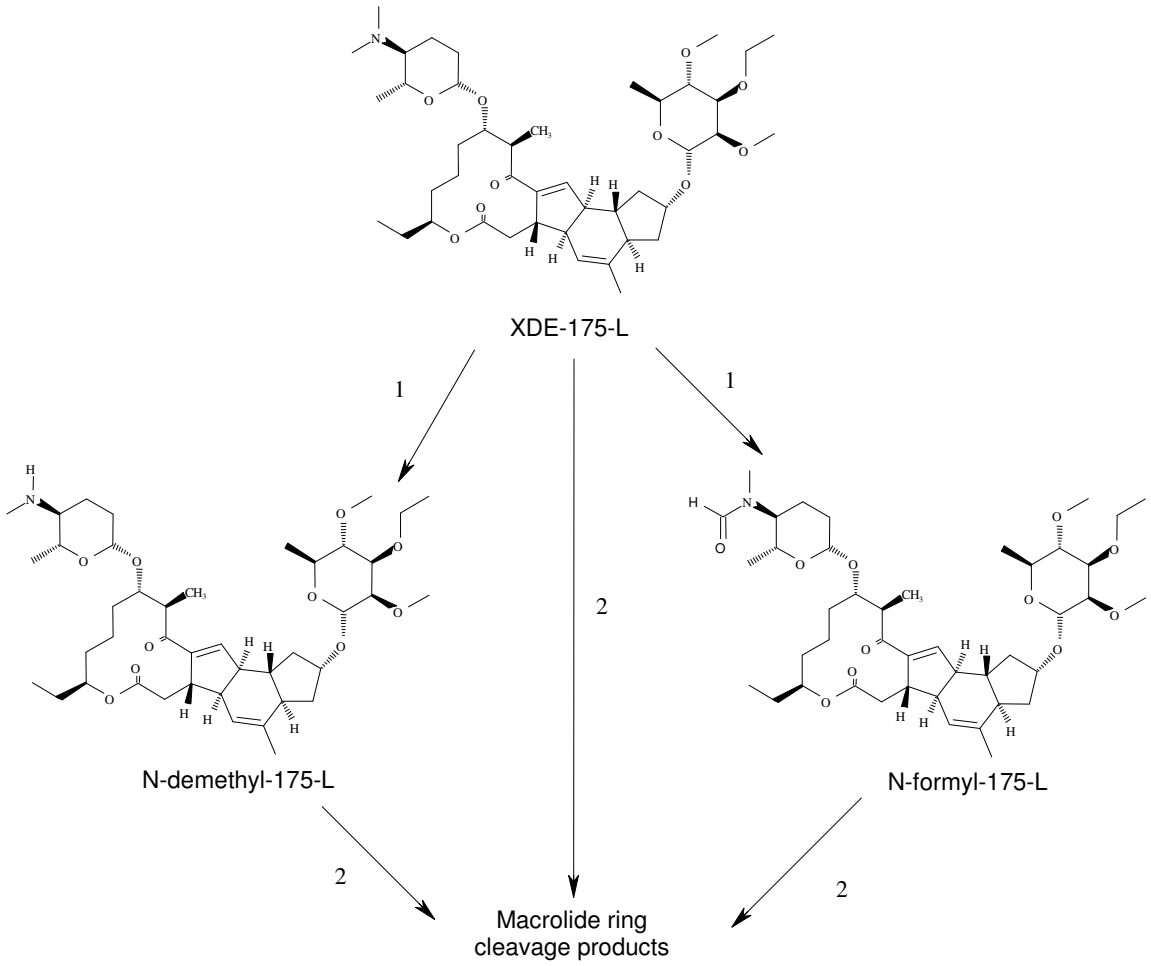



Figure 4 Proposed Metabolic Pathway of XDE-175-L in Plants

***Environmental fate in soil***

The Meeting received information on the aerobic soil metabolism, aqueous photolysis and hydrolysis, and residues in succeeding crops. Since spinetoram is intended for use on root vegetables and supervised trials were conducted on sugar beet, the above mentioned information was reviewed. The Meeting also received information on anaerobic soil metabolism and photolysis in soil but they were not reviewed.

In laboratory studies on aerobic soil metabolism, the same $^{14}$C-labelled and $^{14}$C- and D-labelled active substances were used as in the animal and plant metabolism studies. Unless otherwise indicated, the radiochemical purity was > 97%.

*Aerobic soil metabolism*

Yoder *et al*. (2005; Report 040068) incubated radio-labelled spinetoram for up to one year under aerobic condition in the dark at 25 °C and 75% of 1/3 bar moisture in four soils in the USA: Commerce loam from Mississippi; Fayette silt loam from Iowa; Kimberlina/Nord sandy loam from California; and Slagle loam from Virginia. XDE-175-J was applied at the rate of 0.21 mg ai/kg soil, equivalent to 375 g ai/ha (1×). XDE-175-L was also applied at 0.21 mg ai/kg soil, or approximately equivalent to 3× its anticipated maximum use rate.

The degradation of spinetoram in European soils under aerobic conditions in the dark was also studied (Smith-Drake, *et. al*., 2007; Report 050076.1). Radio-labelled spinetoram was incubated at 20 °C for 127 days in four soils: a sandy loam from Longwoods Quarry, Lincolnshire, UK; a loamy sand from Hanhofen, Rheinlan-Pfalz, Germany; a sandy clay loam from Little Shelford, UK; and a sandy clay loam from Altlussheim, Baden-Wurttemberg, Germany. XDE-175-J was applied at the rate of 0.8 mg ai/kg soil, equivalent to 300 g ai/ha. XDE-175-L was applied at the same rate equivalent to 3× the label rate. Radio-labelled spinetoram was also incubated at 10 °C in the dark for 127 days in the sandy loam from Longwoods Quarry.

The results of these studies are summarized in Table 9.

Under aerobic conditions, spinetoram applied to soil was degraded relatively rapidly. In all soils tested, XDE-175-L was degraded faster than XDE-175-J. After one year of incubation, 1.2–2.8% and 0.3–2.9% of applied XDE-175-J and XDE-175-L respectively (dose rate, 0.21 mg/kg soil; 25 °C) remained as the parent in US soils tested. In European soils except the loamy sand, after 127 days of incubation, 2.0–4.9% and 1.4–5.0% of applied XDE-175-J and XDE-175-L respectively (dose rate, 0.80 mg/kg soil; 20 °C) remained as the parent. In the loamy sand, 50% and 33% of the applied XDE-175-J and XDE-175-L respectively (dose rate, 0.80 mg/kg soil; 20 °C) remained as the parent). In the sandy loam maintained at 10 °C, 4.9% and 2.1% of the applied XDE-175-J and XDE-175-L respectively (same dose rate as at 20 °C) remained as the parent.

Carbon dioxide was evolved slowly from all soils and accounted for 5.0–35% and 9.5–32% of the applied XDE-175-J and XDE-175-L respectively after one year at 25 °C, and 0.8–1.1% and 1.2–3.2% of the applied XDE-175-J and XDE-175-L respectively after 127 days at 20 °C.

As major degradation products, N-demethyl 175-J and N-demethyl-175-L were formed and then degraded during the study periods. As minor products (at or less than 10% of the applied dose), N-demethyl-N-nitroso-175-J, N-demethyl-N-nitroso-175-L, N-succinyl-175-L and N-succinyl-175-L were also formed and degraded. Many other degradates were formed but at very low concentrations.

While extractable radioactivity decreased, non-extractable radioactivity steadily increased to reach 22–29% and 32–37% of the applied XDE-175-J and XDE-175-L respectively after one year at 25 °C; and 5–15% and 11–24% of the applied XDE-175-J and XDE-175-L respectively after 127 days at 20 and 10 °C.

The half-life of spinetoram was calculated to be 21 days and 13 days for XDE-175-J and XDE-175-L respectively at 0.21 mg/kg dose rate (25 °C) and 20 days and 14 days for XDE-175-J and XDE-175-L respectively at 0.80 mg/kg dose rate (20 °C). When maintained at 10 °C the half-life was 21 days and 16 days for XDE-175-J and XDE-175-L respectively at 0.80 mg/kg dose rate. In the sterile sandy clay loam from Little Shelford, the half-life of XDE-175-J and XDE-175-L was much longer at 207 and 298 days respectively than in the respective unsterilized soil. The half-life of XDE-175-J and XDE-175-L under anaerobic condition in sandy loam from Longwoods Quarry was 203 days and 122 days respectively, much longer than the half-life under aerobic condition.

The half-life of N-demethyl-175-J and -L ranged 32–273 days and 5–88 days respectively at 25 °C and 136–301 days and 59–204 days at 20 °C.

Table 9 Results of aerobic soil metabolism studies [(J): XDE-175-J (L): XDE-175-L]

| | |
|---|---|
| Soil: Loam (Commerce) | Ref: Yoder *et al*., 2005; Report 040068 |
| Dose rate: 0.21 mg ai/kg soil (both compounds) | $^{14}$C accountability: 97–108% (J) |
| Duration: 276 days (J) and 365 days (L) | 90–105% (L) |
| Temperature: 25 ± 2 °C | Mineralization at the end: 7.5% (J) |
| pH: 7.5 | 18% (L) |
| Moisture: 75% of 1/3 bar moisture | Unextractable at the end: 22% (J) |
| Organic carbon: 0.6% | 33% (L) |
| Average half-life: 23 days (J) | Average half-life of N-demethyl metabolite: |
| 17 days (L) | 253 days (J) |
| | 88 days (L) |

Spinetoram remaining at the end: 1.2% (J)
0.7% (L)

| Metabolite | Max % of dose | Day |
|---|---|---|
| XDE-175-J | 102 | 0 |
| N-demethyl-175-J | 68 | 98, 125 |
| XDE-175-L | 95 | 0 |
| N-demethyl-175-L | 41 | 32 |

N-demethyl metabolite at the end: 45% (J)
9.1% (L)

Ref: Yoder *et al*., 2005; Report 040068

Soil: Silt loam (Fayette)
Dose rate: 0.21 mg ai/kg soil (both compounds)
Duration: 366 days (J) and 365 days (L)
Temperature: 25 ± 2 °C
pH: 7.4
Moisture: 75% of 1/3 bar moisture
Organic carbon: 1.1%
Average half-life: 29 days (J)
15 days (L)
Spinetoram remaining at the end: 1.7%(J)
0.3% (L)

| Metabolite | Max % of dose | Day |
|---|---|---|
| XDE-175-J | 95 | 0 |
| N-demethyl-175-J | 62 | 63 |
| XDE-175-L | 88 | 0 |
| N-demethyl-175-L | 29 | 7, 32 |

$^{14}$C accountability: 92–109% (J)
92–104% (L)
Mineralization at the end: 6.9% (J)
17% (L)
Unextractable at the end: 23% (J)
37% (L)
Average half-life of N-demethyl metabolite:
273 days (J)
18 days (L)
N-demethyl metabolite at the end: 41% (J)
0.8% (L)

Ref: Yoder *et al*., 2005; Report 040068

Soil: Sandy loam (Kimberlina/Nord)
Dose rate: 0.21 mg ai/kg soil (both compounds)
Duration: 366 days (J) and 365 days (L)
Temperature: 25 ± 2 °C
pH: 8.1
Moisture: 75% of 1/3 bar moisture
Organic carbon: 0.7%
Average half-life: 23 days (J)
17 days (L)
Spinetoram remaining at the end: 2.8%(J)
2.0% (L)

| Metabolite | Max % of dose | Day |
|---|---|---|
| XDE-175-J | 98 | 0 |
| N-demethyl-175-J | 51 | 92 |
| XDE-175-L | 97 | 0 |
| N-demethyl-175-L | 33 | 32 |

$^{14}$C accountability: 97–109% (J)
98-107% (L)
Mineralization at the end: 5.0% (J)
9.5% (L)
Unextractable at the end: 25% (J)
35% (L)
Average half-life of N-demethyl metabolite:
156 days (J)
29 days (L)
N-demethyl metabolite at the end: 23% (J)
6.9% (L)

Ref: Yoder *et al*., 2005; Report 040068

Soil: Loam (Slagle)
Dose rate: 0.21 mg ai/kg soil (both compounds)
Duration: 366 days (J) and 365 days (L)
Temperature: 25 ± 2 °C
pH: 5.8
Moisture: 75% of 1/3 bar moisture
Organic carbon: 0.5%
Average half-life: 8 days (J)
3 days (L)
Spinetoram remaining at the end: 2.0%(J)
2.9% (L)

$^{14}$C accountability: 90–104% (J)
8 8–104% (L)
Mineralization at the end: 35% (J)
32% (L)
Unextractable at the end: 29% (J)
32% (L)
Average half-life of N-demethyl metabolite:
32 days (J)
5 days (L)
N-demethyl metabolite at the end: 6.3% (J)
0.6% (L)

| Metabolite | Max % of dose | Day |
|---|---|---|
| XDE-175-J | 93 | 0 |
| N-demethyl-175-J | 45 | 14 |
| XDE-175-L | 89 | 0 |
| N-demethyl-175-L | 35 | 123 |

Ref: Smith-Drake, *et. al*., 2007; Report 050076.1

Soil: Sandy loam (Longwoods Quarry)
Dose rate: 0.80 mg ai/kg soil (both compounds)
Duration: 127 days
Temperature: 20 ± 2 °C
pH: 7.9
Moisture: 10.8 g/g-soil at 1/3 bar
Organic carbon: 0.8%
Average half-life: 8 days (J)
7 days (L)
Spinetoram remaining at the end: 2.0% (J)
1.4% (L)

| Metabolite | Max % of dose | Day |
|---|---|---|
| XDE-175-J | 93 | 0 |
| N-demethyl-175-J | 46 | 29 |
| N-demethyl-N-nitroso-175-J | 19 | 85 |
| XDE-175-L | 93 | 0 |
| N-demethyl-175-L | 38 | 21 |
| N-demethyl-N-nitroso-175-L | 13 | 42 |
| N-succinyl-175-L | 13 | 42 |

$^{14}$C accountability: 95–103% (J)
94–100% (L)
Mineralization at the end: 0.9% (J)
3.2% (L)
Unextractable at the end: 13% (J)
24% (L)
Average half-life of N-demethyl metabolite:
136 days (J)
59 days (L)
N-demethyl metabolite at the end: 36%(J)
15% (L)
In anaerobic condition, spinetoram remaining at the end (125 days): 4.2%(J)
7.1% (L)
Average half-life: 203 days (J)
122 days (L)

Ref: Smith-Drake, *et. al*., 2007; Report 050076.1

Soil: Sandy loam (Longwoods Quarry)
Dose rate: 0.80 mg ai/kg soil (both compounds)
Duration: 127 days
Temperature: 10 ± 2 °C
pH: 7.9
Moisture: 10.8 g/g-soil at 1/3 bar
Organic carbon: 0.8%
Average half-life: 21 days (J)
16 days (L)
Spinetoram remaining at the end: 4.9% (J)
2.1% (L)

| Metabolite | Max % of dose | Day |
|---|---|---|
| XDE-175-J | 93 [a] | 0 |
| N-demethyl-175-J | 45 | 43, 85 |
| N-demethyl-N-nitroso-175-J | 19 | 71, 99 |
| XDE-175-L | 93 [1] | 0 |
| N-demethyl-175-L | 31 | 42 |
| N-demethyl-N-nitroso-175-L | 16 | 56 |

[a] Data from 20 °C experiment.

$^{14}$C accountability: 95–103% (J)
94–100% (L)
Mineralization at the end: 0.9% (J)
1.4% (L)
Unextractable at the end: 13% (J)
13% (L)
Average half-life of N-demethyl metabolite:
386 days (J)
110 days (L)
N-demethyl metabolite at the end: 36% (J)
27%(L)

Ref: Smith-Drake, *et. al*., 2007; Report 050076.1

Soil: Loamy sand (Hanhofen)
Dose rate: 0.80 mg ai/kg soil (both compounds)
Duration: 127 days
Temperature: 20 ± 2 °C
pH: 6.0
Moisture: 9.7 g/g-soil at 1/3 bar

$^{14}$C accountability: 95–102% (J)
94–101% (L)
Mineralization at the end: 0.4% (J)
1.2% (L)
Unextractable at the end: 4.7% (J)
11% (L)

Organic carbon: 1.8%
Average half-life: 129 days (J)
71 days (L)
Spinetoram remaining at the end: 50% (J)
33% (L)

| Metabolite | Max % of dose | Day |
|---|---|---|
| XDE-175-J | 88 | 0 |
| N-demethyl-175-J | 30 | 113 |
| N-demethyl-N-nitroso-175-J | 1.6 | 113 |
| XDE-175-L | 92 | 0 |
| N-demethyl-175-L | 20 | 84 |
| N-demethyl-N-nitroso-175-L | 3.7 | 7 |

Average half-life of N-demethyl metabolite:
180 days (J)
72 days (L)
N-demethyl metabolite at the end: 30% (J)
17% (L)

Ref: Smith-Drake, *et. al*., 2007; Report 050076.1

Soil: Sandy clay loam (Little Shelford)
Dose rate: 0.80 mg ai/kg soil (both compounds)
Duration: 127 days
Temperature: 20 ± 2°C
pH: 7.8
Moisture: 16.9 g/g-soil at 1/3 bar
Organic carbon: 1.2%
Average half-life: 11 days (J)
8 days (L)
Spinetoram remaining at the end: 3.2% (J)
2.1% (L)

| Metabolite | Max % of dose | Day |
|---|---|---|
| XDE-175-J | 91 | 0 |
| N-demethyl-175-J | 57 | 99 |
| N-demethyl-N-nitroso-175-J | 5.4 | 85 |
| XDE-175-L | 93 | 0 |
| N-demethyl-175-L | 37 | 14 |
| N-demethyl-N-nitroso-175-L | 2.8 | 28 |

$^{14}$C accountability: 96–106% (J)
91–101% (L)
Mineralization at the end: 0.8% (J)
2.9% (L)
Unextractable at the end: 15% (J)
24% (L)
Average half-life of N-demethyl metabolite:
292 days (J)
112 days (L)
N-demethyl metabolite at the end: 55% (J)
22% (L)
In the sterile soil, spinetoram remaining at the end (120 days): 54% (J)
74% (L)
Average half-life: 207 days (J)
298 days (L)

Ref: Smith-Drake, *et. al*., 2007; Report 050076.1

Soil: Sandy clay loam (Altlussheim)
Dose rate: 0.80 mg ai/kg soil (both compounds)
Duration: 127 days
Temperature: 20 ± 2 °C
pH: 7.8
Moisture: 20.4 g/g-soil at 1/3 bar
Organic carbon: 1.3%
Average half-life: 15 days (J)
12 days (L)
Spinetoram remaining at the end: 4.9% (J)
5.0% (L)

| Metabolite | Max % of dose | Day |
|---|---|---|
| XDE-175-J | 87 | 0 |
| N-demethyl-175-J | 59 | 85 |
| N-demethyl-N-nitroso-175-J | 10 | 57 |
| XDE-175-L | 91 | 0 |
| N-demethyl-175-L | 42 | 84, 98 |
| N-demethyl-N-nitroso-175-L | 5.6 | 70 |

$^{14}$C accountability: 95–103% (J)
95–100% (L)
Mineralization at the end: 1.1% (J)
2.5% (L)
Unextractable at the end: 12% (J)
17% (L)
Average half-life of N-demethyl metabolite:
301 days (J)
204 days (L)
N-demethyl metabolites at the end: 58% (J)
39% (L)

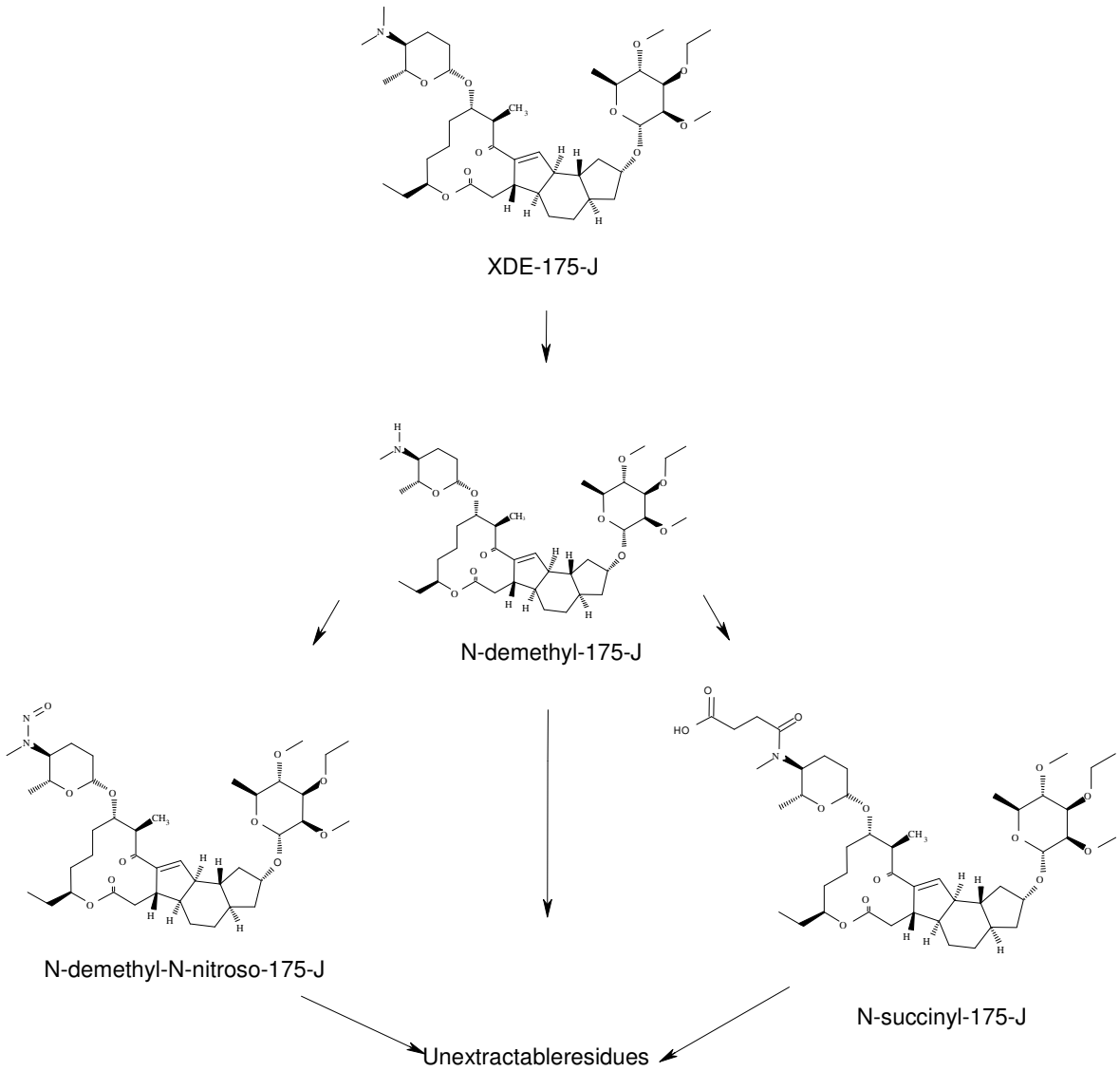



Figure 5 Proposed Degradation Pathway of XDE-175-J in Soil under Aerobic Conditions

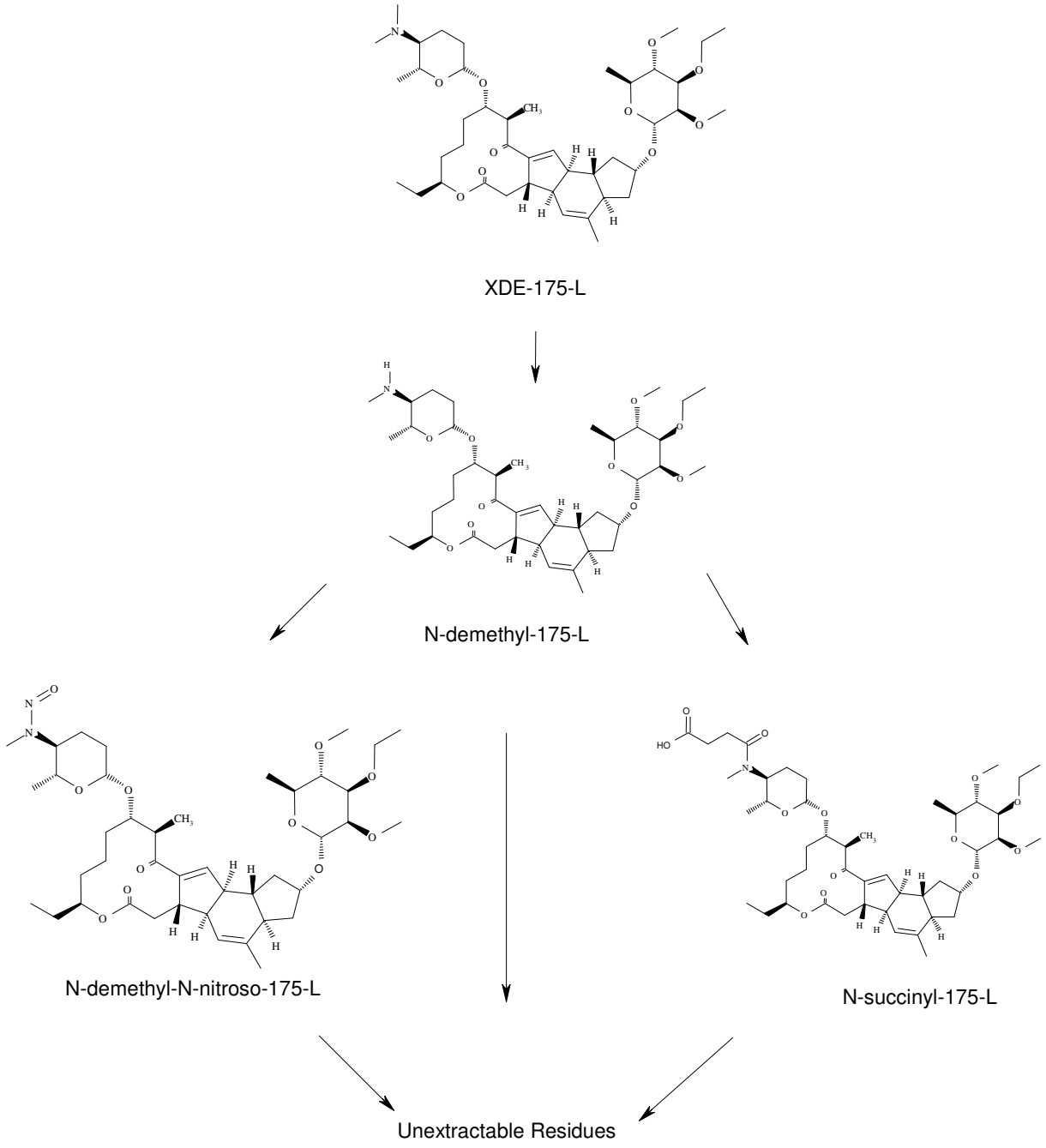



Figure 6 Proposed Degradation Pathway of XDE-175-L in Soil under Aerobic Conditions

*Aqueous photolysis*

The aqueous photolysis of radiolabelled spinetoram was studied at 25 °C in sterile aqueous tris buffer solution at pH 7 (Yoder, *et al*., 2005, Report 040079). XDE-175-J was added at approximately 0.3 mg ai/L and XDE-175-L at 0.5 mg ai/L. Acetonitrile (0.5%) was used as a co-solvent. Samples were continuously irradiated using a xenon lamp as a light source. No trapping of $CO_2$ or organic volatiles took place.

Samples were analysed after 4, 8 and 16 h, and 1, 2, 4, 7, 13 and 19 days of continuous irradiation; 24 h of continuous exposure (1 DAT) was equivalent to 1.8 days of summer sunlight at

40° N latitude. Time 0 samples and dark controls were also analysed. Aliquots of each sample were directly analysed by LSC and HPLC. Characterization of transformation products was done by LC/MS.

A PNAP/pyridine chemical actinometer solution was used to quantify the amount of light that the sample solutions received. Based on actinometer data, the 19 experimental days of continuous irradiation was equivalent to 34 days of irradiation in the summer sun at 40° N latitude.

Material balance was 101.1 ± 3.7% of applied radioactivity for the irradiated XDE-175-J solutions and 95.8 ± 3.4% for the dark XDE-175-J controls. Irradiated and dark XDE-175-L solutions had a material balance of 94.2 ± 2.0% and 96.7 ± 4.0%, respectively. At test termination, greater than 90% of the applied amount remained as parent in the dark controls indicating that negligible transformation of the parent compounds occurred in the dark. No degradates were observed in the dark controls.

In the irradiated solutions, the concentration of XDE-175-J decreased from 98% at 0 DAT to 0% (95% to 0% for XDE-175-L) of the applied amount at study termination. The major transformation product detected in the irradiated XDE-175-J solutions had a molecular weight of 813 g/mol and a chemical formula of $C_{42}H_{71}NO_{14}$. This degradate reached a maximum concentration of 11% of the applied amount at 7 DAT before declining to approximately 1% at study termination. The major product formed from XDE-175-L photodegradation was N-demethyl-L, which reached a maximum concentration of 13% of the applied radioactivity after 4 h of irradiation and declined to less that 1% after 2 DAT. Numerous minor degradate peaks were observed that were not identified, but all were less than 10% of the applied radioactivity from either parent compounds. No loss of radioactivity due to volatiles was observed.

Photodegradation of XDE-175-J resulted in a complex pathway containing numerous degradates, of which only one (Molecular Weight 813) was observed at concentrations greater than 10% of applied. Photoproducts with additional oxygen atoms or hydroxyl groups were observed. As many as 80 peaks, each accounting for less than 5% of the applied radioactivity, were observed at study termination. Photodegradates became increasingly polar over time.

As a major degradate, N-demethyl-175-L was observed in solutions dosed with XDE-175-L. The N-demethyl-175-L degradate and parent XDE-175-L degraded rapidly to numerous increasingly polar compounds. As many as 75 minor peaks (less than 5% of the applied radioactivity) were detected at 19 DAT. The polar degradates formed from the photolysis of both XDE-175-J and XDE-175-L were presumed to be products created by cleavage of the macrolide ring.

*Aqueous hydrolysis*

Hydrolysis of radiolabelled XDE-175-J and XDE-175-L at 0.5 mg ai/L was studied in the dark at 25 °C in sterile aqueous buffered solutions at pH 5 (acetate), pH 7 (THAM), and pH 9 (borate) for 30 days (Rutherford*, et al*., 2005, Report 040108). Samples were analysed at 0, 2, 7, 14, 21, and 30 days by diluting the samples with a mixture of acetonitrile and methanol to adjust the concentration of acetonitrile at 20%.

For XDE-175-J-containing solutions, material balance was 98.4 ± 1.0%, 97.6 ± 1.4% and 97.0 ± 1.4% of the applied radioactivity at pH 5, pH 7 and pH 9, respectively. The concentration of parent compound remained constant at 96.2 ± 1.6%, 94.5 ± 2.1%, and 91.3 ± 2.5% of applied radioactivity at pH 5, pH 7, and pH 9, respectively. At pH 9, a minor transformation product, N-demethyl-175-J, with a maximum average concentration of 6.7% of applied radioactivity, was observed on the $30^{th}$ day of incubation. Volatiles were not trapped. The average unidentified radioactivity was 2.7, 3.3 and 1.6% of the applied amount at pH 5, pH 7 and pH 9, respectively, at study termination.

XDE-175-J was stable to hydrolysis at pH 5 and pH 7. Although a degradate product formed at pH 9, the averaged parent compound concentration did not decrease below 89% of the applied radiocarbon.

For XDE-175-L-containing solutions, material balance was 94.0 ± 1.2%, 96.9 ± 0.8% and 97.0 ± 1.3% of the applied radioactivity at pH 5, pH 7, and pH 9, respectively. The concentration of parent compound remained constant at 90.4 ± 2.4% and 92.6 ± 2.1% of applied radiocarbon at pH 5 and pH 7, respectively. At test termination, the average concentration of the parent compound had decreased from 92.1 to 81.6% of the initial applied radioactivity at pH 9. At pH 9 the major transformation product was N-demethyl-175-L, with a maximum average concentration of 11.9% of applied radioactivity, observed on the 30th day of incubation. No minor transformation products were detected. Volatiles were not trapped. The average unidentified radioactivity was 4.8, 5.1 and 3.1% of the applied amount at pH 5, pH 7 and pH 9, respectively, at study termination.

XDE-175-L was stable to hydrolysis at pH 5 and pH 7. Using simple first-order kinetics, the degradation rate constant of XDE-175-L at pH 9 was 0.0045 $days^{-1}$, which corresponds to a half-life of 154 days and a $DT_{90}$ of 512 days.

In summary, no hydrolysis of XDE-175-J or XDE-175-L occurred at pH 5 or pH 7. At pH 9, N-demethyl-175-J and N-demethyl-175-L were observed as minor transformation products on the 30th day of incubation. No other minor transformation products were detected.

***Residues in Succeeding Crops***

In an rotational crop study conducted in the USA in 2008 (Graper and Smith, 2008; Report 040086.01), applications of $^{14}$C-XDE-175-J or $^{14}$C-XDE-175-L were made to confined, outdoor plots of sandy loam soil at rates of 405 and 135 g ai/ha, respectively, which were equivalent to the seasonal maximum use rate (based on 3 to 1 ratio in the final formulation). The test plots were aged for 30, 120 and 365 days after treatment (DAT), and radishes, lettuce, and wheat were planted into the plots at each interval (plant-back). Immature and mature radishes, immature and mature lettuce, wheat forage, wheat hay, and mature wheat grain and straw were harvested from the plots.

Total radioactive residues (TRR) in the raw agricultural commodities (RAC), expressed as mg of XDE-175 equivalents per kg plant tissue (mg/kg) are summarized in Table 10.

Table 10 Total radioactive residues determined in rotational crops planted 30, 120, and 365 days after application of radiolabelled spinetoram to soil

| | Total radioactive residues (mg-XDE-175 equivalents/kg) | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | RADISH | | | LETTUCE | | WHEAT | | | |
| | Immature tops | Mature tops | Mature roots | Immature | Mature | Forage | Hay | Straw | Grain |
| 30 DAT [a] | (68) | (89) | (89) | (82) | (89) | (105) | (140) | (177) | (177) |
| Control [b] | < LOQ | < LOD | < LOD | < LOD | < LOQ | < LOD | < LOD | < LOD | < LOD |
| XDE-175-J | 0.079 | 0.014 | 0.005 | 0.085 | 0.027 | 0.004 | 0.013 [c] | 0.022 | < LOQ |
| XDE-175-L | 0.021 | < LOQ | < LOD | 0.033 | 0.016 | < LOQ | < LOQ | 0.015 | < LOQ |
| 120 DAT [a] | (56) | (69) | (69) | (56) | (69) | (77) | (93) | (128) | (128) |
| XDE-175-J | 0.013 | 0.006 | < LOQ | 0.012 | < LOQ | 0.011 | 0.014 | 0.023 | < LOQ |
| XDE-175-L | < LOQ | < LOQ | < LOQ | < LOQ | < LOQ | < LOQ | 0.008 [d] | 0.020 | < LOQ |
| 365 DAT [a] | (31) | (58) | (58) | (31) | (58) | (31) | (129) | (205) | (205) |
| XDE-175-J | 0.018 | 0.016 | 0.006 | 0.013 | 0.005 | 0.013 | 0.031 | 0.045 | 0.008 |
| XDE-175-L | 0.006 | < LOQ | < LOQ | 0.016 | < LOQ | < LOQ | 0.021 | 0.029 | 0.006 |

[a] Days between planting and harvest.

[b] Limits of detection (LOD): 0.001 and 0.002 mg/kg for XDE-175-J and XDE-175-L, respectively.
Limits of quantification (LOQ): 0.004 and 0.006 mg/kg for XDE-175-J and XDE-175-L, respectively.

[c] Measured TRR value of 0.009 mg/kg (40% moisture) adjusted to 0.013 mg/kg (15% moisture).

[d] Measured TRR value of 0.007 mg/kg (23.5% moisture) adjusted to 0.008 mg/kg (15% moisture).

No radioactive residues greater than 0.085 mg/kg, in spinetoram-equivalents, were found in any of the crops evaluated. Crop samples that contained TRR of 0.010 mg/kg or higher were subjected to solvent extraction, and unextractable residues in tissue were less than 0.019 mg/kg. The extractable radioactivity was characterised further by solvent partitioning. Residues of no greater than 0.065, 0.004 and 0.007 mg/kg were found in the neutral organic phases, acidic organic phases, and in the extracted aqueous phase, respectively. The organic phases were purified with silica solid phase extraction (SPE), and then eluates were analysed by HPLC. In any immature or mature sample, no single component exceeded 0.024 mg/kg or 0.007 mg/kg, respectively. The N-demethyl-175-J and N-formyl-175-J metabolites and parent XDE-175-J were tentatively identified at 0.024 mg/kg or less in 30 DAT immature samples and at 0.007 mg/kg or less in 30 DAT mature samples.

At 120 DAT and 365 DAT, no radioactive residues were associated with any of XDE-175-L, N-demethyl-175-L or N-formyl-175-L. The results of 120 DAT indicated that no single residue greater than 0.006 mg/kg was present. Of the XDE-175-J treated crop samples, radish (immature tops), radish (mature tops), lettuce (immature), and wheat forage, hay, and straw contained TRR greater than 0.010 mg/kg but the concentrations were too low for identification.

The characterization and identification of radioactive residues in 30 DAT samples following treatment with radiolabelled XDE-175-J is summarized in Table 11. Identification of the residues from XDE-175-L treated crops was not possible due to low concentration of residues.

Table 11 Characterization and identification of radioactive residues in rotational crops following application of radiolabelled spinetoram

| Compound | Radish | | | | Lettuce | | | | Wheat hay | | Wheat straw | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Immature | | Mature | | Immature | | Mature | | | | | |
| | %TRR | mg/kg | %TRR | mg/kg | %TRR | mg/kg | %TRR | mg/kg | %TRR | mg/kg | %TRR | mg/kg |
| Application of $^{14}$C-XDE-175-J at 0.41 kg ai/ha (30DAT) | | | | | | | | | | | | |
| XDE-175-J | 16.1 | 0.012 | - | - | 14.1 | 0.012 | -- | -- | -- | -- | -- | -- |
| N-demethyl-175-J | | | - | - | | | | | -- | -- | -- | -- |
| O-deethyl-175-J | 26.8 | 0.021 | - | - | 29.3 | 0.025 | 25.3 | 0.007 | -- | -- | -- | -- |
| N-formyl-175-J | | | - | - | | | | | -- | -- | -- | -- |
| Total identified | 42.9 | 0.033 | - | - | 43.4 | 0.037 | 25.3 | 0.007 | -- | -- | -- | -- |
| Total characterised | 43.2 | 0.035 | 75.4 | 0.010 | 42.3 | 0.036 | 62.7 | 0.017 | 55.3 | 0.007 | 56.7 | 0.013 |
| Total extractable | 86.1 | 0.068 | 75.4 | 0.010 | 85.7 | 0.073 | 88.0 | 0.024 | 55.3 | 0.007 | 56.7 | 0.013 |
| Total unextractable | 13.9 | 0.011 | 24.6 | 0.003 | 14.3 | 0.012 | 15.4 | 0.004 | 44.7 | 0.006 | 43.3 | 0.010 |
| Total measured (TRR in ( )) | 100 | (0.079) | 100 | (0.014) | 100 | (0.085) | 103.4 | (0.027) | 100 | (0.013) | 100 | (0.022) |
| Application of $^{14}$C-XDE-175-L at 0.13 kg ai/ha (30DAT) | | | | | | | | | | | | |
| Total identified | - | - | | | - | - | - | - | | | - | - |
| Total characterised | 60.2 | 0.013 | | | 66.3 | 0.022 | 66.5 | 0.010 | | | 50.7 | 0.007 |
| Total extractable | 60.2 | 0.013 | | | 66.3 | 0.022 | 66.5 | 0.010 | | | 50.7 | 0.007 |
| Total unextractable | 39.8 | 0.009 | | | 33.7 | 0.011 | 33.5 | 0.005 | | | 49.3 | 0.007 |
| Total measured (TRR in ( )) | 100 | (0.021) | | | 100 | (0.033) | 100 | (0.015) | | | 100 | (0.015) |

| Compound | Radish tops, immature | | Lettuce, immature | | Wheat forage | | Wheat hay | | Wheat straw | |
|---|---|---|---|---|---|---|---|---|---|---|
| | %TRR | mg/kg | %TRR | mg/kg | %TRR | mg/kg | %TRR | mg/kg | %TRR | mg/kg |
| Application of $^{14}$C-XDE-175-J at 0.41 kg ai/ha (120DAT) | | | | | | | | | | |
| Total identified | - | - | | | | | | | | |
| Total characterised | 71.4 | 0.009 | 76.6 | 0.009 | 70.7 | 0.008 | 53.9 | 0.007 | 57.3 | 0.013 |
| Total extractable | 71.4 | 0.009 | 76.6 | 0.009 | 70.7 | 0.008 | 53.9 | 0.007 | 57.3 | 0.013 |
| Total unextractable | 28.6 | 0.004 | 23.4 | 0.003 | 29.3 | 0.003 | 46.1 | 0.006 | 42.7 | 0.010 |
| Total measured (TRR in ( )) | 100 | (0.013) | 100 | (0.012) | 103 | (0.011) | 100 | (0.014) | 100 | (0.023) |

| Compound | Radish, immature tops | | Radish mature tops | | Lettuce, immature | | Wheat forage | | Wheat hay | | Wheat straw | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | %TRR | mg/kg | %TRR | mg/kg | %TRR | mg/kg | %TRR | mg/kg | %TRR | mg/kg | %TRR | mg/kg |
| Application of $^{14}$C-XDE-175-J at 0.41 kg ai/ha (365DAT) | | | | | | | | | | | | |
| Total identified | - | - | - | - | - | - | - | - | - | - | - | - |
| Total characterised | 63.4 | 0.011 | 64.7 | 0.010 | 71.8 | 0.010 | 73.2 | 0.010 | 51.1 | 0.016 | 58.2 | 0.026 |
| Total extractable | 63.4 | 0.011 | 64.7 | 0.010 | 71.8 | 0.010 | 73.2 | 0.010 | 51.1 | 0.016 | 58.2 | 0.026 |
| Total unextractable | 36.6 | 0.006 | 35.3 | 0.006 | 28.2 | 0.004 | 26.8 | 0.003 | 48.9 | 0.015 | 41.8 | 0.019 |
| Total measured (TRR in ( )) | 100 | (0.018) | 100 | (0.016) | 100 | (0.013) | 103 | (0.013) | 100 | (0.031) | 100 | (0.045) |
| Application of $^{14}$C-XDE-175-L at 0.13 kg ai/ha (365DAT) | | | | | | | | | | | | |
| Total identified | | | | | - | - | | | - | - | - | - |
| Total characterised | | | | | 60.2 | 0.010 | | | 32.6 | 0.007 | 54.1 | 0.015 |
| Total extractable | | | | | 60.2 | 0.010 | | | 32.6 | 0.007 | 54.1 | 0.015 |
| Total unextractable | | | | | 39.8 | 0.006 | | | 67.4 | 0.014 | 45.9 | 0.013 |
| Total measured (TRR in ( )) | | | | | 100 | (0.016) | | | 100 | (0.021) | 100 | (0.029) |

The levels of radioactivity taken up from soil treated with [$^{14}$C]spinetoram into the three succeeding crops (radish, lettuce, and wheat) planted 30, 120, or 365 days after treatment, were below 0.085 mg/kg spinetoram equivalents. Since such low radioactive residues were found in analysed fractions of these rotational crop samples, spinetoram is unlikely to be taken up readily by succeeding crops.

## METHODS OF RESIDUE ANALYSIS

### *Analytical methods*

Analytical methods for determination of residues of XDE-175-J and XDE-175-L and a set of their metabolites, including N-demethyl-175-J and -L, N-formyl-175-J and -L were developed for a wide range of plant matrices and animal matrices.

After an extraction specific to matrix, and a reasonably standard cleanup, spinetoram and the metabolites were determined by HPLC with positive-ion electron-spray (ESI) tandem mass spectrometry (HPLC-MS/MS). The methods have been extensively validated with numerous recovery tests on a wide range of matrices.

*Multi-residue Methods*

*FDA Multi-Residue Method PAM, Vol. I*

The applicability of the multi-residue screen methods outlined in the FDA Pesticide Analytical Manual, Volume I, Third Edition was assessed for spinetoram and its metabolites, XDE-175-J and XDE-175-L, N-demethyl-175-J and -L, and N-formyl-175-J and –L (Peyton, 2005, Report 051013). The test substances were analysed in accordance with selected multiresidue methods (MRMs) described in Protocols A and C of the FDA PAM I. Results of the tests summarized below show that the FDA PAM I multi-residue methods are not suitable for enforcement purposes.

Protocol A: XDE-175-J, XDE-175-L, N-demethyl-XDE-175-J, N-demethyl-XDE-175-L, N-formyl-XDE-175-J and N-formyl-XDE-175-L were each injected at approximately 1000 ng and showed no response in FSD when analysed according to the above procedures outlined in Protocol A. Because no fluorescence was demonstrated for any of the test substances, no further procedures were required for Protocol A

Protocol B: Because the analytes are not acids or phenols, testing through Protocol B was not required.

Protocol C: All test substances were subjected to Protocol C, modules DG-1, DG-5, DG-13, DG-17, and DG-18. GC systems for each module were set up appropriately according to the parameters described in the PAM Volume 1, Section 302. The test substances were analysed using a nitrogen-phosphorous detector set to nitrogen mode detection according to modules DG-5 and DG-17. DG-5 analysed the test substances at 1000 ng. Results for the analytes at 1000 ng were off-scale and therefore were reanalysed at 200 ng. DG-5 analysis resulted in sample peaks that were broad with significant tailing. DG-17 analysis resulted in sample peaks that were less broad but still contained a significantly large tailing factor. Modules DG-5 and DG-17 were unsuccessful for XDE-175 due to broad peak shape and tailing factors observed. Failure of the test substances to produce quantifiable peaks using the nitrogen-specific modules led to analysis of the test substance by more general modules, non-specific for chemical properties, DG-1, DG-13 and DG-18. No peaks were observed for any of the test substances using electron capture detection modules. Protocol C was unsuccessful due to chromatographic peak broadening and tailing and (for electron capture detection) a lack of detectability.

Protocols D, E, and F: Due to the poor sensitivity and poor chromatographic properties of the test substances to detection by methods described in Protocol C, no further analyses were performed by Protocol D and Protocol E.

Protocol G: Because the test substances are not carbamates, testing through Protocol G was not required.

*Multi-Residue Enforcement Method, DFG S19*

The German multi-residue enforcement method DFG S19 was intended for the determination of pesticides by gas chromatography (GC). Recently it was proposed to extend the S19 based modular multi-method L 00.00-34 (German §35 LMBG) by a module using liquid chromatography with tandem mass spectrometric detection (LC-MS/MS) for the determination of a multitude of active substances.

Various DFG S19 extraction modules were employed: extraction module E 1 was used for apple; extraction module E 2 was assessed for wheat grain; module E 3 was used for whole orange and grape; and module E 7 was assessed for oilseed rape seed. Basic steps of the E 1, E 2, and E 3 extraction modules are adjustment of neutral pH (if necessary) and total water to 100 mL followed by extraction with water/acetone (1/2, v/v) and subsequent partition into the organic phase by addition of sodium chloride and ethyl acetate/cyclohexane (1/1, v/v). Module E 7 uses extraction with acetonitrile/acetone (225/25, v/v) with addition of synthetic calcium silicate (Calflo E) and Celite to eliminate oily plant matrix and facilitate filtration.

The DFG S19 multiresidue method, in conjunction with LC-MS/MS determination, using matrix matched standards for external calibration, was validated for XDE-175-J and XDE-175-L, N-demethyl-175-J and -L, and N-formyl-175-J and -L in three plant matrices, apple, grape, and orange (Class, 2007; Report 061066). Validation was also attempted for wheat grain and for oilseed rape seed but was unsuccessful most likely due to incomplete extraction and excessive matrix effects. Recoveries of spinetoram and its metabolites in apples, oranges and grape using the multiresidue method are summarized in Table 12**.** Average recoveries ranged between 73% and 102%, with relative standard deviations of ≤ 14% for all analytes and matrices.

Table 12 Procedural recovery for spinetoram and its metabolites from fortified apple, grape, and orange samples, using the multiresidue method, DFG S-19

| Analyte | Matrix | Fortification Level (mg/kg) | Recovery Rate (%) | | RSD (%) | n |
|---|---|---|---|---|---|---|
| | | | mean | range | | |
| XDE-175-J *m/z* Q1/Q3 748.7/142.1 | Apple | 0.010 | 83 | 77–91 | 8 | 5 |
| | | 1.0 | 84 | 81–88 | 3 | 5 |
| | | 0.010–1.0 | 84 | 77–91 | 6 | 10 |
| XDE-175-L *m/z* Q1/Q3 760.7/142.1 | Apple | 0.010 | 83 | 77–91 | 8 | 5 |
| | | 1.0 | 79 | 71–86 | 8 | 5 |
| | | 0.010–1.0 | 81 | 71–91 | 8 | 10 |
| *N*-demethyl-175-J *m/z* Q1/Q3 734.7/127.9 | Apple | 0.010 | 82 | 75–90 | 7 | 5 |
| | | 1.0 | 79 | 76–81 | 3 | 5 |
| | | 0.010–1.0 | 80 | 75–90 | 6 | 10 |
| *N*-demethyl-175-L *m/z* Q1/Q3 746.8/127.9 | Apple | 0.010 | 82 | 69–95 | 13 | 5 |
| | | 1.0 | 73 | 69–78 | 5 | 5 |
| | | 0.010–1.0 | 79 | 69–95 | 11 | 10 |
| *N*-formyl-175-J *m/z* Q1/Q3 762.3/156.1 | Apple | 0.010 | 96 | 90–100 | 9 | 5 |
| | | 1.0 | 91 | 88–92 | 5 | 5 |
| | | 0.010–1.0 | 93 | 88–100 | 8 | 10 |
| *N*-formyl-175-L *m/z* Q1/Q3 774.3/155.9 | Apple | 0.010 | 103 | 85–112 | 11 | 5 |
| | | 1.0 | 87 | 84–89 | 2 | 5 |
| | | 0.010–1.0 | 95 | 84–112 | 12 | 10 |
| XDE-175-J *m/z* Q1/Q3 748.7/98.2 | Apple | 0.010 | 85 | 79–92 | 6 | 5 |
| | | 1.0 | 82 | 79–86 | 4 | 5 |
| | | 0.010–1.0 | 83 | 79–92 | 5 | 10 |
| XDE-175-L *m/z* Q1/Q3 760.7/98.2 | Apple | 0.010 | 80 | 77–87 | 5 | 5 |
| | | 1.0 | 79 | 71–86 | 8 | 5 |
| | | 0.010–1.0 | 79 | 71–87 | 6 | 10 |
| *N*-demethyl-175-J *m/z* Q1/Q3 734.7/84.2 | Apple | 0.010 | 85 | 77–94 | 8 | 5 |
| | | 1.0 | 72 | 69–75 | 4 | 5 |
| | | 0.010–1.0 | 78 | 69–94 | 11 | 10 |
| *N*-demethyl-175-L *m/z* Q1/Q3 746.8/84.2 | Apple | 0.010 | 86 | 76–98 | 12 | 5 |
| | | 1.0 | 73 | 69–79 | 6 | 5 |
| | | 0.010–1.0 | 80 | 69–98 | 12 | 10 |
| *N*-formyl-175-J *m/z* Q1/Q3 762.3/203.0 | Apple | 0.010 | 105 | 98–112 | 5 | 5 |
| | | 1.0 | 89 | 88–90 | 1 | 5 |
| | | 0.010–1.0 | 97 | 88–112 | 9 | 10 |
| *N*-formyl-175-L *m/z* Q1/Q3 774.3/203.0 | Apple | 0.010 | 105 | 91–115 | 9 | 5 |
| | | 1.0 | 87 | 84–89 | 2 | 5 |

| Analyte | Matrix | Fortification Level (mg/kg) | Recovery Rate (%) | | RSD (%) | n |
|---|---|---|---|---|---|---|
| | | | mean | range | | |
| | | 0.010–1.0 | 96 | 84–115 | 12 | 10 |
| XDE-175-J *m/z* Q1/Q3 748.7/142.1 | Grape | 0.010 | 92 | 84–96 | 6 | 4 |
| | | 1.0 | 84 | 77–91 | 7 | 5 |
| | | 0.010–1.0 | 87 | 77–96 | 7 | 9 |
| XDE-175-L *m/z* Q1/Q3 760.7/142.1 | Grape | 0.010 | 88 | 80–92 | 6 | 4 |
| | | 1.0 | 78 | 71–84 | 7 | 5 |
| | | 0.010–1.0 | 82 | 71–92 | 9 | 9 |
| *N*-demethyl-175-J *m/z* Q1/Q3 734.7/127.9 | Grape | 0.010 | 84 | 77–91 | 7 | 4 |
| | | 1.0 | 78 | 73–83 | 6 | 5 |
| | | 0.010–1.0 | 81 | 73–91 | 7 | 9 |
| *N*-demethyl-175-L *m/z* Q1/Q3 746.8/127.9 | Grape | 0.010 | 86 | 76–100 | 12 | 4 |
| | | 1.0 | 73 | 69–77 | 4 | 5 |
| | | 0.010–1.0 | 79 | 69–100 | 12 | 9 |
| *N*-formyl-175-J *m/z* Q1/Q3 762.3/156.1 | Grape | 0.010 | 102 | 88–111 | 9 | 5 |
| | | 1.0 | 94 | 90–100 | 5 | 5 |
| | | 0.010–1.0 | 98 | 88–111 | 8 | 10 |
| *N*-formyl-175-L *m/z* Q1/Q3 774.3/155.9 | Grape | 0.010 | 97 | 89–106 | 8 | 5 |
| | | 1.0 | 94 | 91–98 | 3 | 5 |
| | | 0.010–1.0 | 96 | 89–106 | 6 | 10 |
| XDE-175-J *m/z* Q1/Q3 748.7/98.2 | Grape | 0.010 | 90 | 85–95 | 5 | 4 |
| | | 1.0 | 82 | 76–89 | 7 | 5 |
| | | 0.010–1.0 | 86 | 76–95 | 8 | 9 |
| XDE-175-L *m/z* Q1/Q3 760.7/98.2 | Grape | 0.010 | 84 | 77–89 | 7 | 4 |
| | | 1.0 | 76 | 69–83 | 8 | 5 |
| | | 0.010–1.0 | 80 | 69–89 | 9 | 9 |
| *N*-demethyl-175-J *m/z* Q1/Q3 734.7/84.2 | Grape | 0.010 | 85 | 78–95 | 9 | 4 |
| | | 1.0 | 77 | 72–81 | 5 | 5 |
| | | 0.010–1.0 | 80 | 72–95 | 9 | 9 |
| *N*-demethyl-175-L *m/z* Q1/Q3 746.8/84.2 | Grape | 0.010 | 91 | 77–106 | 13 | 4 |
| | | 1.0 | 75 | 72–79 | 4 | 5 |
| | | 0.010–1.0 | 82 | 72–106 | 14 | 9 |
| *N*-formyl-175-J *m/z* Q1/Q3 762.3/203.0 | Grape | 0.010 | 97 | 89–106 | 7 | 5 |
| | | 1.0 | 94 | 89–100 | 5 | 5 |
| | | 0.010–1.0 | 96 | 89–106 | 6 | 10 |
| *N*-formyl-175-L *m/z* Q1/Q3 774.3/203.0 | Grape | 0.010 | 96 | 83–113 | 12 | 5 |
| | | 1.0 | 91 | 86–98 | 6 | 5 |
| | | 0.010–1.0 | 94 | 83–113 | 9 | 10 |
| XDE-175-J *m/z* Q1/Q3 748.7/142.1 | Orange | 0.010 | 102 | 92–114 | 9 | 5 |
| | | 1.0 | 97 | 93–100 | 3 | 5 |
| | | 0.010–1.0 | 99 | 92–114 | 7 | 10 |
| XDE-175-L *m/z* Q1/Q3 760.7/142.1 | Orange | 0.010 | 94 | 86–104 | 7 | 5 |
| | | 1.0 | 92 | 81–99 | 9 | 5 |
| | | 0.010–1.0 | 93 | 81–104 | 7 | 10 |
| *N*-demethyl-175-J | Orange | 0.010 | 96 | 89–112 | 10 | 5 |

| Analyte | Matrix | Fortification Level (mg/kg) | Recovery Rate (%) | | RSD (%) | n |
|---|---|---|---|---|---|---|
| | | | mean | range | | |
| *m/z* Q1/Q3 734.7/127.9 | | 1.0 | 93 | 88–96 | 4 | 5 |
| | | 0.010–1.0 | 95 | 88–112 | 7 | 10 |
| *N*-demethyl-175-L | Orange | 0.010 | 91 | 83–113 | 14 | 5 |
| *m/z* Q1/Q3 746.8/127.9 | | 1.0 | 89 | 79–95 | 8 | 5 |
| | | 0.010–1.0 | 90 | 79–113 | 11 | 10 |
| *N*-formyl-175-J | Orange | 0.010 | 102 | 95–105 | 4 | 5 |
| *m/z* Q1/Q3 762.3/156.1 | | 1.0 | 95 | 92–98 | 3 | 5 |
| | | 0.010–1.0 | 99 | 92–105 | 5 | 10 |
| *N*-formyl-175-L | Orange | 0.010 | 96 | 90–107 | 7 | 5 |
| *m/z* Q1/Q3 774.3/155.9 | | 1.0 | 102 | 97–105 | 3 | 5 |
| | | 0.010–1.0 | 99 | 90–107 | 6 | 10 |
| XDE-175-J | Orange | 0.010 | 95 | 87–104 | 8 | 5 |
| *m/z* Q1/Q3 748.7/98.2 | | 1.0 | 93 | 89–96 | 3 | 5 |
| | | 0.010–1.0 | 94 | 87–104 | 6 | 10 |
| XDE-175-L | Orange | 0.010 | 93 | 86–101 | 6 | 5 |
| *m/z* Q1/Q3 760.7/98.2 | | 1.0 | 90 | 79–97 | 9 | 5 |
| | | 0.010–1.0 | 92 | 79–101 | 8 | 10 |
| *N*-demethyl-175-J | Orange | 0.010 | 89 | 82–100 | 8 | 5 |
| *m/z* Q1/Q3 734.7/84.2 | | 1.0 | 90 | 85–92 | 4 | 5 |
| | | 0.010–1.0 | 90 | 82–100 | 6 | 10 |
| *N*-demethyl-175-L | Orange | 0.010 | 92 | 84–106 | 10 | 5 |
| *m/z* Q1/Q3 746.8/84.2 | | 1.0 | 87 | 79–92 | 8 | 5 |
| | | 0.010–1.0 | 89 | 79–106 | 9 | 10 |
| *N*-formyl-175-J | Orange | 0.010 | 102 | 98–108 | 5 | 5 |
| *m/z* Q1/Q3 762.3/203.0 | | 1.0 | 95 | 91–98 | 3 | 5 |
| | | 0.010–1.0 | 99 | 91–108 | 5 | 10 |
| *N*-formyl-175-L | Orange | 0.010 | 101 | 92–104 | 6 | 5 |
| *m/z* Q1/Q3 774.3/203.0 | | 1.0 | 101 | 98–104 | 3 | 5 |
| | | 0.010–1.0 | 101 | 92–104 | 4 | 10 |

The FDA PAM I multi-residue methods were found to be unsuitable for the determination of spinetoram and its metabolites in plant and animal matrices for enforcement purposes.

The DFG S19 multi-residue method was only successfully validated for the determination of spinetoram and it metabolites in apples, grapes and oranges when using matrix-matched calibration standards. It was not successful for the determination of the analytes in wheat grain and for oilseed rape seed due to matrix effects. The addition of GPC clean-up was not helpful in achieving successful results, most likely due to the fact that the large molecules elute early with the oily biomolecules. It was therefore predicted that this method would not work for determination of spinetoram and its metabolites in animal matrices.

*Analytical Methods for Determination of Residues in Foods and Feeding stuffs of Plant Origin Used in Supervised Trials*

Methods GRM 05.03 and GRM 05.04 have been developed for the determination of residues of XDE-175-J and XDE-175-L and their metabolites N-demethyl-175-J and -L, N-formyl-175-J and -L in plant matrices using HPLC with positive-ion electron-spray (ESI) tandem mass spectrometry (LC-MS/MS).

*Method GRM 05.03*

In method GRM 05.03 developed by Hastings and Wendelburg (2005), residues of spinetoram and its metabolites are extracted from agricultural commodities by shaking with an acetonitrile/water (80:20, v/v) solution. An aliquot of the extraction solvent is removed and a stable isotope internal standard solution containing XDE-175 and metabolites is added. XDE-175 and its metabolites are analysed without sample clean-up by liquid chromatography with positive-ion electron-spray ionization (ESI) tandem mass spectrometry (LC-MS/MS).

The method was validated over the concentration range of 0.01-1.0 mg/kg for all crops (dry crops, wet crops, oily crops and acidic crops), except lettuce which was validated over the concentration range of 0.01–10 mg/kg. The validated limit of quantification (LOQ) for all crops was 0.01 mg/kg (Hastings, 2005, Report 041021). Recoveries of spinetoram and its metabolites in a number of crops fortified at various levels are summarized in Table 13**.** Average recovery ranged between 82% and 106%, with relative standard deviations of ≤ 15% for analytes in all crops, except for N-formyl-175-L in oily crops, where the relative standard deviation was 27%.

Table 13 Procedural recovery for spinetoram and its metabolites in various plant matrices using Method GRM 05.03

| Analyte | Matrix | Fortification Level (mg/kg) | Recovery Rate (%) | | RSD (%) | n |
|---|---|---|---|---|---|---|
| | | | mean | range | | |
| XDE-175-J | Wet Crop [a] | 0.01 | 99 | 89–107 | 5.1 | 18 |
| | | 1.0 | 96 | 88–101 | 3.7 | 16 |
| | | 10 | 93 | 89–99 | 4.3 | 6 |
| | | 0.01–10 | 97 | 88–107 | 4.9 | 40 |
| XDE-175-L | Wet Crop | 0.01 | 106 | 97–116 | 4.4 | 18 |
| | | 1.0 | 102 | 97–107 | 3.0 | 16 |
| | | 10 | 93 | 90–96 | 2.1 | 6 |
| | | 0.01–10 | 103 | 90–116 | 5.4 | 40 |
| N-demethyl-175-J | Wet Crop | 0.01 | 99 | 89–116 | 7.1 | 18 |
| | | 1.0 | 102 | 98–109 | 3.1 | 16 |
| | | 10 | 92 | 87–97 | 3.6 | 6 |
| | | 0.01–10 | 99 | 87–116 | 6.4 | 40 |
| N-demethyl-175-L | Wet Crop | 0.01 | 98 | 89–114 | 6.6 | 18 |
| | | 1.0 | 100 | 96–103 | 2.1 | 16 |
| | | 10 | 93 | 90–95 | 1.8 | 6 |
| | | 0.01–10 | 98 | 89–114 | 5.2 | 40 |
| N-formyl-175-J | Wet Crop | 0.01 | 90 | 79–106 | 8.1 | 18 |
| | | 1.0 | 94 | 87–100 | 4.6 | 16 |
| | | 10 | 88 | 82–96 | 5.9 | 6 |
| | | 0.01–10 | 91 | 79–106 | 6.8 | 40 |
| N-formyl-175-L | Wet Crop | 0.01 | 89 | 76–101 | 6.9 | 18 |
| | | 1.0 | 92 | 86–100 | 4.0 | 16 |
| | | 10 | 89 | 87–92 | 2.6 | 6 |
| | | 0.01–10 | 90 | 76–101 | 5.4 | 40 |
| XDE-175-J | Dry Crop [b] | 0.01 | 90 | 83–103 | 5.6 | 20 |
| | | 1.0 | 91 | 84–100 | 4.5 | 20 |
| | | 0.01–1.0 | 91 | 83–103 | 5.0 | 40 |
| XDE-175-L | Dry Crop | 0.01 | 104 | 97–114 | 4.5 | 20 |
| | | 1.0 | 102 | 96–109 | 2.9 | 20 |

| Analyte | Matrix | Fortification Level (mg/kg) | Recovery Rate (%) | | RSD (%) | n |
|---|---|---|---|---|---|---|
| | | | mean | range | | |
| | | 0.01–1.0 | 103 | 96–114 | 3.9 | 40 |
| N-demethyl-175-J | Dry Crop | 0.01 | 102 | 94–109 | 3.6 | 20 |
| | | 1.0 | 103 | 97–110 | 3.1 | 20 |
| | | 0.01–1.0 | 103 | 94–110 | 3.3 | 40 |
| N-demethyl-175-L | Dry Crop | 0.01 | 103 | 94–109 | 4.1 | 20 |
| | | 1.0 | 104 | 98–113 | 3.5 | 20 |
| | | 0.01–1.0 | 104 | 94–113 | 3.8 | 40 |
| N-formyl-175-J | Dry Crop | 0.01 | 99 | 77–115 | 9.1 | 20 |
| | | 1.0 | 100 | 82–111 | 7.0 | 20 |
| | | 0.01–1.0 | 99 | 77–115 | 8.0 | 40 |
| N-formyl-175-L | Dry Crop | 0.01 | 91 | 71–109 | 11.0 | 20 |
| | | 1.0 | 96 | 81–109 | 8.5 | 20 |
| | | 0.01–1.0 | 93 | 71–109 | 10.1 | 40 |
| XDE-175-J | Acidic Crop [c] | 0.01 | 99 | 89–114 | 7.2 | 17 |
| | | 1.0 | 94 | 86–100 | 4.0 | 18 |
| | | 0.01–1.0 | 96 | 86–114 | 6.4 | 35 |
| XDE-175-L | Acidic Crop | 0.01 | 107 | 97–116 | 5.4 | 17 |
| | | 1.0 | 102 | 95–107 | 3.1 | 18 |
| | | 0.01–1.0 | 104 | 95–116 | 5.2 | 35 |
| N-demethyl-175-J | Acidic Crop | 0.01 | 102 | 95–115 | 5.1 | 17 |
| | | 1.0 | 100 | 96–106 | 2.8 | 18 |
| | | 0.01–1.0 | 101 | 95–115 | 4.2 | 35 |
| N-demethyl-175-L | Acidic Crop | 0.01 | 102 | 93–112 | 5.7 | 17 |
| | | 1.0 | 101 | 96–106 | 2.8 | 18 |
| | | 0.01–1.0 | 101 | 93–112 | 4.4 | 35 |
| N-formyl-175-J | Acidic Crop | 0.01 | 94 | 79–112 | 10.5 | 17 |
| | | 1.0 | 94 | 80–106 | 7.2 | 18 |
| | | 0.01–1.0 | 94 | 79–112 | 8.8 | 35 |
| N-formyl-175-L | Acidic Crop | 0.01 | 93 | 73–107 | 10.2 | 17 |
| | | 1.0 | 94 | 86–100 | 3.9 | 18 |
| | | 0.01–1.0 | 93 | 73–107 | 7.5 | 35 |
| XDE-175-J | Oily Crop [d] | 0.01 | 94 | 88–102 | 5.3 | 10 |
| | | 1.0 | 95 | 87–102 | 4.7 | 10 |
| | | 0.01–1.0 | 95 | 87–102 | 4.9 | 20 |
| XDE-175-L | Oily Crop | 0.01 | 96 | 90–102 | 4.5 | 10 |
| | | 1.0 | 100 | 95–107 | 4.3 | 10 |
| | | 0.01–1.0 | 98 | 90–107 | 4.8 | 20 |
| N-demethyl-175-J | Oily Crop | 0.01 | 96 | 92–101 | 3.2 | 10 |
| | | 1.0 | 102 | 95–110 | 5.1 | 10 |
| | | 0.01–1.0 | 99 | 92–110 | 5.1 | 20 |
| N-demethyl-175-L | Oily Crop | 0.01 | 89 | 80–99 | 6.0 | 10 |
| | | 1.0 | 94 | 84–101 | 5.2 | 10 |
| | | 0.01–1.0 | 92 | 80–101 | 6.3 | 20 |
| N-formyl-175-J | Oily Crop | 0.01 | 89 | 71–110 | 15.0 | 10 |

| Analyte | Matrix | Fortification Level (mg/kg) | Recovery Rate (%) | | RSD (%) | n |
|---|---|---|---|---|---|---|
| | | | mean | range | | |
| | | 1.0 | 96 | 85–105 | 6.8 | 10 |
| | | 0.01–1.0 | 92 | 71–110 | 11.7 | 20 |
| | | | | | | |
| N-formyl-175-L | Oily Crop | 0.01 | 80 | 48–113 | 27.5 | 10 |
| | | 1.0 | 83 | 48–108 | 25.1 | 10 |
| | | 0.01–1.0 | 82 | 48–113 | 25.7 | 20 |

[a] "Wet crop" includes: broccoli, cabbage, grape, green beans (succulent), leek, lettuce, onion, sweet peppers, and tomato.

[b] "Dry crop" includes: barley grain, forage and straw, grass forage, maize grain forage and stover, and wheat grain, forage and straw.

[c] "Acidic crop" includes: apple, cherry (without seed), lemon, orange (whole fruit, peel and pulp), peach (without seed), pear and plum (without seed).

[d] "Oily crop" canola seed, cotton seed, olive (without seed), olive oil and soya bean.

An independent validation was carried out at PTRL Europe by analysts having no previous experience with the residue method (Richter, 2005; Report 050051). The limit of quantification was established at 0.01 mg/kg for spinetoram and its metabolites in lettuce (wet crop) and in whole oranges (acidic crop). The final extracts were analysed for residues of spinetoram and its metabolites by LC-MS/MS. Table 14 summarizes the validation data for spinetoram and its metabolites in lettuce and oranges. Average recoveries ranged between 82% and 110%, with relative standard deviations of ≤ 14%. Method GRM 05.03 was demonstrated to be applicable for use in the determination of spinetoram and its metabolites residues in these matrices.

Table 14 Procedural recovery obtained by an independent laboratory validation of Method GRM 05.03

| Analyte | Matrix | Fortification Level (mg/kg) | Recovery Rate (%) | | RSD (%) | n |
|---|---|---|---|---|---|---|
| | | | mean | range | | |
| XDE-175-J | Lettuce (Wet Crop) | 0.01 | 92 | 89–95 | 3 | 5 |
| | | 10 | 83 | 80–86 | 3 | 5 |
| | | 0.01–10 | 88 | 80–95 | 6 | 10 |
| XDE-175-L | Lettuce (Wet Crop) | 0.01 | 82 | 77–86 | 5 | 5 |
| | | 10 | 85 | 83–90 | 4 | 5 |
| | | 0.01–10 | 84 | 77–90 | 5 | 10 |
| N-demethyl-175-J | Lettuce (Wet Crop) | 0.01 | 89 | 84–92 | 3 | 5 |
| | | 10 | 92 | 85–98 | 6 | 5 |
| | | 0.01–10 | 90 | 84–98 | 5 | 10 |
| N-demethyl-175-L | Lettuce (Wet Crop) | 0.01 | 87 | 71–105 | 14 | 5 |
| | | 10 | 87 | 75–101 | 14 | 5 |
| | | 0.01–10 | 87 | 71–105 | 13 | 10 |
| N-formyl-175-J | Lettuce (Wet Crop) | 0.01 | 100 | 93–111 | 8 | 5 |
| | | 10 | 83 | 76–97 | 10 | 5 |
| | | 0.01–10 | 91 | 76–111 | 13 | 10 |
| N-formyl-175-L | Lettuce (Wet Crop) | 0.01 | 99 | 89–112 | 10 | 5 |
| | | 10 | 82 | 77–88 | 5 | 5 |
| | | 0.01–10 | 91 | 77–112 | 12 | 10 |

| Analyte | Matrix | Fortification Level (mg/kg) | Recovery Rate (%) | | RSD (%) | n |
|---|---|---|---|---|---|---|
| | | | mean | range | | |
| XDE-175-J | Whole Orange (Acidic Crop) | 0.01 | 95 | 87–100 | 5 | 5 |
| | | 0.10 | 92 | 89–98 | 4 | 5 |
| | | 0.01–0.10 | 94 | 87–100 | 5 | 10 |
| XDE-175-L | Whole Orange (Acidic Crop) | 0.01 | 85 | 76–92 | 8 | 5 |
| | | 0.10 | 93 | 89–98 | 5 | 5 |
| | | 0.01–0.10 | 89 | 76–98 | 8 | 10 |
| N-demethyl-175-J | Whole Orange (Acidic Crop) | 0.01 | 89 | 87–93 | 3 | 5 |
| | | 0.10 | 87 | 84–91 | 4 | 5 |
| | | 0.01–0.10 | 88 | 84–93 | 4 | 40 |
| N-demethyl-175-L | Whole Orange (Acidic Crop) | 0.01 | 90 | 81–105 | 11 | 5 |
| | | 0.10 | 90 | 73–100 | 12 | 5 |
| | | 0.01–0.10 | 90 | 73–105 | 11 | 10 |
| N-formyl-175-J | Whole Orange (Acidic Crop) | 0.01 | 91 | 83–104 | 11 | 5 |
| | | 0.10 | 89 | 71–95 | 11 | 5 |
| | | 0.01–0.10 | 90 | 71–104 | 10 | 10 |
| N-formyl-175-L | Whole Orange (Acidic Crop) | 0.01 | 98 | 96–104 | 3 | 5 |
| | | 0.10 | 95 | 87–103 | 6 | 5 |
| | | 0.01–0.10 | 97 | 87–104 | 5 | 10 |
| XDE-175-J | Lettuce (Wet Crop) | 0.01 | 90 | 81–102 | 9 | 5 |
| | | 10 | 90 | 87–94 | 2 | 5 |
| | | 0.01–10 | 90 | 81–102 | 6 | 10 |
| XDE-175-L | Lettuce (Wet Crop) | 0.01 | 96 | 92–103 | 7 | 5 |
| | | 10 | 86 | 84–90 | 2 | 5 |
| | | 0.01–10 | 91 | 84–103 | 8 | 10 |
| N-demethyl-175-J | Lettuce (Wet Crop) | 0.01 | 89 | 82–93 | 5 | 5 |
| | | 10 | 89 | 83–99 | 7 | 5 |
| | | 0.01–10 | 89 | 82- 99 | 6 | 10 |
| N-demethyl-175-L | Lettuce (Wet Crop) | 0.01 | 83 | 72–92 | 9 | 5 |
| | | 10 | 87 | 77–95 | 8 | 5 |
| | | 0.01–10 | 85 | 72–95 | 8 | 10 |
| N-formyl-175-J | Lettuce (Wet Crop) | 0.01 | 88 | 82–96 | 7 | 5 |
| | | 10 | 98 | 92–110 | 10 | 5 |
| | | 0.01–10 | 93 | 82–110 | 10 | 10 |
| N-formyl-175-L | Lettuce (Wet Crop) | 0.01 | 93 | 70–104 | 15 | 5 |
| | | 10 | 89 | 74–109 | 14 | 5 |
| | | 0.01–10 | 91 | 70–109 | 14 | 10 |
| XDE-175-J | Whole Orange (Acidic Crop) | 0.01 | 89 | 82–100 | 8 | 5 |
| | | 0.10 | 96 | 91–100 | 4 | 5 |
| | | 0.01–0.10 | 93 | 82–100 | 7 | 10 |
| XDE-175-L | Whole Orange (Acidic Crop) | 0.01 | 91 | 87–96 | 4 | 5 |
| | | 0.10 | 92 | 84–100 | 6 | 5 |
| | | 0.01–0.10 | 91 | 84–100 | 5 | 10 |

| Analyte | Matrix | Fortification Level (mg/kg) | Recovery Rate (%) | | RSD (%) | n |
|---|---|---|---|---|---|---|
| | | | mean | range | | |
| N-demethyl-175-J | Whole Orange (Acidic Crop) | 0.01 | 89 | 84–93 | 4 | 5 |
| | | 0.10 | 72 | 70–76 | 4 | 5 |
| | | 0.01–0.10 | 80 | 70–93 | 11 | 40 |
| N-demethyl-175-L | Whole Orange (Acidic Crop) | 0.01 | 94 | 87–107 | 8 | 5 |
| | | 0.10 | 106 | 100- 109 | 3 | 5 |
| | | 0.01–0.10 | 100 | 87–109 | 9 | 10 |
| N-formyl-175-J | Whole Orange (Acidic Crop) | 0.01 | 100 | 88–119 | 13 | 5 |
| | | 0.10 | 110 | 101 -118 | 7 | 5 |
| | | 0.01–0.10 | 105 | 88–119 | 11 | 10 |
| N-formyl-175-L | Whole Orange (Acidic Crop) | 0.01 | 100 | 88–108 | 7 | 5 |
| | | 0.10 | 110 | 103- 118 | 6 | 5 |
| | | 0.01–0.10 | 105 | 88–118 | 8 | 10 |

*Method GRM 05.04*

Method GRM 05.04 was developed for the determination of residues of XDE-175-J and XDE-175-L and their corresponding metabolites, N-demethyl-175-J and -L, and N-formyl-175-J and -L in plant matrices by on-line solid phase extraction and HPLC with positive-ion electron-spray ionization (ESI) tandem mass spectrometry (LC-MS/MS) (Hastings and Wendelburg, 2005). The method was proposed as a data-gathering method.

Residues of spinetoram and its metabolites are extracted from agricultural commodities by shaking with an acetonitrile/water (80:20, v/v). An aliquot of the extraction solvent is removed and a stable isotope internal standard solution containing XDE-175 and metabolites is added. The solution is purified by on-line solid phase extraction (SPE) using a C18 cartridge. The extract is loaded onto the SPE cartridge with an acetonitrile/methanol/water (15:15:70, v/v) containing ammonium acetate. The SPE cartridge is washed with an acetonitrile/methanol/water (25:25:50, v/v) containing ammonium acetate and eluted onto the analytical C18 column with a gradient elution technique using the HPLC mobile phase. Spinetoram and its metabolites are analysed by liquid chromatography with positive-ion electron-spray ionization (ESI) tandem mass spectrometry (LC-MS/MS).

The method was validated over the concentration range of 0.01–.0 mg/kg for dry crops, oily crops and acidic crops, and over the concentration range of 0.01–10 mg/kg for wet crops. The validated limit of quantification (LOQ) for all crops was 0.01 mg/kg (Hastings, 2005, Report 041021). Recoveries of spinetoram and its metabolites in a number of crops fortified at various levels are summarized in Table 15. Average recovery ranged between 88% and 111%, with relative standard deviations of ≤ 20% for all analytes and crops tested.

Table 15 Procedural recovery for spinetoram and its metabolites in various plant matrices using Method GRM 05.04 (Hastings, 2005)

| Analyte | Matrix | Fortification Level (mg/kg) | Recovery Rate (%) | | RSD (%) | n |
|---|---|---|---|---|---|---|
| | | | mean | range | | |
| XDE-175-J | Wet Crop [a] | 0.01 | 103 | 95–118 | 5.6 | 18 |
| | | 1.0 | 99 | 92–103 | 3.5 | 16 |
| | | 10 | 92 | 90–94 | 1.3 | 6 |
| | | 0.01–10 | 100 | 90–118 | 5.7 | 40 |
| XDE-175-L | Wet Crop | 0.01 | 104 | 96–114 | 5.3 | 18 |
| | | 1.0 | 101 | 95–107 | 3.0 | 16 |
| | | 10 | 92 | 89–98 | 3.2 | 6 |

| Analyte | Matrix | Fortification Level (mg/kg) | Recovery Rate (%) | | RSD (%) | n |
|---|---|---|---|---|---|---|
| | | | mean | range | | |
| | | 0.01–10 | 101 | 89–114 | 6.0 | 40 |
| N-demethyl-175-J | Wet Crop | 0.01 | 100 | 94–117 | 5.8 | 18 |
| | | 1.0 | 100 | 94–105 | 2.8 | 16 |
| | | 10 | 94 | 89–101 | 5.5 | 6 |
| | | 0.01–10 | 99 | 89–117 | 5.3 | 40 |
| N-demethyl-175-L | Wet Crop | 0.01 | 102 | 92–120 | 6.6 | 18 |
| | | 1.0 | 101 | 95–104 | 2.3 | 16 |
| | | 10 | 91 | 87–99 | 5.4 | 6 |
| | | 0.01–10 | 100 | 87–120 | 6.3 | 40 |
| N-formyl-175-J | Wet Crop | 0.01 | 93 | 86–106 | 7.2 | 18 |
| | | 1.0 | 101 | 91–116 | 6.9 | 16 |
| | | 10 | 91 | 84–101 | 7.6 | 6 |
| | | 0.01–10 | 96 | 84–116 | 8.3 | 40 |
| N-formyl-175-L | Wet Crop | 0.01 | 100 | 79–119 | 11.2 | 18 |
| | | 1.0 | 99 | 91–111 | 4.9 | 16 |
| | | 10 | 90 | 81–99 | 7.8 | 6 |
| | | 0.01–10 | 98 | 79–119 | 9.3 | 40 |
| XDE-175-J | Dry Crop [b] | 0.01 | 88 | 81–105 | 6.0 | 20 |
| | | 1.0 | 88 | 81–98 | 5.6 | 20 |
| | | 0.01–1.0 | 88 | 81–105 | 5.7 | 40 |
| XDE-175-L | Dry Crop | 0.01 | 102 | 94–114 | 5.5 | 20 |
| | | 1.0 | 102 | 92–114 | 6.2 | 20 |
| | | 0.01–1.0 | 102 | 92–114 | 5.8 | 40 |
| N-demethyl-175-J | Dry Crop | 0.01 | 102 | 92–114 | 5.1 | 20 |
| | | 1.0 | 102 | 95–114 | 3.8 | 20 |
| | | 0.01–1.0 | 102 | 92–114 | 4.4 | 40 |
| N-demethyl-175-L | Dry Crop | 0.01 | 105 | 99–114 | 4.3 | 20 |
| | | 1.0 | 104 | 96–110 | 3.6 | 20 |
| | | 0.01–1.0 | 105 | 96–114 | 4.0 | 40 |
| N-formyl-175-J | Dry Crop | 0.01 | 105 | 77–131 | 12.5 | 20 |
| | | 1.0 | 110 | 97–121 | 5.3 | 20 |
| | | 0.01–1.0 | 108 | 77–131 | 9.7 | 40 |
| N-formyl-175-L | Dry Crop | 0.01 | 95 | 78–119 | 12.1 | 20 |
| | | 1.0 | 93 | 77–117 | 12.5 | 20 |
| | | 0.01–1.0 | 94 | 77–119 | 12.2 | 40 |
| XDE-175-J | Acidic Crop [c] | 0.01 | 102 | 93–115 | 6.0 | 17 |
| | | 1.0 | 101 | 94–105 | 3.3 | 18 |
| | | 0.01–1.0 | 102 | 93–115 | 4.8 | 35 |
| XDE-175-L | Acidic Crop | 0.01 | 106 | 98–114 | 4.6 | 17 |
| | | 1.0 | 100 | 92–104 | 3.0 | 18 |
| | | 0.01–1.0 | 103 | 92–114 | 5.0 | 35 |
| N-demethyl-175-J | Acidic Crop | 0.01 | 104 | 96–114 | 5.6 | 17 |
| | | 1.0 | 102 | 97–107 | 3.1 | 18 |
| | | 0.01–1.0 | 103 | 96–114 | 4.5 | 35 |

| Analyte | Matrix | Fortification Level (mg/kg) | Recovery Rate (%) | | RSD (%) | n |
|---|---|---|---|---|---|---|
| | | | mean | range | | |
| N-demethyl-175-L | Acidic Crop | 0.01 | 100 | 87–113 | 7.8 | 17 |
| | | 1.0 | 101 | 96–107 | 2.9 | 18 |
| | | 0.01–1.0 | 100 | 87–113 | 5.8 | 35 |
| N-formyl-175-J | Acidic Crop | 0.01 | 97 | 76–118 | 12.4 | 17 |
| | | 1.0 | 103 | 94–118 | 6.0 | 18 |
| | | 0.01–1.0 | 100 | 76–118 | 9.8 | 35 |
| N-formyl-175-L | Acidic Crop | 0.01 | 94 | 86–107 | 7.4 | 17 |
| | | 1.0 | 97 | 91–107 | 4.2 | 18 |
| | | 0.01–1.0 | 95 | 86–107 | 6.1 | 35 |
| XDE-175-J | Oily Crop [d] | 0.01 | 94 | 84–105 | 7.4 | 10 |
| | | 1.0 | 96 | 89–102 | 4.9 | 10 |
| | | 0.01–1.0 | 95 | 84–105 | 6.2 | 20 |
| XDE-175-L | Oily Crop | 0.01 | 99 | 90–110 | 6.3 | 10 |
| | | 1.0 | 101 | 95–107 | 4.7 | 10 |
| | | 0.01–1.0 | 100 | 90–110 | 5.5 | 20 |
| N-demethyl-175-J | Oily Crop | 0.01 | 97 | 86–105 | 6.2 | 10 |
| | | 1.0 | 103 | 93–111 | 5.1 | 10 |
| | | 0.01–1.0 | 100 | 86–111 | 6.3 | 20 |
| N-demethyl-175-L | Oily Crop | 0.01 | 90 | 83–101 | 6.4 | 10 |
| | | 1.0 | 98 | 92–102 | 3.4 | 10 |
| | | 0.01–1.0 | 94 | 83–102 | 6.2 | 20 |
| N-formyl-175-J | Oily Crop | 0.01 | 103 | 82–120 | 11.2 | 10 |
| | | 1.0 | 115 | 102–140 | 9.8 | 10 |
| | | 0.01–1.0 | 109 | 82–140 | 11.7 | 20 |
| N-formyl-175-L | Oily Crop | 0.01 | 105 | 63–138 | 19.7 | 10 |
| | | 1.0 | 111 | 76–139 | 15.4 | 10 |
| | | 0.01–1.0 | 108 | 63–139 | 17.3 | 20 |

[a] "Wet crop" includes: broccoli, cabbage, grape, green beans (succulent), leek, lettuce, onion, sweet peppers, and tomato.

[b] "Dry crop" includes: barley grain, forage and straw, grass forage, maize grain forage and stover, and wheat grain, forage and straw.

[c] "Acidic crop" includes: apple, cherry (without seed), lemon, orange (whole fruit, peel and pulp), peach (without seed), pear and plum (without seed).

[d] "Oily crop" canola seed, cotton seed, olive (without seed), olive oil and soya bean.

Methods GRM 05.03 and GRM 05.04 are considered to be suitable for the determination of residues of spinetoram and its metabolites in commodities of plant origin. The methods were fully validated for a range of crops and crop types, with a limit of quantification (LOQ) of 0.01 mg/kg for all crops. Method GRM 05.03 has been designated by EPA as an appropriate enforcement method for analysis of residues of spinetoram and its metabolites in plant matrices.

*Analytical Methods for Determination of Residues in Foods and Feedstuffs of Animal Origin Used in Supervised Trials*

Methods GRM 05.15 and GRM 06.08 were developed for the determination of residues of spinetoram and its metabolites in bovine and poultry tissues, milk, cream, and eggs, using HPLC with positive ion electron-spray (ESI) tandem mass spectrometry (LC-MS/MS).

*Method GRM 05.15*

Method GRM 05.15 was developed for the quantitative determination of XDE-175-J and XDE-175-L and their metabolites N-demethyl-175-J, N-demethyl-175-L, 3′-*O*-deethyl-175-J and 3′-*O*-deethyl-175-L in bovine tissues (muscle, kidney, liver and fat), milk (whole and skim) and cream, and poultry tissues (muscle, liver and fat) and poultry eggs (Shackelford and Hastings, 2005).

Residues of spinetoram and its metabolites are extracted from the samples of animal origin by homogenizing and shaking with an acetonitrile/water solution (80:20, v/v). The samples are filtered through a 0.45 µm membrane GHP filter. A stable isotope internal standard solution containing XDE-175-J, XDE-175-L, and metabolites N-demethyl-175-J and N-demethyl-175-L is added to each sample. Spinetoram and its metabolites are analysed without sample cleanup by liquid chromatography with positive-ion electron-spray ionization (ESI) tandem mass spectrometry (LC-MS/MS).

The method was validated over the concentration range of 0.01-15 mg/kg for bovine and poultry muscle and bovine kidney; 0.01-50 mg/kg for bovine and poultry liver, bovine milk and cream and poultry eggs; and 0.01-150 mg/kg for bovine and poultry fat (Shackelford, 2005; Report 051022). The validated limit of quantification (LOQ) for all of the animal matrices was 0.01 mg/kg. Recoveries of spinetoram and its metabolites in animal matrices fortified at various levels are summarized in Table 16**.** Average recovery ranged between 87% and 119%, with relative standard deviations of ≤ 18.2% for all analytes and matrices tested.

Table 16 Procedural recovery for spinetoram and its metabolites in various animal matrices, using Method GRM 05.15 (Shackelford, 2005)

| Analyte | Matrix | Fortification Level (mg/kg) | Recovery Rate (%) | | RSD (%) | n |
|---|---|---|---|---|---|---|
| | | | mean | range | | |
| XDE-175-J | Muscle (bovine & poultry) | 0.010 | 108 | 101–115 | 4.3 | 12 |
| | | 1.0 | 98 | 93–102 | 3.2 | 10 |
| | | 15 | 100 | 93–106 | 4.6 | 10 |
| | | 0.010–15 | 103 | 93–115 | 6.0 | 32 |
| XDE-175-L | Muscle (bovine & poultry) | 0.010 | 100 | 92–114 | 6.5 | 12 |
| | | 1.0 | 92 | 89–95 | 2.1 | 10 |
| | | 15 | 97 | 92–102 | 3.7 | 10 |
| | | 0.010–15 | 97 | 89–114 | 5.7 | 32 |
| *N*-demethyl-175-J | Muscle (bovine & poultry) | 0.010 | 111 | 106–117 | 2.7 | 12 |
| | | 1.0 | 103 | 99–108 | 3.1 | 10 |
| | | 15 | 105 | 96–112 | 5.6 | 10 |
| | | 0.010–15 | 106 | 96–117 | 5.0 | 32 |
| *N*-demethyl-175-L | Muscle (bovine & poultry) | 0.010 | 101 | 92–110 | 6.2 | 12 |
| | | 1.0 | 100 | 95–104 | 2.4 | 10 |
| | | 15 | 108 | 96–116 | 7.4 | 10 |
| | | 0.010–15 | 103 | 92–116 | 6.6 | 32 |
| 3′-*O*-deethyl-175-J | Muscle (bovine & poultry) | 0.010 | 113 | 103–118 | 4.9 | 12 |
| | | 1.0 | 93 | 90–99 | 3.4 | 10 |
| | | 15 | 102 | 96–109 | 3.9 | 10 |
| | | 0.010–15 | 103 | 90–118 | 9.0 | 32 |
| 3′-*O*-deethyl-175-L | Muscle (bovine & poultry) | 0.010 | 119 | 115–125 | 2.0 | 12 |
| | | 1.0 | 91 | 87–93 | 2.2 | 10 |
| | | 15 | 104 | 97–109 | 4.2 | 10 |
| | | 0.010–15 | 105 | 87–125 | 11.3 | 32 |

| Analyte | Matrix | Fortification Level (mg/kg) | Recovery Rate (%) | | RSD (%) | n |
|---|---|---|---|---|---|---|
| | | | mean | range | | |
| XDE-175-J | Liver (bovine & poultry) | 0.010 | 102 | 84–117 | 9.6 | 12 |
| | | 1.0 | 100 | 91–108 | 6.2 | 10 |
| | | 15 | 103 | 100–109 | 3.1 | 10 |
| | | 50 | 99 | 84–111 | 11.7 | 10 |
| | | 0.010–50 | 101 | 84–117 | 8.2 | 42 |
| XDE-175-L | Liver (bovine & poultry) | 0.010 | 104 | 83–119 | 11.5 | 12 |
| | | 1.0 | 93 | 87–99 | 4.7 | 10 |
| | | 15 | 98 | 93–101 | 2.5 | 10 |
| | | 50 | 93 | 81–103 | 9.9 | 10 |
| | | 0.010–50 | 97 | 81–119 | 9.3 | 42 |
| *N*-demethyl-175-J | Liver (bovine & poultry) | 0.010 | 108 | 99–118 | 4.3 | 12 |
| | | 1.0 | 97 | 92–102 | 3.2 | 10 |
| | | 15 | 102 | 97–114 | 5.4 | 10 |
| | | 50 | 97 | 80–112 | 14.6 | 10 |
| | | 0.010–50 | 101 | 80–118 | 8.9 | 42 |
| *N*-demethyl-175-L | Liver (bovine & poultry) | 0.010 | 103 | 95–115 | 6.7 | 12 |
| | | 1.0 | 104 | 93–119 | 8.2 | 10 |
| | | 15 | 103 | 97–116 | 6.9 | 10 |
| | | 50 | 98 | 80–113 | 15.9 | 10 |
| | | 0.010–50 | 102 | 80–119 | 9.7 | 42 |
| 3′-*O*-deethyl-175-J | Liver (bovine & poultry) | 0.010 | 112 | 101–120 | 5.4 | 12 |
| | | 1.0 | 103 | 93–118 | 8.6 | 10 |
| | | 15 | 106 | 95–116 | 6.7 | 10 |
| | | 50 | 100 | 86–114 | 11.0 | 10 |
| | | 0.010–50 | 106 | 86–120 | 8.7 | 42 |
| 3′-*O*-deethyl-175-L | Liver (bovine & poultry) | 0.010 | 110 | 104–116 | 4.4 | 12 |
| | | 1.0 | 93 | 85–100 | 5.7 | 10 |
| | | 15 | 102 | 91–115 | 8.1 | 10 |
| | | 50 | 101 | 85–116 | 11.4 | 10 |
| | | 0.010–50 | 102 | 85–116 | 9.6 | 42 |
| XDE-175-J | Kidney (bovine) | 0.010 | 101 | 91–112 | 7.1 | 6 |
| | | 1.0 | 90 | 88–92 | 2.1 | 5 |
| | | 15 | 97 | 95–100 | 2.3 | 5 |
| | | 0.010–15 | 96 | 88–112 | 6.4 | 16 |
| XDE-175-L | Kidney (bovine) | 0.010 | 103 | 97–109 | 4.6 | 6 |
| | | 1.0 | 87 | 84–93 | 4.2 | 5 |
| | | 15 | 91 | 87–96 | 4.6 | 5 |
| | | 0.010–15 | 94 | 84–109 | 8.4 | 16 |
| *N*-demethyl-175-J | Kidney (bovine) | 0.010 | 111 | 103–116 | 4.4 | 6 |
| | | 1.0 | 105 | 100–112 | 5.3 | 5 |
| | | 15 | 110 | 99–119 | 8.3 | 5 |
| | | 0.010–15 | 109 | 99–119 | 6.2 | 16 |

| Analyte | Matrix | Fortification Level (mg/kg) | Recovery Rate (%) | | RSD (%) | n |
|---|---|---|---|---|---|---|
| | | | mean | range | | |
| *N*-demethyl-175-L | Kidney (bovine) | 0.010 | 108 | 102–116 | 4.8 | 6 |
| | | 1.0 | 104 | 101–110 | 3.9 | 5 |
| | | 15 | 112 | 102–119 | 7.3 | 5 |
| | | 0.010–15 | 108 | 101–119 | 6.0 | 16 |
| 3′-*O*-deethyl-175-J | Kidney (bovine) | 0.010 | 107 | 104–112 | 3.1 | 6 |
| | | 1.0 | 93 | 90–96 | 3.2 | 5 |
| | | 15 | 100 | 94–104 | 4.7 | 5 |
| | | 0.010–15 | 100 | 90–112 | 7.0 | 16 |
| 3′-*O*-deethyl-175-L | Kidney (bovine) | 0.010 | 106 | 90–119 | 11.9 | 6 |
| | | 1.0 | 88 | 84–92 | 3.6 | 5 |
| | | 15.0 | 99 | 94–102 | 3.7 | 5 |
| | | 0.010–15.0 | 98 | 84–119 | 11.3 | 16 |
| XDE-175-J | Fat (bovine & poultry) | 0.01 | 105 | 87–119 | 8.2 | 14 |
| | | 1.0 | 101 | 92–109 | 5.2 | 10 |
| | | 15 | 105 | 102–110 | 2.3 | 10 |
| | | 150 | 95 | 89–101 | 3.9 | 10 |
| | | 0.010–150 | 102 | 87–119 | 6.8 | 44 |
| XDE-175-L | Fat (bovine & poultry) | 0.01 | 100 | 88–114 | 7.2 | 14 |
| | | 1.0 | 92 | 88–96 | 2.9 | 10 |
| | | 15 | 98 | 94–101 | 1.7 | 10 |
| | | 150 | 93 | 88–97 | 3.2 | 10 |
| | | 0.010–150 | 96 | 88–114 | 5.8 | 44 |
| *N*-demethyl-175-J | Fat (bovine & poultry) | 0.01 | 110 | 102–117 | 3.6 | 14 |
| | | 1.0 | 101 | 95–109 | 4.5 | 10 |
| | | 15 | 100 | 94–111 | 6.0 | 10 |
| | | 150 | 99 | 95–104 | 2.8 | 10 |
| | | 0.010–150 | 103 | 94–117 | 6.0 | 44 |
| *N*-demethyl-175-L | Fat (bovine & poultry) | 0.01 | 102 | 89–118 | 8.6 | 14 |
| | | 1.0 | 103 | 100–108 | 2.5 | 10 |
| | | 15 | 102 | 93–118 | 7.5 | 10 |
| | | 150 | 101 | 97–106 | 2.8 | 10 |
| | | 0.010–150 | 102 | 89–118 | 6.1 | 44 |
| 3′-*O*-deethyl-175-J | Fat (bovine & poultry) | 0.01 | 109 | 91–118 | 8.0 | 14 |
| | | 1.0 | 94 | 86–100 | 4.5 | 10 |
| | | 15 | 103 | 99–110 | 2.8 | 10 |
| | | 150 | 94 | 89–100 | 3.4 | 10 |
| | | 0.010–150 | 101 | 86–118 | 8.6 | 44 |
| 3′-*O*-deethyl-175-L | Fat (bovine & poultry) | 0.01 | 111 | 102–119 | 5.4 | 14 |
| | | 1.0 | 93 | 89–97 | 2.6 | 10 |
| | | 15 | 105 | 100–113 | 3.1 | 10 |
| | | 150 | 94 | 89–99 | 3.6 | 10 |
| | | 0.010–150 | 102 | 89–119 | 8.8 | 44 |

| Analyte | Matrix | Fortification Level (mg/kg) | Recovery Rate (%) | | RSD (%) | n |
|---|---|---|---|---|---|---|
| | | | mean | range | | |
| XDE-175-J | Milk | 0.010 | 100 | 92–109 | 6.2 | 12 |
| | | 1.0 | 95 | 86–104 | 6.5 | 10 |
| | | 15 | 99 | 88–107 | 6.1 | 10 |
| | | 50 | 93 | 82–110 | 13.7 | 10 |
| | | 0.010–50 | 97 | 82–110 | 8.7 | 42 |
| XDE-175-L | Milk | 0.010 | 103 | 93–115 | 5.9 | 12 |
| | | 1.0 | 96 | 92–103 | 3.5 | 10 |
| | | 15 | 95 | 86–99 | 4.2 | 10 |
| | | 50 | 88 | 79–102 | 11.6 | 10 |
| | | 0.010–50 | 96 | 79–115 | 8.7 | 42 |
| *N*-demethyl-175-J | Milk | 0.010 | 114 | 105–119 | 3.9 | 12 |
| | | 1.0 | 99 | 95–107 | 3.7 | 10 |
| | | 15 | 103 | 95–109 | 4.7 | 10 |
| | | 50 | 94 | 80–115 | 16.9 | 10 |
| | | 0.010–50 | 103 | 80–119 | 10.9 | 42 |
| *N*-demethyl-175-L | Milk | 0.010 | 108 | 96–117 | 6.5 | 12 |
| | | 1.0 | 102 | 98–109 | 3.8 | 10 |
| | | 15 | 107 | 99–114 | 5.6 | 10 |
| | | 50 | 95 | 80–116 | 17.3 | 10 |
| | | 0.010–50 | 103 | 80–117 | 10.3 | 42 |
| 3′-*O*-deethyl-175-J | Milk | 0.010 | 107 | 96–114 | 6.4 | 12 |
| | | 1.0 | 93 | 86–99 | 4.5 | 10 |
| | | 15 | 98 | 83–107 | 7.6 | 10 |
| | | 50 | 93 | 83–108 | 11.6 | 10 |
| | | 0.010–50 | 98 | 83–114 | 9.6 | 42 |
| 3′-*O*-deethyl-175-L | Milk | 0.010 | 110 | 98–115 | 4.6 | 12 |
| | | 1.0 | 92 | 86–102 | 5.0 | 10 |
| | | 15 | 99 | 84–106 | 6.9 | 10 |
| | | 50 | 92 | 81–105 | 11.0 | 10 |
| | | 0.010–50 | 99 | 81–115 | 10.1 | 42 |
| XDE-175-J | Egg | 0.010 | 100 | 92–113 | 8.8 | 6 |
| | | 1.0 | 96 | 93–102 | 4.2 | 4 |
| | | 15 | 100 | 98–102 | 1.5 | 5 |
| | | 50 | 96 | 82–106 | 12.0 | 5 |
| | | 0.010–50 | 99 | 82–113 | 7.6 | 20 |
| XDE-175-L | Egg | 0.010 | 98 | 95–103 | 3.0 | 6 |
| | | 1.0 | 92 | 88–95 | 3.4 | 4 |
| | | 15 | 96 | 94–97 | 1.0 | 5 |
| | | 50 | 90 | 78–97 | 10.1 | 5 |
| | | 0.010–50 | 94 | 78–103 | 6.1 | 20 |
| *N*-demethyl-175-J | Egg | 0.010 | 108 | 97–116 | 6.6 | 6 |
| | | 1.0 | 100 | 99–103 | 1.9 | 4 |
| | | 15 | 103 | 97–113 | 7.1 | 5 |
| | | 50 | 102 | 79–116 | 16.6 | 5 |

| Analyte | Matrix | Fortification Level (mg/kg) | Recovery Rate (%) | | RSD (%) | n |
|---|---|---|---|---|---|---|
| | | | mean | range | | |
| | | 0.010–50 | 104 | 79–116 | 9.4 | 20 |
| *N*-demethyl-175-L | Egg | 0.010 | 95 | 83–108 | 11.7 | 6 |
| | | 1.0 | 104 | 102–105 | 1.1 | 4 |
| | | 15 | 106 | 100–115 | 7.5 | 5 |
| | | 50 | 103 | 78–119 | 18.2 | 5 |
| | | 0.010–50 | 101 | 78–119 | 11.7 | 20 |
| 3′-*O*-deethyl-175-J | Egg | 0.010 | 104 | 96–111 | 5.7 | 6 |
| | | 1.0 | 91 | 90–92 | 1.1 | 4 |
| | | 15 | 99 | 97–102 | 2.6 | 5 |
| | | 50 | 92 | 78–101 | 11.3 | 5 |
| | | 0.010–50 | 97 | 78–111 | 8.2 | 20 |
| 3′-*O*-deethyl-175-L | Egg | 0.010 | 108 | 100–117 | 5.8 | 6 |
| | | 1.0 | 90 | 89–92 | 1.3 | 4 |
| | | 15 | 98 | 94–102 | 3.7 | 5 |
| | | 50 | 93 | 78–102 | 11.6 | 5 |
| | | 0.010–50 | 98 | 78–117 | 9.6 | 20 |

An independent validation was carried out at PTRL Europe by analysts having no previous experience with the residue method (Richter, 2005, Report 050049). Bovine milk (high water content) and poultry muscle were used as the representative matrices. Control bovine milk samples and control poultry muscle samples that were fortified at 0.01 mg/kg (at LOQ) and 15 mg/kg (1500× the LOQ) were analysed by liquid chromatography with positive-ion electron-spray ionization (ESI) tandem mass spectrometry (LC-MS/MS).

Table 17summarizes the validation data for spinetoram and its metabolites in bovine and poultry tissues. Average recovery ranged between 85% and 109%, with relative standard deviations of ≤ 12 %.

Table 17 Procedural recovery obtained by an independent laboratory validation of Method GRM 05.15

| Analyte | Matrix | Fortification Level (mg/kg) | Recovery Rate (%) | | RSD (%) | n |
|---|---|---|---|---|---|---|
| | | | mean | range | | |
| XDE-175-J | Bovine Milk | 0.010 | 103 | 97–108 | 4 | 5 |
| | | 15 | 91 | 87–99 | 5 | 5 |
| | | 0.010–15 | 97 | 87–108 | 8 | 10 |
| XDE-175-L | Bovine Milk | 0.010 | 105 | 99–109 | 4 | 5 |
| | | 15 | 89 | 84–98 | 6 | 5 |
| | | 0.010–15 | 97 | 84–109 | 10 | 10 |
| *N*-demethyl-175-J | Bovine Milk | 0.010 | 93 | 89–102 | 6 | 5 |
| | | 15 | 89 | 82–94 | 7 | 5 |
| | | 0.010–15 | 91 | 82–102 | 6 | 10 |
| *N*-demethyl-175-L | Bovine Milk | 0.010 | 85 | 72–97 | 12 | 5 |
| | | 15 | 93 | 81–107 | 11 | 5 |
| 3'-*O*-deethyl-175-J | Bovine Milk | 0.010 | 109 | 91–118 | 10 | 5 |
| | | 15 | 100 | 90–105 | 6 | 5 |
| | | 0.010–15 | 105 | 90–118 | 9 | 10 |

| Analyte | Matrix | Fortification Level (mg/kg) | Recovery Rate (%) | | RSD (%) | n |
|---|---|---|---|---|---|---|
| | | | mean | range | | |
| 3'-*O*-deethyl-175-L | Bovine Milk | 0.010 | 108 | 100–115 | 6 | 5 |
| | | 15 | 92 | 86–99 | 5 | 5 |
| | | 0.010–15 | 100 | 86–115 | 10 | 10 |
| XDE-175-J | Poultry Muscle | 0.010 | 105 | 94–117 | 8 | 5 |
| | | 15 | 101 | 98–105 | 3 | 5 |
| | | 0.010–15 | 103 | 94–117 | 6 | 10 |
| XDE-175-L | Poultry Muscle | 0.010 | 103 | 97–113 | 7 | 5 |
| | | 15 | 101 | 96–105 | 3 | 5 |
| | | 0.010–15 | 102 | 96–113 | 5 | 10 |
| N-demethyl-175-J | Poultry Muscle | 0.010 | 107 | 102–117 | 6 | 5 |
| | | 15 | 94 | 90–98 | 3 | 5 |
| | | 0.010–15 | 101 | 90–117 | 8 | 10 |
| N-demethyl-175-J | Poultry Muscle | 0.010 | 98 | 88–112 | 10 | 5 |
| | | 15 | 98 | 87–109 | 10 | 5 |
| | | 0.010–15 | 98 | 87–112 | 9 | 10 |
| 3'-O-deethyl-175-J | Poultry Muscle | 0.010 | 98 | 91–119 | 12 | 5 |
| | | 15 | 93 | 90–98 | 5 | 5 |
| | | 0.010–15 | 95 | 90–119 | 9 | 10 |
| 3'-O-deethyl-175-J | Poultry Muscle | 0.010 | 109 | 100–113 | 5 | 5 |
| | | 15 | 101 | 94–107 | 5 | 5 |
| | | 0.010–15 | 105 | 94–113 | 6 | 10 |

*Method GRM 06.08*

Method GRM 06.08 supersedes method GRM 05.15. Initially method GRM 05.15 was developed to include the determination of metabolites N-demethyl-175-J, N-demethyl-175-L, 3′-*O*-deethyl-175-J and 3′-*O*-deethyl-175-L. Method GRM 06.08 was developed to only include the determination of N-demethyl-175-J and N-formyl-175-J (Shackelford, *et. al.,* 2007).

Residues of spinetoram and its metabolites are extracted from the animal matrices by homogenizing and by shaking with an acetonitrile/water solution (80:20, v/v). A stable isotope internal standard solution containing XDE-175-J, XDE-175-L, and metabolites N-demethyl-175-J and N-demethyl-175-L is added to each sample. Samples are analysed by direct injection of the extraction solution without further cleanup. Determination of spinetoram and its metabolites is by liquid chromatography with positive ion electron-spray ionization (ESI) tandem mass spectrometry (LC-MS/MS).

Method GRM 06.08 is identical to method GRM 05.15, except in slight differences in the metabolites determined. Method GRM 06.08 was validated for the quantitative determination of XDE-175-J and XDE-175-L and the major metabolites of XDE-175-J, i.e., N-demethyl-175-J and N-formyl-175-J, in bovine tissues (muscle, kidney, liver and fat), milk (whole and skim) and cream, and poultry tissues (muscle, liver and fat) and poultry eggs (Shackelford, 2007, Report 061023). With the exception of metabolite N-formyl-175-J, the method was validated over the concentration range of 0.01–15 mg/kg in bovine and poultry muscle and bovine kidney; 0.01–50 mg/kg for bovine and poultry liver, bovine milk and cream and poultry eggs; and 0.01-150 mg/kg for bovine and poultry fat.

For N-formyl-175-J, the method was validated over the concentration range of 0.01-0.10 mg/kg for all bovine matrices and over the concentration range of 0.01–5.0 mg/kg for all poultry matrices. The validated limit of quantification (LOQ) for all of the animal matrices was

0.01 mg/kg. Recoveries of spinetoram and its metabolites in bovine and poultry tissues are summarized Table 18**.** Average recovery ranged between 83% and 118%, with relative standard deviations of ≤ 17% for all analytes and matrices tested.

Table 18 Procedural recovery for spinetoram and its metabolites in various animal matrices, using Method 06.08 (Shackelford, 2007)

| Analyte | Matrix | Fortification Level (mg/kg) | Recovery Rate (%) | | RSD (%) | n |
|---|---|---|---|---|---|---|
| | | | mean | range | | |
| XDE-175-J | Muscle (bovine & poultry) | 0.010 | 99 | 73–117 | 13.9 | 22 |
| | | 0.10 | 90 | 85–94 | 3.2 | 10 |
| | | 1.0 | 98 | 93–102 | 3.4 | 10 |
| | | 15 | 100 | 93–106 | 4.7 | 10 |
| | | 0.010–15 | 97 | 73–117 | 10.2 | 52 |
| XDE-175-L | Muscle (bovine & poultry) | 0.010 | 90 | 68–114 | 13.3 | 22 |
| | | 0.10 | 86 | 82–89 | 2.3 | 10 |
| | | 1.0 | 92 | 89–95 | 2.2 | 10 |
| | | 15 | 97 | 92–102 | 3.7 | 10 |
| | | 0.010–15 | 91 | 68–114 | 9.6 | 52 |
| *N*-demethyl-175-J | Muscle (bovine & poultry) | 0.010 | 106 | 90–117 | 6.6 | 22 |
| | | 0.10 | 111 | 107–117 | 2.8 | 10 |
| | | 1.0 | 103 | 99–108 | 3.1 | 10 |
| | | 15 | 105 | 96–112 | 5.6 | 10 |
| | | 0.010–15 | 106 | 90–117 | 5.7 | 52 |
| *N*-formyl-175-J | Muscle (bovine & poultry) | 0.010 | 89 | 77–109 | 9.7 | 24 |
| | | 0.10 | 88 | 79–107 | 7.9 | 20 |
| | | 5.0 | 105 | 100–110 | 3.2 | 6 |
| | | 0.010–5.0 | 90 | 77–110 | 10.2 | 50 |
| XDE-175-J | Liver (bovine & poultry) | 0.010 | 103 | 84–117 | 9.6 | 12 |
| | | 1.0 | 100 | 91–108 | 6.2 | 10 |
| | | 15 | 103 | 100–109 | 3.0 | 10 |
| | | 50 | 99 | 84–111 | 11.8 | 10 |
| | | 0.010–50 | 101 | 84–117 | 8.3 | 42 |
| XDE-175-L | Liver (bovine & poultry) | 0.010 | 104 | 83–119 | 11.5 | 12 |
| | | 1.0 | 94 | 87–99 | 4.7 | 10 |
| | | 15 | 98 | 93–101 | 2.6 | 10 |
| | | 50 | 93 | 81–103 | 10.0 | 10 |
| | | 0.010–50 | 98 | 81–119 | 9.3 | 42 |
| *N*-demethyl-175-J | Liver (bovine & poultry) | 0.010 | 108 | 99–118 | 4.4 | 12 |
| | | 1.0 | 97 | 92–102 | 3.3 | 10 |
| | | 15 | 102 | 97–114 | 5.5 | 10 |
| | | 50 | 98 | 80–112 | 14.8 | 10 |
| | | 0.010–50 | 101 | 80–118 | 9.0 | 42 |
| *N*-formyl-175-J | Liver (bovine & poultry) | 0.010 | 89 | 72–107 | 13.1 | 14 |
| | | 0.10 | 91 | 80–102 | 7.8 | 10 |
| | | 5.0 | 107 | 103–114 | 3.7 | 6 |
| | | 0.010–5.0 | 93 | 72–114 | 12.4 | 30 |

| Analyte | Matrix | Fortification Level (mg/kg) | Recovery Rate (%) | | RSD (%) | n |
|---|---|---|---|---|---|---|
| | | | mean | range | | |
| XDE-175-J | Kidney (bovine) | 0.010 | 101 | 91–112 | 7.0 | 6 |
| | | 1.0 | 91 | 88–92 | 1.8 | 5 |
| | | 15.0 | 97 | 95–100 | 2.2 | 5 |
| | | 0.010–15.0 | 96 | 88–112 | 6.3 | 16 |
| XDE-175-L | Kidney (bovine) | 0.010 | 103 | 97–109 | 4.6 | 6 |
| | | 1.0 | 87 | 84–93 | 4.3 | 5 |
| | | 15 | 91 | 87–96 | 4.7 | 5 |
| | | 0.010–15 | 95 | 84–109 | 8.5 | 16 |
| *N*-demethyl-175-J | Kidney (bovine) | 0.010 | 111 | 103–116 | 4.4 | 6 |
| | | 1.0 | 105 | 100–112 | 5.4 | 5 |
| | | 15 | 110 | 99–119 | 8.5 | 5 |
| | | 0.010–15 | 109 | 99–119 | 6.2 | 16 |
| *N*-formyl-175-J | Kidney (bovine) | 0.010 | 83 | 75–103 | 12.6 | 8 |
| | | 0.10 | 86 | 80–91 | 5.3 | 4 |
| | | 0.010–0.10 | 84 | 75–103 | 10.4 | 12 |
| XDE-175-J | Fat (bovine & poultry) | 0.01 | 105 | 87–119 | 8.0 | 14 |
| | | 1.0 | 101 | 92–109 | 5.1 | 10 |
| | | 15 | 105 | 102–110 | 2.3 | 10 |
| | | 150 | 95 | 89–101 | 3.9 | 10 |
| | | 0.010–150 | 102 | 87–119 | 6.7 | 44 |
| XDE-175-L | Fat (bovine & poultry) | 0.01 | 100 | 88–114 | 7.1 | 14 |
| | | 1.0 | 92 | 88–96 | 2.9 | 10 |
| | | 15 | 98 | 94–101 | 1.9 | 10 |
| | | 150 | 93 | 88–97 | 3.1 | 10 |
| | | 0.010–150 | 96 | 88–114 | 5.8 | 44 |
| *N*-demethyl-175-J | Fat (bovine & poultry) | 0.01 | 110 | 102–117 | 3.7 | 14 |
| | | 1.0 | 101 | 95–109 | 4.6 | 10 |
| | | 15 | 101 | 94–111 | 6.0 | 10 |
| | | 150 | 99 | 95–104 | 2.9 | 10 |
| | | 0.010–150 | 103 | 94–117 | 6.1 | 44 |
| *N*-formyl-175-J | Fat (bovine & poultry) | 0.01 | 94 | 79–122 | 9.3 | 42 |
| | | 0.10 | 96 | 84–109 | 6.5 | 24 |
| | | 5.0 | 113 | 105–121 | 5.4 | 6 |
| | | 0.010–5.0 | 96 | 79–122 | 9.6 | 72 |
| XDE-175-J | Milk and Cream | 0.010 | 100 | 92–109 | 6.1 | 12 |
| | | 1.0 | 95 | 86–104 | 6.6 | 10 |
| | | 15 | 99 | 88–107 | 6.2 | 10 |
| | | 50 | 93 | 82–110 | 13.6 | 10 |
| | | 0.010–50 | 97 | 82–110 | 8.7 | 42 |
| XDE-175-L | Milk and Cream | 0.010 | 103 | 93–115 | 5.9 | 12 |
| | | 1.0 | 96 | 92–103 | 3.4 | 10 |
| | | 15 | 95 | 86–99 | 4.3 | 10 |
| | | 50 | 88 | 79–102 | 11.6 | 10 |
| | | 0.010–50 | 96 | 79–115 | 8.6 | 42 |

| Analyte | Matrix | Fortification Level (mg/kg) | Recovery Rate (%) | | RSD (%) | n |
|---|---|---|---|---|---|---|
| | | | mean | range | | |
| *N*-demethyl-175-J | Milk and Cream | 0.010 | 114 | 105–119 | 3.9 | 12 |
| | | 1.0 | 99 | 95–107 | 3.7 | 10 |
| | | 15 | 103 | 95–109 | 4.7 | 10 |
| | | 50 | 94 | 80–115 | 17.0 | 10 |
| | | 0.010–50 | 103 | 80–119 | 11.0 | 42 |
| *N*-formyl-175-J | Milk and Cream | 0.010 | 88 | 67–100 | 7.8 | 38 |
| | | 0.10 | 92 | 81–104 | 6.4 | 18 |
| | | 1.0 | 91 | 87–94 | NA | 2 |
| | | 0.010–1.0 | 89 | 67–104 | 7.5 | 58 |
| XDE-175-J | Egg | 0.010 | 101 | 92–113 | 8.8 | 6 |
| | | 1.0 | 96 | 93–102 | 4.2 | 4 |
| | | 15 | 100 | 98–102 | 1.7 | 5 |
| | | 50 | 96 | 82–106 | 12.0 | 5 |
| | | 0.010–50 | 99 | 82–113 | 7.6 | 20 |
| XDE-175-L | Egg | 0.010 | 98 | 95–103 | 3.1 | 6 |
| | | 1.0 | 92 | 88–95 | 3.4 | 4 |
| | | 15 | 96 | 94–97 | 1.3 | 5 |
| | | 50 | 89 | 78–97 | 10.2 | 5 |
| | | 0.010–50 | 94 | 78–103 | 6.3 | 20 |
| *N*-demethyl-175-J | Egg | 0.010 | 108 | 97–116 | 6.5 | 6 |
| | | 1.0 | 101 | 99–103 | 1.9 | 4 |
| | | 15 | 104 | 97–113 | 7.1 | 5 |
| | | 50 | 102 | 79–116 | 16.6 | 5 |
| | | 0.010–50 | 104 | 79–116 | 9.3 | 20 |
| *N*-formyl-175-J | Egg | 0.010 | 93 | 74–111 | 15.5 | 6 |
| | | 0.10 | 92 | 88–96 | 4.1 | 6 |
| | | 5.0 | 106 | 98–111 | 5.2 | 6 |
| | | 0.010–5.0 | 97 | 74–111 | 11.1 | 18 |

Methods GRM 05.15 and GRM 06.08 are considered to be suitable for the determination of spinetoram and its metabolites in commodities of animal origin. The methods were fully validated for a range of animal matrices, with a limit of quantification (LOQ) of 0.01 mg/kg for all matrices. Method GRM 06.08 has been designated by EPA as an appropriate enforcement method for analysis of residues of spinetoram and its metabolites in animal matrices.

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

The Meeting received information on frozen storage stability of residues of spinetoram and its metabolites in wheat, soya bean, orange, lettuce and sugar beet samples.

*Commodities of plant origin*

A storage stability study was conducted with XDE-175 and metabolites in agricultural commodities to determine the stability of the residues in homogenized samples while stored frozen (Wendelburg, 2006; Report 050027.01). Wheat grain, soya bean seed, orange whole fruit, lettuce leaf, and sugar beet root were selected to represent each of five crop groupings (dry, oily, acidic, wet, and root crops) to obtain information on a wide variety of crops. Samples taken from trial studies (report 040063 and 040051) were used.

The stability of the compounds, XDE-175-J, XDE-175-L, N-demethyl-175-J, N-demethyl-175-L, N-formyl-175-J, and N-formyl-175-L, were investigated for twelve months. All samples were weighed into individual high density polyethylene (HDPE) containers, fortified at 0.10 mg/kg and stored in a temperature-monitored freezer set at approximately –20 °C. Samples were analysed at 0, 63, 90, 231, and 372 days of frozen storage after fortification, by LC-MS/MS following method GRM 05.04.

The efficiency of the analytical method was determined at the time of analysis of each sampling event by creating two concurrent recovery samples and analysing them according to the method above. Concurrent recovery results confirmed the suitability of the method for determination of spinetoram and its metabolites in the agricultural commodities selected.

Results of the storage stability tests are summarized in Table 19. The results show that spinetoram and its metabolites are stable for at least 12 months in agricultural commodities, except lettuce, under frozen storage at approximately –20 °C. In lettuce, remaining XDE-175-L and N-dimethyl-175-L were 60 and 65% respectively (unadjusted for procedural recovery) on 372 days after initiation of the study.

Table 19 Stability of spinetoram and specific metabolites in agricultural commodities at fortification level of 0.10 mg/kg following storage at –20 °C (Wendelburg, 2006)

| Commodity | Storage interval (days) | % Remaining [a] | Concurrent recovery (%) |
|---|---|---|---|
| XDE-175-J | | | |
| Wheat Grain | 0 | 104 | 103 |
| | 63 | 119 | 119 |
| | 90 | 95 | 110 |
| | 231 | 102 | 102 |
| | 372 | 99 | 109 |
| Soya bean Seed | 0 | 105 | 108 |
| | 63 | 97 | 95 |
| | 90 | 98 | 110 |
| | 231 | 103 | 96 |
| | 372 | 95 | 92 |
| Orange Fruit | 0 | 101 | 102 |
| | 63 | 99 | 104 |
| | 90 | 95 | 106 |
| | 231 | 100 | 98 |
| | 372 | 98 | 97 |
| Lettuce Leaf | 0 | 90 | 96 |
| | 63 | 91 | 89 |
| | 90 | 95 | 91 |
| | 231 | 85 | 87 |
| | 372 | 87 | 96 |
| Sugar Beet Root | 0 | 100 | 98 |
| | 63 | 105 | 103 |
| | 90 | 96 | 106 |
| | 231 | 98 | 101 |
| | 372 | 87 | 99 |

| Commodity | Storage interval (days) | % Remaining [a] | Concurrent recovery (%) |
|---|---|---|---|
| XDE-175-L | | | |
| Wheat Grain | 0 | 107 | 104 |
| | 63 | 98 | 100 |
| | 90 | 108 | 111 |
| | 231 | 95 | 103 |
| | 372 | 91 | 98 |
| Soya bean Seed | 0 | 101 | 108 |
| | 63 | 100 | 85 |
| | 90 | 104 | 108 |
| | 231 | 92 | 99 |
| | 372 | 84 | 89 |
| Orange Fruit | 0 | 98 | 96 |
| | 63 | 88 | 94 |
| | 90 | 102 | 104 |
| | 231 | 92 | 96 |
| | 372 | 86 | 88 |
| Lettuce Leaf | 0 | 88 | 89 |
| | 63 | 87 | 76 |
| | 90 | 105 | 105 |
| | 231 | 73 | 77 |
| | 372 | 60 | 81 |
| Sugar Beet Root | 0 | 100 | 102 |
| | 63 | 88 | 91 |
| | 90 | 100 | 105 |
| | 231 | 100 | 93 |
| | 372 | 111 | 102 |
| N-demethyl-175-J | | | |
| Wheat Grain | 0 | 106 | 116 |
| | 63 | 96 | 95 |
| | 90 | 103 | 114 |
| | 231 | 97 | 101 |
| | 372 | 89 | 101 |
| Soya bean Seed | 0 | 100 | 106 |
| | 63 | 97 | 89 |
| | 90 | 96 | 104 |
| | 231 | 92 | 93 |
| | 372 | 86 | 87 |
| Orange Fruit | 0 | 99 | 99 |
| | 63 | 96 | 95 |
| | 90 | 94 | 100 |
| | 231 | 91 | 95 |
| | 372 | 83 | 84 |

| Commodity | Storage interval (days) | % Remaining [a] | Concurrent recovery (%) |
|---|---|---|---|
| Lettuce Leaf | 0 | 94 | 109 |
| | 63 | 90 | 98 |
| | 90 | 91 | 102 |
| | 231 | 84 | 102 |
| | 372 | 80 | 95 |
| Sugar Beet Root | 0 | 98 | 96 |
| | 63 | 97 | 93 |
| | 90 | 89 | 102 |
| | 231 | 88 | 91 |
| | 372 | 83 | 90 |
| N-demethyl-175-L | | | |
| Wheat Grain | 0 | 103 | 105 |
| | 63 | 85 | 90 |
| | 90 | 98 | 106 |
| | 231 | 90 | 98 |
| | 372 | 80 | 94 |
| Soya bean Seed | 0 | 101 | 107 |
| | 63 | 89 | 95 |
| | 90 | 96 | 102 |
| | 231 | 87 | 93 |
| | 372 | 85 | 88 |
| Orange Fruit | 0 | 100 | 102 |
| | 63 | 88 | 93 |
| | 90 | 88 | 97 |
| | 231 | 91 | 95 |
| | 372 | 84 | 87 |
| Lettuce Leaf | 0 | 93 | 104 |
| | 63 | 91 | 95 |
| | 90 | 90 | 99 |
| | 231 | 77 | 93 |
| | 372 | 65 | 89 |
| Sugar Beet Root | 0 | 100 | 102 |
| | 63 | 88 | 94 |
| | 90 | 87 | 102 |
| | 231 | 88 | 95 |
| | 372 | 80 | 95 |
| N-formyl-175-J | | | |
| Wheat Grain | 0 | 133 | 141 |
| | 63 | 83 | 82 |
| | 90 | 90 | 105 |
| | 231 | 83 | 88 |
| | 372 | 78 | 85 |

| Commodity | Storage interval (days) | % Remaining [a] | Concurrent recovery (%) |
|---|---|---|---|
| Soya bean Seed | 0 | 106 | 108 |
| | 63 | 102 | 87 |
| | 90 | 111 | 127 |
| | 231 | 91 | 95 |
| | 372 | 96 | 92 |
| Orange Fruit | 0 | 91 | 94 |
| | 63 | 93 | 90 |
| | 90 | 86 | 90 |
| | 231 | 90 | 88 |
| | 372 | 79 | 75 |
| Lettuce Leaf | 0 | 98 | 101 |
| | 63 | 105 | 110 |
| | 90 | 89 | 94 |
| | 231 | 90 | 85 |
| | 372 | 86 | 95 |
| Sugar Beet Root | 0 | 92 | 90 |
| | 63 | 86 | 88 |
| | 90 | 91 | 99 |
| | 231 | 77 | 102 |
| | 372 | 79 | 85 |
| N-formyl-175-L | | | |
| Wheat Grain | 0 | 99 | 106 |
| | 63 | 94 | 94 |
| | 90 | 112 | 117 |
| | 231 | 97 | 87 |
| | 372 | 79 | 96 |
| Soya bean Seed | 0 | 116 | 108 |
| | 63 | 84 | 88 |
| | 90 | 129 | 145 |
| | 231 | 104 | 108 |
| | 372 | 98 | 90 |
| Orange Fruit | 0 | 89 | 85 |
| | 63 | 78 | 96 |
| | 90 | 94 | 101 |
| | 231 | 90 | 90 |
| | 372 | 84 | 75 |
| Lettuce Leaf | 0 | 80 | 99 |
| | 63 | 75 | 75 |
| | 90 | 102 | 102 |
| | 231 | 86 | 84 |
| | 372 | 86 | 91 |

| Commodity | Storage interval (days) | % Remaining [a] | Concurrent recovery (%) |
|---|---|---|---|
| Sugar Beet Root | 0 | 82 | 83 |
| | 63 | 89 | 79 |
| | 90 | 93 | 101 |
| | 231 | 93 | 104 |
| | 372 | 77 | 87 |

[a] Mean of three subsamples.

*Commodities of animal origin*

Samples of animal tissues, milk, and eggs from the metabolism and feeding studies were analysed within 20 days of sample collection in the supervised trials. Therefore, storage stability tests were not conducted.

## USE PATTERN

The Meeting received approved labels from Canada and the USA.

Information on registered formulations, applications methods and dosage rates of spinetoram for uses on the crops for which supervised trial data were provided is summarized in Table 20. Unless otherwise noted, each of the following GAPs are for field use and all applications are foliar applications. Other than the crops for which supervised trial data were available to the Meeting, spinetoram is approved for use on asparagus, banana, beet greens, Brassica vegetables, bulb vegetables, bush berries, caneberries, cereals, corn (field and sweet), cotton, cranberry, cucurbits, fig, grape, herbs, legume vegetables, mint, peanuts, pulses, soya bean, strawberry, tropical fruits and turnip greens in these countries.

Table 20 Registered uses of spinetoram

| Crop | Country | Formulation | | Application | | | | | PHI days |
|---|---|---|---|---|---|---|---|---|---|
| | | g ai/L or g ai/kg | type | Method | Rate g ai/ha | Rate g ai/hL | Min. interval days | Max. no./ g ai/ season | |
| Apricot | Canada | 250 | WG | Foliar | 53–105 | | 7 | 3 | 14 |
| Apricot | USA | 250 | WG | Foliar | 53–123 | | 7 | 4 / (490)[a] | 14 |
| Cherry | Canada | 250 | WG | Foliar | 53–105 | | 7 | 3 | 7 |
| Cherry | USA | 250 | WG | Foliar | 53–123 | | 3 | 4 / (490)[a] | 7 |
| Citrus fruits | USA | 250 | WG | Foliar | 53–105 | | 7 | 3 / (210)[a] | 1 |
| Fruiting vegetables | Canada | 250 | WG | Foliar | 35–50 | | 5 | 3 | 1 |
| Fruiting vegetables | USA | 120 | SC | Foliar | 44–88 | | 4 | 6 / (298)[a] | 1 |
| Leaf of root, tuber & legume vegetables | USA | 120 | SC | Foliar | 44–88 | | 7 | 6 / (298) [a] | 3 |
| Leafy vegetables | Canada | 250 | WG | Foliar | 35–50 | | 5 | 3 | 1 |
| Leafy vegetables | USA | 120 | SC | Foliar | 44–88 | | 4 | 6 / (298) [a] | 1 |
| Nectarine | Canada | 250 | WG | Foliar | 53–105 | | 7 | 3 | 14 |
| Nectarine | USA | 250 | WG | Foliar | 53–123 | | 7 | 4 / (490)[a] | 1 |
| Peach | Canada | 250 | WG | Foliar | 53–105 | | 7 | 3 | 14 |

| Crop | Country | Formulation | | Application | | | | | PHI days |
|---|---|---|---|---|---|---|---|---|---|
| | | g ai/L or g ai/kg | type | Method | Rate g ai/ha | Rate g ai/hL | Min. interval days | Max. no./ g ai/ season | |
| Peach | USA | 250 | WG | Foliar | 53–123 | | 7 | 4 / (490)[a] | 14 |
| Plums, prunes | Canada | 250 | WG | Foliar | 53–105 | | 7 | 3 | 7 |
| Plums, prunes | USA | 250 | WG | Foliar | 53–123 | | 3 | 4 / (490)[a] | 7 |
| Pome fruits | Canada | 250 | WG | Foliar | 53–105 | | 7 | 3 | 7 |
| Pome fruits | New Zealand | 250 | WG | Foliar | 50 | 2.5 | 14 | 4 | 7 |
| Pome fruits | USA | 250 | WG | Foliar | 79–123 | | 7 | 4 / (490)[a] | 7 |
| Root vegetables (carrot, horse radish, radish, rutabaga, turnip) | Canada | 250 | WG | Foliar | 35–50 | | 5 | 3 | 3 |
| Root vegetables (potato, sugar beet, etc) | USA | 120 | SC | Foliar | 53–70 | | 7 | 4 / (281) [a] | 7 |
| Root vegetables (radish, turnip) | USA | 120 | SC | Foliar | 53–70 | | 4 | 3 / (210)[a] | 3 |
| Tree nuts | USA | 250 | WG | Foliar | 26–123 | | 7 | 4 / (490)[a] | 14 |

[a] Total application per season or year.

## RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

The Meeting received information on supervised field trials of spinetoram on the following crops:

| Commodity | Group | Table No. |
|---|---|---|
| Oranges | Citrus | Table 21 |
| Apple | Pome fruits | Table 22 |
| Apple | Pome fruits | Table 23 |
| Apple | Pome fruits | Table 24 |
| Pear | Pome fruits | Table 25 |
| Cherry | Stone fruit | Table 26 |
| Peach | Stone fruit | Table 27 |
| Nectarine | Stone fruit | Table 28 |
| Apricot | Stone fruit | Table 29 |
| Tomatoes | Fruiting vegetables, Other than Cucurbits | Table 30 |
| Leafy lettuce | Leafy vegetables | Table 31 |
| Sugarbeet | Root and tuber vegetables | Table 32 |
| Almonds | Tree nuts | Table 33 |
| Pecan nut | Tree nuts | Table 34 |
| Sugar beet leaves and tops | Animal feedstuffs | Table 35 |
| Almond hulls | Animal feedstuffs | Table 36 |

Trials were generally well documented with full laboratory and field reports. Laboratory reports included method validation, with batch recoveries at residue levels similar to those occurring in samples from the supervised trials. Dates of analyses or duration of residue sample storage were also provided. In general, data on procedural recoveries were within the acceptable range 70–120%, with relative standard deviation of < 20%. In addition, frozen storage periods from harvest to analysis of all samples were within those confirmed to be stable through separate storage stability determinations.

Field reports provide data on the dates of spray applications, methods used and sampling dates. Although trials included control plots, but no control data are recorded in the tables below unless residues in control samples exceeded the LOQ.

The residue concentrations are reported for XDE-175-J, XDE-175-L, N-demethyl-175-J and N-formyl-175-J. They are unadjusted for procedural recovery. Where residues were below the limit of detection (0.003 mg/kg in US and Canadian trials and 0.05 mg/kg in trials in Australia and New Zealand), they are expressed as "ND". Where they are below the limit of quantification (0.01 mg/kg) and at or above the limit of detection, they are expressed as "< 0.01".

Total residues for estimation of maximum residue levels were calculated by summing up the concentrations of XDE-175-J and XDE-175-L. XDE-175-J was the primary residue reflecting higher ratio in spinetoram formulations. The method for calculation of the total residues is illustrated below. In the calculation, "ND" is treated in the same manner as "< 0.01".

| XDE-175-J | XDE-175-L | Total |
|---|---|---|
| mg/kg | | |
| < 0.01 | < 0.01 | < 0.01 |
| 0.05 | < 0.01 | 0.05 |
| 0.06 | 0.02 | 0.08 |

Total residues for estimation of STMRs were calculated by summing up the concentrations of XDE-175-J, XDE-175-L, *N*-demethyl-175-J and *N*-formyl-175-J. In most trials, XDE-175-J was the primary residue at shorter PHIs while, in many cases, *N*-formyl-175-J was found at higher concentrations than XDE-175-J at longer PHIs. On the other hand, XDE-175-L, with its concentration being one third of that of XDE-175-J, was a minor component in the four compounds. *N*-demethyl-J was in most cases at lower concentrations than XDE-175-J and in a number of trials was not determined. The method for calculation of the total residues taking the above into account is illustrated below. In the calculation, "ND" is treated in the same manner as "< 0.01".

| XDE-175-J | XDE-175-L | *N*-demethyl-175-J | *N*-formyl-175-J | Total |
|---|---|---|---|---|
| mg/kg | | | | |
| < 0.01 | < 0.01 | < 0.01 | < 0.01 | < 0.02 |
| 0.05 | < 0.01 | < 0.01 | < 0.01 | 0.06 |
| < 0.01 | < 0.01 | < 0.01 | 0.05 | 0.06 |
| 0.05 | < 0.01 | < 0.01 | 0.05 | 0.10 |
| 0.06 | 0.02 | 0.02 | 0.06 | 0.16 |

In all the US trials, duplicate samples were taken and analysed. In accordance with the 2007 JMPR's decision, the higher value of the two was used for estimating maximum residue level.

Total values of XDE-175-J and XDE-175-L residues from the trials conducted according to the GAP have been underlined and used for the estimation of maximum residue levels. Corresponding total values of XDE-175-J, XDE-175-L, N-demethyl-175-J and N-formyl-175-J residues were used for estimation of STMRs and they are double underlined.

*Orange*

Twelve supervised trials on oranges were conducted in commercial citrus growing areas in the US in 2004 and 2007 (Dolder and Wendelberg, 2005; Report 040063; McKellar, 2008; Report ARAP 07D-001).

Trials were conducted in six sites, each site consisting of a control plot and 2 treated plots. Trial plots had a minimum of 6 orange trees, with samples taken from the 4 inner trees. In each site, one plot was treated using a low spray volume (700 L/ha) and the other, a high spray volume (approximately 3300 L/ha). Each treated plot received three applications of a suspension concentrate (SC) formulation containing 100 g/L of spinetoram at the nominal rate of 70 g ai/ha or a seasonal total of 210 g ai/ha. The applications were made at 4-day intervals and mature fruits were collected at the PHI of 1 day. In one of each of the low volume and high volume spray trials, samples were also taken on days 0, 3, 7, and 14 after the last application.

Approximately 4.5 kg of mature oranges were collected by hand. At each sampling, a single composite sample was taken from the control plot while duplicate composite samples were taken independently from each treated plot. Samples were frozen within 4 h of collection, placed in plastic bags, labelled, shipped frozen, and kept in temperature-monitored freezers at –20 °C until analysis.

Spinetoram and the metabolites were analysed using the method GRM 05.04 with an LOQ of 0.01 mg/kg and LOD of 0.003 mg/kg. Samples from the 2008 trials were analysed using method GRM 05.03, with the same LOQ and LOD values as GRM 05.04.

Table 21-1 Residues of spinetoram from supervised trials on orange in the USA (for estimation of maximum residue level)

| Location, year (Variety) | Form | Application | | | Total/ season, g ai/ha | PHI, days | Residue, mg/kg | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | Total [a] | |
| GAP, USA Citrus fruits | SC or WG | | 53–105 | 3 | 210 | 1 | | | | |
| Foliar application using low spray volume (~700 L/ha) | | | | | | | | | | |
| Deleon Springs, FL, 2004 (Valencia) | SC | 10 | 70–72 | 3 | 213 | 1 | 0.030 | < 0.01 | 0.030 | 040063 |
| | | | | | | | 0.028 | < 0.01 | 0.028 | |
| Mount Dora, FL, 2004 (Valencia) | SC | 11 | 71–72 | 3 | 214 | 1 | 0.011 | ND | 0.011 | 040063 |
| | | | | | | | 0.022 | < 0.01 | 0.022 | |
| Raymondville, TX, 2004 (N-33) | SC | 10 | 70–72 | 3 | 213 | 1 | < 0.01 | ND | < 0.01 | 040063 |
| | | | | | | | < 0.01 | ND | < 0.01 | |
| Richgrove, CA, 2004 (Olinda) | SC | 9 | 70–71 | 3 | 211 | 1 | < 0.01 | ND | < 0.01 | 040063 |
| | | | | | | | < 0.01 | ND | < 0.01 | |
| Porterville,CA, 2004 (Cutter Valencia) | SC | 9 | 70 | 3 | 210 | 0 | 0.011 | ND | 0.011 | 040063 |
| | | | | | | | 0.014 | < 0.01 | 0.014 | |
| | | | | | | 1 | 0.012 | < 0.01 | 0.012 | |
| | | | | | | | 0.010 | ND | 0.010 | |
| | | | | | | 3 | < 0.01 | ND | < 0.01 | |
| | | | | | | | 0.011 | ND | 0.011 | |
| | | | | | | 7 | < 0.01 | ND | < 0.01 | |
| | | | | | | | < 0.01 | ND | < 0.01 | |
| | | | | | | 14 | < 0.01 | ND | < 0.01 | |
| | | | | | | | < 0.01 | ND | < 0.01 | |
| FL, 2007 (Not specified) | WG | 11 | 69- 72 | 3 | 212 | 1 | 0.028 | < 0.01 | 0.028 | ARAP 07D-001 |
| | | | | | | | 0.025 | ND | 0.025 | |
| Foliar application using high spray volume (~3300 L/ha) | | | | | | | | | | |
| Deleon Springs, | SC | 2 | 70-71 | 3 | 212 | 1 | 0.015 | < 0.01 | 0.015 | 040063 |

| Location, year (Variety) | Form | Application | | | Total/ season, g ai/ha | PHI, days | Residue, mg/kg | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | Total [a] | |
| GAP, USA Citrus fruits | SC or WG | | 53–105 | 3 | 210 | 1 | | | | |
| FL, 2004 (Valencia) | | | | | | | 0.014 | ND | 0.014 | |
| Mount Dora, FL, 2004 (Valencia) | SC | 2 | 70-71 | 3 | 212 | 1 | 0.017 | < 0.01 | 0.017 | 040063 |
| | | | | | | | 0.018 | < 0.01 | 0.018 | |
| Raymondville, TX, 2004 (N-33) | SC | 2 | 70-71 | 3 | 211 | 1 | < 0.01 | ND | < 0.01 | 040063 |
| | | | | | | | ND | ND | < 0.01 | |
| Richgrove, CA, 2004 (Olinda) | SC | 2 | 70-71 | 3 | 211 | 1 | 0.021 | < 0.01 | 0.021 | 040063 |
| | | | | | | | 0.020 | < 0.01 | 0.020 | |
| Porterville, CA, 2004 (Cutter Valencia) | SC | 2 | 70 | 3 | 210 | 0 | 0.021 | < 0.01 | 0.021 | 040063 |
| | | | | | | | 0.012 | < 0.01 | 0.012 | |
| | | | | | | 1 | 0.010 | ND | 0.010 | |
| | | | | | | | 0.012 | ND | 0.012 | |
| | | | | | | 3 | < 0.01 | ND | < 0.01 | |
| | | | | | | | ND | ND | < 0.01 | |
| | | | | | | 7 | < 0.01 | ND | < 0.01 | |
| | | | | | | | < 0.01 | ND | < 0.01 | |
| | | | | | | 14 | < 0.01 | ND | < 0.01 | |
| | | | | | | | < 0.01 | ND | < 0.01 | |
| FL, USA, 2007 (Not specified) | WG | 2 | 69- 70 | 3 | 209 | 1 | 0.021 | < 0.01 | 0.021 | ARAP 07D-001 |
| | | | | | | | 0.024 | < 0.01 | 0.024 | |

[a] Total residues = XDE-175-J + XDE-175-L

Table 21-2 Residues of spinetoram and metabolites from supervised trials on orange in the USA (for estimation of STMR)

| Location, year (Variety) | Form | Application | | | Total/ season, g ai/ha | PHI, days | Residue, mg/kg | | | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | ND-J | NF-J | Total [a] | |
| GAP, USA Citrus fruits | SC or WG | | 53-105 | 3 | 210 | 1 | | | | | | |
| Foliar application using low spray volume (~700 L/ha) | | | | | | | | | | | | |
| Deleon Springs, FL, 2004 (Valencia) | SC | 10 | 70-72 | 3 | 213 | 1 | 0.030 | < 0.01 | 0.011 | 0.016 | 0.057 | 040063 |
| | | | | | | | 0.028 | < 0.01 | 0.014 | 0.024 | 0.066 | |
| Mount Dora, FL, 2004 (Valencia) | SC | 11 | 71-72 | 3 | 214 | 1 | 0.011 | ND | < 0.01 | < 0.01 | 0.021 | 040063 |
| | | | | | | | 0.022 | < 0.01 | 0.012 | 0.017 | 0.051 | |
| Raymondville, TX, 2004 (N-33) | SC | 10 | 70-72 | 3 | 213 | 1 | < 0.01 | ND | < 0.01 | 0.012 | 0.022 | 040063 |
| | | | | | | | < 0.01 | ND | < 0.01 | 0.011 | 0.021 | |
| Richgrove, | SC | 9 | 70- | 3 | 211 | 1 | < 0.01 | ND | ND | ND | < 0.02 | 040063 |

| Location, year (Variety) | Form | Application | | | Total/ season, g ai/ha | PHI, days | Residue, mg/kg | | | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | ND-J | NF-J | Total [a] | |
| GAP, USA Citrus fruits | SC or WG | | 53-105 | 3 | 210 | 1 | | | | | | |
| CA, 2004 (Olinda) | | | 71 | | | | < 0.01 | ND | ND | < 0.01 | < 0.02 | |
| Porterville,CA, 2004 (Cutter Valencia) | SC | 9 | 70 | 3 | 210 | 0 | 0.011 | ND | < 0.01 | 0.014 | 0.025 | 040063 |
| | | | | | | | 0.014 | < 0.01 | < 0.01 | 0.012 | 0.026 | |
| | | | | | | 1 | 0.012 | < 0.01 | ND | 0.015 | 0.027 | |
| | | | | | | | 0.010 | ND | < 0.01 | 0.020 | 0.030 | |
| | | | | | | 3 | < 0.01 | ND | ND | < 0.01 | < 0.02 | |
| | | | | | | | 0.011 | ND | < 0.01 | 0.022 | 0.033 | |
| | | | | | | 7 | < 0.01 | ND | ND | 0.015 | 0.025 | |
| | | | | | | | < 0.01 | ND | ND | < 0.01 | < 0.02 | |
| | | | | | | 14 | < 0.01 | ND | ND | 0.013 | 0.023 | |
| | | | | | | | < 0.01 | ND | ND | 0.015 | 0.025 | |
| FL, 2007 (Not specified) | WG | 11 | 69-72 | 3 | 212 | 1 | 0.028 | < 0.01 | 0.013 | < 0.01 | 0.051 | ARAP 07D-001 |
| | | | | | | | 0.025 | ND | < 0.01 | ND | 0.035 | |
| Foliar application using high spray volume (~3300 L/ha) | | | | | | | | | | | | |
| Deleon Springs, FL, 2004 (Valencia) | SC | 2 | 70-71 | 3 | 212 | 1 | 0.015 | < 0.01 | < 0.01 | 0.026 | 0.041 | 040063 |
| | | | | | | | 0.014 | ND | < 0.01 | 0.024 | 0.038 | |
| Mount Dora, FL, 2004 (Valencia) | SC | 2 | 70-71 | 3 | 212 | 1 | 0.017 | < 0.01 | < 0.01 | 0.022 | 0.039 | 040063 |
| | | | | | | | 0.018 | < 0.01 | < 0.01 | 0.021 | 0.039 | |
| Raymondville, TX, 2004 (N-33) | SC | 2 | 70-71 | 3 | 211 | 1 | < 0.01 | ND | < 0.01 | < 0.01 | < 0.02 | 040063 |
| | | | | | | | ND | ND | ND | < 0.01 | < 0.02 | |
| Richgrove, CA, 2004 (Olinda) | SC | 2 | 70-71 | 3 | 211 | 1 | 0.021 | < 0.01 | < 0.01 | 0.032 | 0.053 | 040063 |
| | | | | | | | 0.020 | < 0.01 | < 0.01 | 0.049 | 0.069 | |
| Porterville, CA, 2004 (Cutter Valencia) | SC | 2 | 70 | 3 | 210 | 0 | 0.021 | < 0.01 | < 0.01 | 0.038 | 0.059 | 040063 |
| | | | | | | | 0.012 | < 0.01 | < 0.01 | 0.026 | 0.038 | |
| | | | | | | 1 | 0.010 | ND | < 0.01 | 0.035 | 0.045 | |
| | | | | | | | 0.012 | ND | < 0.01 | 0.035 | 0.047 | |
| | | | | | | 3 | < 0.01 | ND | ND | 0.019 | 0.029 | |
| | | | | | | | ND | ND | ND | < 0.01 | < 0.02 | |
| | | | | | | 7 | < 0.01 | ND | ND | 0.026 | 0.036 | |
| | | | | | | | < 0.01 | ND | ND | 0.026 | 0.036 | |
| | | | | | | 14 | < 0.01 | ND | ND | 0.023 | 0.033 | |
| | | | | | | | < 0.01 | ND | ND | 0.020 | 0.03 | |
| FL, USA, 2007 (Not specified) | WG | 2 | 69-70 | 3 | 209 | 1 | 0.021 | < 0.01 | 0.010 | < 0.01 | 0.041 | ARAP 07D-001 |
| | | | | | | | 0.024 | < 0.01 | 0.012 | < 0.01 | 0.046 | |

[a] Total residues = XDE-175-J + XDE-175-L + ND-J + NF-J

*Pome Fruits*

Supervised trials on apples were conducted in Australia, Canada, New Zealand, and the USA during 2004-2007. Trials on pears were also conducted in Australia and New Zealand during 2005/2006 season.

*Supervised trials on apple in the USA*

Supervised trials on apples were conducted in major apple growing areas in the USA in 2004 and 2007 (Dolder and Wendelburg, 2005; Report 040063; McKellar, 2008; Report ARAP 07D-001).

Twelve trials were carried out in six different sites, with ten trials conducted in 2004 and two conducted in 2007. Each site consisted of one control plot and two treated plots. Each treated plot had a minimum of 6 apple trees, with samples taken from the four inner trees. In each site, one plot was treated using low spray volume application (~700 L/ha) and the other, a high volume spray (~3300 L/ha). Five applications of a formulation (100 g/L SC or 250 g/kg WG) were made to each treated plot using tractor-mounted airblast sprayers at a nominal rate of 100 g ai/ha or a total seasonal rate of 500 g ai/ha. Applications were made at intervals of about 28 days and mature fruits were harvested at the PHI of 7 days. In one of each of the low volume and high volume spray trials, samples were also taken on days 0, 1, 3, 7, and 14 after the last application.

Approximately 2.6 kg of apples were harvested by hand on each sampling date. A single composite sample was taken from the control plot while duplicate composite samples were taken independently from each treated plot. Samples were frozen within 4 h of collection, placed in plastic bags, labelled, shipped frozen, and kept in temperature-monitored freezers at –20 °C until analysis.

Spinetoram and the metabolites were analysed using the method GRM 05.04 with an LOQ and LOD of 0.01 mg/kg and 0.003 mg/kg, respectively.

Table 22-1 Residues of spinetoram from supervised trials on apples in the USA (for estimation of maximum residue level)

| Location, year (Variety) | Form | Application | | | Total/ season, g ai/ha | PHI, days | Residue, mg/kg | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | Total [a] | |
| GAP, USA Pome fruits | SC or WG | | 79-123 | 4 | 490 | 7 | | | | |
| Foliar application using low spray volume (~700 L/ha) | | | | | | | | | | |
| North Rose, NY, 2004 (Rome) | SC | 14 | 100 | 5 | 500 | 7 | < 0.01 | ND | < 0.01 | 040063 |
| | | | | | | | < 0.01 | ND | < 0.01 | |
| Blairsville, GA, 2004 (Red Rome) | SC | 14 | 99-103 | 5 | 505 | 7 | 0.011 | ND | 0.011 | 040063 |
| | | | | | | | 0.013 | ND | 0.013 | |
| Paynesville, MN, 2004 (Honey Crisp) | SC | 15 | 101-102 | 5 | 508 | 7 | ND | ND | < 0.01 | 040063 |
| | | | | | | | ND | ND | < 0.01 | |
| Orosi, CA, 2004 (Granny Smith) | SC | 13 | 97-101 | 5 | 494 | 7 | ND | ND | < 0.01 | 040063 |
| | | | | | | | < 0.01 | ND | < 0.01 | |
| Ephrata, WA, 2004 (Red Delicious) | SC | 14 | 100 | 5 | 500 | 0 | 0.048 | 0.014 | 0.062 | 040063 |
| | | | | | | | 0.044 | 0.013 | 0.057 | |
| | | | | | | 1 | 0.019 | < 0.01 | 0.019 | |
| | | | | | | | 0.027 | < 0.01 | 0.027 | |
| | | | | | | 3 | 0.014 | ND | 0.014 | |

| Location, year (Variety) | Form | Application | | | Total/ season, g ai/ha | PHI, days | Residue, mg/kg | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | Total [a] | |
| GAP, USA Pome fruits | SC or WG | | 79-123 | 4 | 490 | 7 | | | | |
| | | | | | | | 0.019 | < 0.01 | 0.019 | |
| | | | | | | 7 | 0.013 | ND | 0.013 | |
| | | | | | | | < 0.01 | ND | < 0.01 | |
| | | | | | | 14 | < 0.01 | ND | < 0.01 | |
| | | | | | | | < 0.01 | ND | < 0.01 | |
| MI, 2007 (Not specified) | WG | 16 | 99-101 | 5 | 501 | 7 | 0.028 | ND | 0.028 | ARAP 07D-001 |
| | | | | | | | 0.023 | ND | 0.023 | |
| Foliar application using high spray volume (~3300 L/ha) | | | | | | | | | | |
| North Rose, NY, 2004 (Rome) | SC | 4 | 98-100 | 5 | 498 | 7 | < 0.01 | ND | < 0.01 | 040063 |
| | | | | | | | < 0.01 | ND | < 0.01 | |
| Blairsville, GA, 2004 (Red Rome) | SC | 3 | 100-103 | 5 | 509 | 7 | 0.012 | ND | 0.012 | 040063 |
| | | | | | | | 0.010 | ND | 0.010 | |
| Paynesville, MN, 2004 (Honey Crisp) | SC | 3 | 101-102 | 5 | 506 | 7 | ND | ND | < 0.01 | 040063 |
| | | | | | | | ND | ND | < 0.01 | |
| Orosi, CA, 2004 (Granny Smith) | SC | 3 | 98-100 | 5 | 498 | 7 | < 0.01 | ND | < 0.01 | 040063 |
| | | | | | | | < 0.01 | ND | < 0.01 | |
| Ephrata, WA, 2004 (Red Delicious) | SC | 3 | 99-100 | 5 | 498 | 0 | 0.041 | 0.011 | 0.052 | 040063 |
| | | | | | | | 0.045 | 0.012 | 0.057 | |
| | | | | | | 1 | 0.015 | ND | 0.015 | |
| | | | | | | | 0.012 | ND | 0.012 | |
| | | | | | | 3 | 0.013 | ND | 0.013 | |
| | | | | | | | 0.011 | ND | 0.011 | |
| | | | | | | 7 | < 0.01 | ND | < 0.01 | |
| | | | | | | | < 0.01 | ND | < 0.01 | |
| | | | | | | 14 | < 0.01 | ND | < 0.01 | |
| | | | | | | | < 0.01 | ND | < 0.01 | |
| MI, 2007 (Not specified) | WG | 3 | 96-101 | 5 | 498 | 7 | 0.020 | ND | 0.020 | ARAP 07D-001 |
| | | | | | | | 0.020 | ND | 0.020 | |

[a] Total residue = XDE-175-J + XDE-175-L.

Table 22-2 Residues of spinetoram and metabolites from supervised trials on apples in the USA (for estimation of STMR)

| Location, year (Variety) | Form | Application | | | Total/ season, g ai/ha | PHI, days | Residue, mg/kg | | | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | ND-J | NF-J | Total [a] | |
| GAP, USA Pome fruits | SC or WG | | 79-123 | 4 | 490 | 7 | | | | | | |

| Location, year (Variety) | Form | Application | | | Total/ season, g ai/ha | PHI, days | Residue, mg/kg | | | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | ND-J | NF-J | Total [a] | |
| GAP, USA Pome fruits | SC or WG | | 79-123 | 4 | 490 | 7 | | | | | | |
| Foliar application using low spray volume (~700 L/ha) | | | | | | | | | | | | |
| North Rose, NY, 2004 (Rome) | SC | 14 | 100 | 5 | 500 | 7 | < 0.01 | ND | ND | ND | < 0.02 | 040063 |
| | | | | | | | < 0.01 | ND | ND | < 0.01 | < 0.02 | |
| Blairsville, GA, 2004 (Red Rome) | SC | 14 | 99-103 | 5 | 505 | 7 | 0.011 | ND | < 0.01 | ND | 0.021 | 040063 |
| | | | | | | | 0.013 | ND | < 0.01 | < 0.01 | 0.023 | |
| Paynesville, MN, 2004 (Honey Crisp) | SC | 15 | 101-102 | 5 | 508 | 7 | ND | ND | ND | ND | < 0.02 | 040063 |
| | | | | | | | ND | ND | ND | ND | < 0.02 | |
| Orosi, CA, 2004 (Granny Smith) | SC | 13 | 97-101 | 5 | 494 | 7 | ND | ND | ND | ND | < 0.02 | 040063 |
| | | | | | | | < 0.01 | ND | ND | ND | < 0.02 | |
| Ephrata, WA, 2004 (Red Delicious) | SC | 14 | 100 | 5 | 500 | 0 | 0.048 | 0.014 | < 0.01 | < 0.01 | 0.072 | 040063 |
| | | | | | | | 0.044 | 0.013 | < 0.01 | < 0.01 | 0.067 | |
| | | | | | | 1 | 0.019 | < 0.01 | < 0.01 | 0.010 | 0.029 | |
| | | | | | | | 0.027 | < 0.01 | 0.012 | 0.014 | 0.053 | |
| | | | | | | 3 | 0.014 | ND | < 0.01 | < 0.01 | 0.024 | |
| | | | | | | | 0.019 | < 0.01 | < 0.01 | 0.013 | 0.032 | |
| | | | | | | 7 | 0.013 | ND | 0.010 | 0.013 | 0.036 | |
| | | | | | | | < 0.01 | ND | < 0.01 | < 0.01 | < 0.02 | |
| | | | | | | 14 | < 0.01 | ND | < 0.01 | 0.010 | 0.020 | |
| | | | | | | | < 0.01 | ND | < 0.01 | < 0.01 | < 0.02 | |
| MI, 2007 (Not specified) | WG | 16 | 99-101 | 5 | 501 | 7 | 0.028 | ND | < 0.01 | 0.010 | 0.038 | ARAP 07D-001 |
| | | | | | | | 0.023 | ND | < 0.01 | 0.010 | 0.033 | |
| Foliar application using high spray volume (~3300 L/ha) | | | | | | | | | | | | |
| North Rose, NY, 2004 (Rome) | SC | 4 | 98-100 | 5 | 498 | 7 | < 0.01 | ND | ND | < 0.01 | < 0.02 | 040063 |
| | | | | | | | < 0.01 | ND | < 0.01 | 0.012 | 0.022 | |
| Blairsville, GA, 2004 (Red Rome) | SC | 3 | 100-103 | 5 | 509 | 7 | 0.012 | ND | < 0.01 | < 0.01 | 0.022 | 040063 |
| | | | | | | | 0.010 | ND | < 0.01 | < 0.01 | 0.020 | |
| Paynesville, MN, 2004 (Honey Crisp) | SC | 3 | 101-102 | 5 | 506 | 7 | ND | ND | ND | ND | < 0.02 | 040063 |
| | | | | | | | ND | ND | ND | ND | < 0.02 | |
| Orosi, CA, 2004 (Granny Smith) | SC | 3 | 98-100 | 5 | 498 | 7 | < 0.01 | ND | ND | < 0.01 | < 0.02 | 040063 |
| | | | | | | | < 0.01 | ND | ND | < 0.01 | < 0.02 | |
| Ephrata, WA, 2004 (Red | SC | 3 | 99-100 | 5 | 498 | 0 | 0.041 | 0.011 | < 0.01 | < 0.01 | 0.062 | 040063 |
| | | | | | | | 0.045 | 0.012 | < 0.01 | < 0.01 | 0.067 | |
| | | | | | | 1 | 0.015 | ND | < 0.01 | 0.017 | 0.032 | |

| Location, year (Variety) | Form | Application | | | Total/ season, g ai/ha | PHI, days | Residue, mg/kg | | | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | ND-J | NF-J | Total [a] | |
| GAP, USA Pome fruits | SC or WG | | 79-123 | 4 | 490 | 7 | | | | | | |
| Delicious) | | | | | | | 0.012 | ND | < 0.01 | 0.016 | 0.028 | |
| | | | | | | 3 | 0.013 | ND | < 0.01 | 0.017 | 0.030 | |
| | | | | | | | 0.011 | ND | < 0.01 | 0.018 | 0.029 | |
| | | | | | | 7 | < 0.01 | ND | ND | 0.012 | 0.022 | |
| | | | | | | | < 0.01 | ND | < 0.01 | 0.016 | 0.026 | |
| | | | | | | 14 | < 0.01 | ND | ND | 0.015 | 0.025 | |
| | | | | | | | < 0.01 | ND | ND | 0.010 | 0.020 | |
| MI, 2007 (Not specified) | WG | 3 | 96-101 | 5 | 498 | 7 | 0.020 | ND | < 0.01 | 0.017 | 0.037 | ARAP 07D-001 |
| | | | | | | | 0.020 | ND | < 0.01 | 0.016 | 0.036 | |

[a] Total residue = XDE-175-J + XDE-175-L + ND-J + NF-J.

*Supervised trials on apple in Canada*

Six supervised trials were conducted on apples in Canada in 2005 (Dolder and Schelle, 2006; Report 050041). Two additional trials included in the study were conducted in Idaho, USA.

A total of eight trials were carried out in four different sites, four trials in two sites in Ontario, two trials in Quebec, and two trials in Idaho. Each site consisted of one control plot and two treated plots. Three applications of a water dispersible granule formulation (WG) containing 250 g /kg spinetoram were made at two different rates, one plot receiving a seasonal rate of 240 g ai/ha and the other, 315 g ai/ha. Applications were made at 6–8-day intervals, using airblast sprayers with 5–7 nozzles, with spray volumes ranging from 916 to 993 L/ha. Mature apples were harvested 5–7 days after the last application.

A minimum of 24 fruits, weighing approximately 2.8 kg were harvested by hand. A single composite sample was taken from the control plot while duplicate composite samples were taken independently from each treated plot. Samples were frozen within 4 h of collection, placed in plastic bags, labelled, shipped frozen, and kept in temperature-monitored freezers at –20 °C until analysis.

Spinetoram and the metabolites were analysed using the method GRM 05.04 with an LOQ and LOD of 0.01 mg/kg and 0.003 mg/kg, respectively.

Table 23-1 Residues of spinetoram from supervised trials on apple in Canada (for estimation of maximum residue level)

| Location, year (Variety) | Form | Application | | | Total/ season, g ai/ha | PHI, days | Residue, mg/kg | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | Total[a] | |
| GAP, Canada | WG | | 53-105 | 3 | | 7 | | | | |
| St. George, Ontario, 2005 (Red Delicious) | WG | 8 | 79- 81 | 3 | 239 | 6 | < 0.01 | ND | < 0.01 | 050041 |
| | | | | | | | < 0.01 | ND | < 0.01 | |

| Location, year (Variety) | Form | Application | | | Total/ season, g ai/ha | PHI, days | Residue, mg/kg | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | Total[a] | |
| St. George, Ontario, 2005 (Red Delicious) | WG | 11 | 103-106 | 3 | 314 | 6 | 0.017 | < 0.01 | 0.017 | 050041 |
| | | | | | | | 0.012 | ND | 0.012 | |
| St.George, Ontario, 2005 (IdaRed) | WG | 8 | 76- 80 | 3 | 233 | 6 | 0.010 | ND | 0.010 | 050041 |
| | | | | | | | 0.010 | ND | 0.010 | |
| St.George, Ontario, 2005 (IdaRed) | WG | 11 | 99-108 | 3 | 309 | 5 | 0.011 | ND | 0.011 | 050041 |
| | | | | | | | 0.015 | ND | 0.015 | |
| S-Paul d'Abbotsford, Quebec, 2005 (McIntosh) | WG | 9 | 77- 80 | 3 | 237 | 7 | < 0.01 | ND | < 0.01 | 050041 |
| | | | | | | | < 0.01 | ND | < 0.01 | |
| S-Paul d'Abbotsford, Quebec, 2005 (McIntosh) | WG | 11 | 101-107 | 3 | 314 | 7 | < 0.01 | ND | < 0.01 | 050041 |
| | | | | | | | < 0.01 | ND | < 0.01 | |
| Rockland, Idaho, USA, 2005 (McIntosh) | WG | 8 | 78- 80 | 3 | 236 | 7 | 0.018 | < 0.01 | 0.018 | 050041 |
| | | | | | | | 0.020 | < 0.01 | 0.020 | |
| Rockland, Idaho, USA, 2005 (McIntosh) | WG | 11 | 101-103 | 3 | 306 | 7 | 0.028 | < 0.01 | 0.028 | 050041 |
| | | | | | | | 0.019 | < 0.01 | 0.019 | |

[a] Total residue = XDE-175-J + XDE-175-L.

Table 23-2. Residues of spinetoram and metabolites from supervised trials on apple in Canada (for estimation of STMR)

| Location, year (Variety) | Form | Application | | | Total/ season, g ai/ha | PHI, days | Residue, mg/kg | | | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | ND-J | NF-J | Total[a] | |
| GAP, Canada | WG | | 53-105 | 3 | | 7 | | | | | | |
| St. George, Ontario, 2005 (Red Delicious) | WG | 8 | 79-81 | 3 | 239 | 6 | < 0.01 | ND | < 0.01 | ND | < 0.02 | 050041 |
| | | | | | | | < 0.01 | ND | < 0.01 | < 0.01 | < 0.02 | |
| St. George, Ontario, 2005 (Red Delicious) | WG | 11 | 103-106 | 3 | 314 | 6 | 0.017 | < 0.01 | 0.011 | < 0.01 | 0.038 | 050041 |
| | | | | | | | 0.012 | ND | < 0.01 | < 0.01 | 0.022 | |
| St.George, Ontario, 2005 (IdaRed) | WG | 8 | 76-80 | 3 | 233 | 6 | 0.010 | ND | < 0.01 | < 0.01 | 0.020 | 050041 |
| | | | | | | | 0.010 | ND | < 0.01 | ND | 0.020 | |
| St.George, Ontario, 2005 (IdaRed) | WG | 11 | 99-108 | 3 | 309 | 5 | 0.011 | ND | < 0.01 | < 0.01 | 0.021 | 050041 |
| | | | | | | | 0.015 | ND | < 0.01 | < 0.01 | 0.025 | |

| Location, year (Variety) | Form | Application | | | Total/ season, g ai/ha | PHI, days | Residue, mg/kg | | | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | ND-J | NF-J | Total [a] | |
| GAP, Canada | WG | | 53-105 | 3 | | 7 | | | | | | |
| S-Paul d'Abbotsford, Quebec, 2005 (McIntosh) | WG | 9 | 77-80 | 3 | 237 | 7 | < 0.01 | ND | < 0.01 | ND | < 0.02 | 050041 |
| | | | | | | | < 0.01 | ND | < 0.01 | ND | < 0.02 | |
| S-Paul d'Abbotsford, Quebec, 2005 (McIntosh) | WG | 11 | 101-107 | 3 | 314 | 7 | < 0.01 | ND | < 0.01 | ND | < 0.02 | 050041 |
| | | | | | | | < 0.01 | ND | < 0.01 | ND | < 0.02 | |
| Rockland, Idaho, USA, 2005 (McIntosh) | WG | 8 | 78-80 | 3 | 236 | 7 | 0.018 | < 0.01 | < 0.01 | < 0.01 | 0.028 | 050041 |
| | | | | | | | 0.020 | < 0.01 | < 0.01 | < 0.01 | 0.030 | |
| Rockland, Idaho, USA, 2005 (McIntosh) | WG | 11 | 101-103 | 3 | 306 | 7 | 0.028 | < 0.01 | < 0.01 | < 0.01 | 0.038 | 050041 |
| | | | | | | | 0.019 | < 0.01 | < 0.01 | < 0.01 | 0.029 | |

[a] Total residue = XDE-175-J + XDE-175-L + ND-J + NF-J.

*Supervised trials on apple in Australia*

A total of 20 supervised field trials on apples and eight on pears were conducted at six sites in Australia during 2004/2005 and 2005/2006 season (Cowles, 2006; Reports 040114 and 050062).

In trials in the 2004/2005 season at one site in Australia, apple trees were treated with one or four applications of a SC formulation of spinetoram, using two treatment regimes. The first treatment schedule consisted of four applications of spinetoram formulation at 63, 49, 35 and 21 days before harvest. The second treatment schedule consisted of one application at 21 days before harvest. Treatments were made as high volume applications at rates of 2.5 g/hL, 3.75 g/hL, 5 g/hL and 7.5 g/hL of spinetoram, respectively.

During the 2005/2006 season, trials were conducted in five sites in Australia where apple plots, consisting of 1 to 6 trees per plot ranging from early flowering to full size maturing fruit, were treated with 4 or 7 applications of a WG formulation of spinetoram (250 g ai/L). Two treatment regimens were used. The first treatment schedule consisted of three applications at 5-day intervals over flowering followed by four applications at 14-day intervals commencing 63 days before harvest so that the last application was 21 days before harvest. The results show that these early season treatments had no cumulative effect on residues resulting from the 4 applications at 14-day interval sprays used later in the season. The second treatment schedule consisted of four applications at 14-day intervals commencing 63 days before commercial harvest so that the last application is 21 days before harvest. Treatments were made as foliar spray applications at rates of 5 g ai/hL and 7.5 g ai/hL of spinetoram.

Twenty-five whole apples or pears, weighing approximately 2–5 kg, were collected from each field at 0, 7, 14, 21, 28 and 35 days after the last application. Samples were collected by hand, packed in plastic bags, labelled, and transported frozen to the laboratory.

Spinetoram and the metabolites were analysed using the method GRM 05.03, with an LOQ 0.01 mg/kg. The LOD was determined to be 0.005 mg/kg.

*Supervised trials on apple and pear in New Zealand*

A total of 20 supervised field trials on apples and 8 on pears were carried out at 6 sites in New Zealand during the 2004/05 and 2005/06 seasons (Cowles, 2006; Reports 040114 and 050074). All the trials were decline trials.

Each field site consisted of one untreated and four treated plots, each with four trees (except for the trial on apple in 2004/2005, which had two trees). Each treated plot received one of four treatment rates of 2.5, 3.75, 5, and 7.5 g ai/hL (SC 100 or 120 g ai/L spinetoram) applied at intervals of 14–21 days. For the 2004/2005 trials, two application regimes were used; one using 4 applications at 60–63, 49, 36, and 21 days before harvest, and the other using a single application 21 days before harvest. The 2005/2006 trials used four applications. Mature apples were harvested 5–7 days after the last application. Applications were by hand-gun to run-off (1500–1800 L water/ha).

About 3–4 kg (12 fruits) of whole mature fruits were harvested by hand on 0, 1, 3, 7, 14, 28, and 35 or 42 days after the last application. Samples were placed in plastic bags, labelled, frozen and transported frozen to the laboratory, where they were kept in freezers at –18 °C until analysis.

Spinetoram and the metabolites were analysed using the method GRM 05.03, previously validated with an LOQ 0.01 mg/kg. The LOD was 0.005 mg/kg.

Table 24-1 Residues of spinetoram from supervised trials on apple in Australia and New Zealand (for estimation of maximum residue level)

| Location, year (Variety) | Form | Application | | | Total/ season, g ai/ha | PHI, days | Residue, mg/kg | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | Total [a] | |
| GAP, New Zealand Pome fruits | WG | 2.5 | | 4 | | 7 | | | | |
| AUSTRALIAN TRIALS, 2004/2005 SEASON | | | | | | | | | | |
| Shepparton East, VIC, 2005 (Sundowner) 040114-01 | SC | 2.5 | 37.5 | 4 | 150 | 0 | 0.051 | < 0.01 | 0.051 | 040114 |
| | | | | | | 3 | 0.021 | ND | 0.021 | |
| | | | | | | 7 | < 0.01 | ND | < 0.01 | |
| | | | | | | 14 | < 0.01 | ND | < 0.01 | |
| | | | | | | 21 | < 0.01 | ND | < 0.01 | |
| | | | | | | 28 | ND | ND | < 0.01 | |
| | | | | | | 35 | ND | ND | < 0.01 | |
| Shepparton East, VIC, 2005 (Sundowner) 040114-01 | SC | 3.75 | 56–57 | 4 | 225 | 0 | 0.120 | 0.023 | 0.143 | 040114 |
| | | | | | | 3 | 0.050 | < 0.01 | 0.05 | |
| | | | | | | 8 | 0.020 | ND | 0.02 | |
| | | | | | | 14 | 0.011 | ND | 0.011 | |
| | | | | | | 21 | < 0.01 | ND | < 0.01 | |
| | | | | | | 28 | < 0.01 | ND | < 0.01 | |
| | | | | | | 36 | ND | ND | < 0.01 | |
| Shepparton East, VIC, 2005 (Sundowner) 040114-01 | SC | 5 | 75 | 4 | 300 | 0 | 0.120 | 0.026 | 0.146 | 040114 |
| | | | | | | 3 | 0.094 | 0.012 | 0.106 | |
| | | | | | | 8 | 0.042 | ND | 0.042 | |
| | | | | | | 14 | 0.016 | ND | 0.016 | |
| | | | | | | 21 | < 0.01 | ND | < 0.01 | |
| | | | | | | 28 | < 0.01 | ND | < 0.01 | |
| | | | | | | 36 | < 0.01 | ND | < 0.01 | |

| Location, year (Variety) | Form | Application | | | Total/ season, g ai/ha | PHI, days | Residue, mg/kg | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | Total [a] | |
| Shepparton East, VIC, 2005 (Sundowner) 040114-01 | SC | 7.5 | 112–113 | 4 | 450 | 0 | 0.186 | 0.039 | 0.225 | 040114 |
| | | | | | | 3 | 0.101 | 0.017 | 0.118 | |
| | | | | | | 8 | 0.049 | ND | 0.049 | |
| | | | | | | 14 | 0.024 | ND | 0.024 | |
| | | | | | | 21 | < 0.01 | ND | < 0.01 | |
| | | | | | | 28 | 0.024 | ND | 0.024 | |
| | | | | | | 36 | < 0.01 | ND | < 0.01 | |
| Shepparton East, VIC, 2005 (Sundowner) 040114-01 | SC | 2.5 | 37.5 | 1 | 37.5 | 0 | 0.030 | < 0.01 | 0.03 | 040114 |
| | | | | | | 3 | 0.024 | ND | 0.024 | |
| | | | | | | 8 | < 0.01 | ND | < 0.01 | |
| | | | | | | 14 | ND | ND | < 0.01 | |
| | | | | | | 21 | ND | ND | < 0.01 | |
| | | | | | | 28 | ND | ND | < 0.01 | |
| | | | | | | 36 | ND | ND | < 0.01 | |
| Shepparton East, VIC, 2005 (Sundowner) 040114-01 | SC | 3.75 | 56 | 1 | 56 | 0 | 0.053 | 0.012 | 0.065 | 040114 |
| | | | | | | 3 | 0.026 | ND | 0.026 | |
| | | | | | | 8 | < 0.01 | ND | < 0.01 | |
| | | | | | | 14 | ND | ND | < 0.01 | |
| | | | | | | 21 | ND | ND | < 0.01 | |
| | | | | | | 28 | ND | ND | < 0.01 | |
| | | | | | | 36 | ND | ND | < 0.01 | |
| Shepparton East, VIC, 2005 (Sundowner) 040114-01 | SC | 5 | 75 | 1 | 75 | 0 | 0.095 | 0.022 | 0.117 | 040114 |
| | | | | | | 3 | 0.040 | < 0.01 | 0.04 | |
| | | | | | | 8 | 0.017 | ND | 0.017 | |
| | | | | | | 14 | 0.010 | ND | 0.01 | |
| | | | | | | 21 | ND | ND | < 0.01 | |
| | | | | | | 28 | ND | ND | < 0.01 | |
| | | | | | | 36 | ND | ND | < 0.01 | |
| Shepparton East, VIC, 2005 (Sundowner) 040114-01 | SC | 7.5 | 112.5 | 1 | 112.5 | 0 | 0.123 | 0.031 | 0.154 | 040114 |
| | | | | | | 3 | 0.076 | 0.014 | 0.09 | |
| | | | | | | 8 | 0.014 | ND | 0.014 | |
| | | | | | | 14 | 0.034 | ND | 0.034 | |
| | | | | | | 21 | < 0.01 | ND | < 0.01 | |
| | | | | | | 28 | < 0.01 | ND | < 0.01 | |
| | | | | | | 36 | ND | ND | < 0.01 | |
| AUSTRALIAN TRIALS, 2005/2006 SEASON | | | | | | | | | | |
| Ardmona VIC, 2006 (Granny Smith) 050062-01 | SC | 5 | 62 | 4 | 248 | 0 | 0.04 | < 0.01 | 0.04 | 050062 |
| | | | | | | 7 | < 0.01 | ND | < 0.01 | |
| | | | | | | 14 | ND | ND | < 0.01 | |
| | | | | | | 28 | ND | ND | < 0.01 | |
| | | | | | | 35 | ND | ND | < 0.01 | |

| Location, year (Variety) | Form | Application | | | Total/ season, g ai/ha | PHI, days | Residue, mg/kg | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | Total [a] | |
| Ardmona VIC, 2006 (Granny Smith) 050062-01 | SC | 7.5 | 93 | 4 | 372 | 0 | 0.06 | 0.02 | 0.08 | 050062 |
| | | | | | | 7 | 0.01 | ND | 0.01 | |
| | | | | | | 14 | ND | ND | < 0.01 | |
| | | | | | | 28 | ND | ND | < 0.01 | |
| | | | | | | 35 | ND | ND | < 0.01 | |
| Poziers, QLD, 2005-06 (Summer Del) 050062-02[2] | WG | 5 | 186–229 | 4 | 805 | 0 | 0.05 | 0.01 | 0.06 | 050062 |
| | | | | | | 7 | < 0.01 | ND | < 0.01 | |
| | | | | | | 28 | ND | ND | < 0.01 | |
| | | | | | | 35 | ND | ND | < 0.01 | |
| Poziers, QLD, 2005-06 (Summer Del) 050062-02[2] | WG | 7.5 | 278–344 | 4 | 1207 | 0 | 0.04 | 0.02 | 0.06 | 050062 |
| | | | | | | 7 | 0.03 | ND | 0.03 | |
| | | | | | | 28 | 0.01 | ND | 0.01 | |
| | | | | | | 35 | ND | ND | < 0.01 | |
| Spreyton, Tasmania, 2006 (Fuji) 050062-03 | WG | 5 | 128–149 | 4 | 551 | 0 | 0.09 | 0.01 | 0.10 | 050062 |
| | | | | | | 7 | 0.03 | ND | 0.03 | |
| | | | | | | 14 | 0.02 | ND | 0.02 | |
| | | | | | | 28 | < 0.01 | ND | < 0.01 | |
| | | | | | | 35 | ND | ND | < 0.01 | |
| Spreyton, Tasmania, 2006 (Fuji) 050062-03 | WG | 7.5 | 188-207 | 4 | 797 | 0 | 0.11 | 0.02 | 0.13 | 050062 |
| | | | | | | 7 | 0.04 | ND | 0.04 | |
| | | | | | | 14 | 0.02 | ND | 0.02 | |
| | | | | | | 28 | < 0.01 | ND | < 0.01 | |
| | | | | | | 35 | ND | ND | < 0.01 | |
| Ardmona VIC, 2006 (Granny Smith) 050062-01 | SC | 5 | 62 | 7 | 434 | 0 | 0.06 | 0.02 | 0.08 | 050062 |
| | | | | | | 7 | < 0.01 | ND | < 0.01 | |
| | | | | | | 14 | ND | ND | < 0.01 | |
| | | | | | | 28 | ND | ND | < 0.01 | |
| | | | | | | 35 | ND | ND | < 0.01 | |
| Ardmona VIC, 2006 (Granny Smith) 050062-01 | SC | 7.5 | 93 | 7 | 651 | 0 | 0.04 | 0.01 | 0.05 | 050062 |
| | | | | | | 7 | 0.01 | ND | 0.01 | |
| | | | | | | 14 | ND | ND | < 0.01 | |
| | | | | | | 28 | ND | ND | < 0.01 | |
| | | | | | | 35 | ND | ND | < 0.01 | |
| Poziers, QLD 2005-06 [b] (Summer Del) 050062-02 | WG | 5 | 65–229 | 7 | 848 | 0 | ND | ND | < 0.01 | 050062 |
| | | | | | | 35 | ND | ND | < 0.01 | |
| Poziers, QLD, 2005-06 [b] (Summer Del) 050062-02 | WG | 7.5 | 98–344 | 7 | 1549 | 0 | ND | ND | < 0.01 | 050062 |
| | | | | | | 35 | ND | ND | < 0.01 | |

| Location, year (Variety) | Form | Application | | | Total/ season, g ai/ha | PHI, days | Residue, mg/kg | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | Total [a] | |
| Spreyton, Tasmania, 2006 (Fuji) APPLE 050062-03 | WG | 5 | 70–149 | 7 | 785 | 0 | 0.05 | < 0.01 | 0.05 | 050062 |
| | | | | | | 7 | 0.03 | ND | 0.03 | |
| | | | | | | 14 | 0.02 | ND | 0.02 | |
| | | | | | | 28 | ND | ND | < 0.01 | |
| | | | | | | 35 | ND | ND | < 0.01 | |
| Spreyton, Tasmania, 2006 (Fuji) 050062-03 | WG | 7.5 | 101–207 | 7 | 1017 | 0 | 0.11 | 0.02 | 0.13 | 050062 |
| | | | | | | 7 | 0.03 | ND | 0.03 | |
| | | | | | | 14 | 0.01 | ND | 0.01 | |
| | | | | | | 28 | 0.01 | ND | 0.01 | |
| | | | | | | 35 | ND | ND | < 0.01 | |
| NEW ZEALAND TRIALS, 2004/2005 SEASON | | | | | | | | | | |
| Hort Research, 2005 (Fuji) 040114-02 | SC | 2.5 | 52–62 | 4 | 233 | 0 | 0.032 | < 0.01 | 0.032 | 040114 |
| | | | | | | 3 | < 0.01 | ND | < 0.01 | |
| | | | | | | 8 | ND | ND | < 0.01 | |
| | | | | | | 14 | ND | ND | < 0.01 | |
| | | | | | | 21 | ND | ND | < 0.01 | |
| | | | | | | 28 | ND | ND | < 0.01 | |
| | | | | | | 36 | ND | ND | < 0.01 | |
| Hort Research, 2005 (Fuji) 040114-02 | SC | 3.75 | 69–83 | 4 | 307 | 0 | 0.050 | 0.016 | 0.066 | 040114 |
| | | | | | | 3 | < 0.01 | ND | < 0.01 | |
| | | | | | | 8 | < 0.01 | ND | < 0.01 | |
| | | | | | | 14 | ND | ND | < 0.01 | |
| | | | | | | 21 | ND | ND | < 0.01 | |
| | | | | | | 28 | ND | ND | < 0.01 | |
| | | | | | | 36 | ND | ND | < 0.01 | |
| Hort Research, 2005 (Fuji) 040114-02 | SC | 5 | 122–130 | 4 | 498 | 0 | 0.068 | 0.020 | 0.088 | 040114 |
| | | | | | | 3 | 0.012 | ND | 0.012 | |
| | | | | | | 8 | < 0.01 | ND | < 0.01 | |
| | | | | | | 14 | ND | ND | < 0.01 | |
| | | | | | | 21 | ND | ND | < 0.01 | |
| | | | | | | 28 | ND | ND | < 0.01 | |
| | | | | | | 36 | ND | ND | < 0.01 | |
| Hort Research, 2005 (Fuji) 040114-02 | SC | 7.5 | 159–180 | 4 | 681 | 0 | 0.075 | 0.023 | 0.098 | 040114 |
| | | | | | | 3 | 0.025 | < 0.01 | 0.025 | |
| | | | | | | 8 | < 0.01 | ND | < 0.01 | |
| | | | | | | 14 | < 0.01 | ND | < 0.01 | |
| | | | | | | 21 | ND | ND | < 0.01 | |
| | | | | | | 28 | ND | ND | < 0.01 | |
| | | | | | | 36 | ND | ND | < 0.01 | |

| Location, year (Variety) | Form | Application | | | Total/ season, g ai/ha | PHI, days | Residue, mg/kg | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | Total [a] | |
| Hort Research, 2005 (Fuji) 040114-02 | SC | 2.5 | 62 | 1 | 62 | 0 | 0.021 | < 0.01 | 0.021 | 040114 |
| | | | | | | 3 | ND | ND | < 0.01 | |
| | | | | | | 8 | ND | ND | < 0.01 | |
| | | | | | | 14 | ND | ND | < 0.01 | |
| | | | | | | 21 | ND | ND | < 0.01 | |
| | | | | | | 28 | ND | ND | < 0.01 | |
| | | | | | | 36 | ND | ND | < 0.01 | |
| Hort Research, 2005 (Fuji) 040114-02 | SC | 3.75 | 81 | 1 | 81 | 0 | 0.046 | 0.015 | 0.061 | 040114 |
| | | | | | | 3 | 0.015 | ND | 0.015 | |
| | | | | | | 8 | < 0.01 | ND | < 0.01 | |
| | | | | | | 14 | ND | ND | < 0.01 | |
| | | | | | | 21 | ND | ND | < 0.01 | |
| | | | | | | 28 | ND | ND | < 0.01 | |
| | | | | | | 36 | ND | ND | < 0.01 | |
| Hort Research, 2005 (Fuji) 040114-02 | SC | 5 | 104 | 1 | 104 | 0 | 0.086 | 0.027 | 0.113 | 040114 |
| | | | | | | 3 | 0.016 | ND | 0.016 | |
| | | | | | | 8 | < 0.01 | ND | < 0.01 | |
| | | | | | | 14 | ND | ND | < 0.01 | |
| | | | | | | 21 | ND | ND | < 0.01 | |
| | | | | | | 28 | ND | ND | < 0.01 | |
| | | | | | | 36 | ND | ND | < 0.01 | |
| Hort Research, 2005 (Fuji) 040114-02 | SC | 7.5 | 147 | 1 | 147 | 0 | 0.127 | 0.041 | 0.168 | 040114 |
| | | | | | | 3 | 0.026 | < 0.01 | 0.026 | |
| | | | | | | 8 | 0.015 | ND | 0.015 | |
| | | | | | | 14 | ND | ND | < 0.01 | |
| | | | | | | 21 | ND | ND | < 0.01 | |
| | | | | | | 28 | ND | ND | < 0.01 | |
| | | | | | | 36 | ND | ND | < 0.01 | |
| NEW ZEALAND TRIALS, 2005/2006 SEASON | | | | | | | | | | |
| Twyford, Hawkes Bay, 2006 (Braeburn) 050074-01 | SC | 2.5 | 38-39 | 4 | 154 | 0 | 0.015 | ND | 0.015 | 050074 |
| | | | | | | 3 | 0.012 | ND | 0.012 | |
| | | | | | | 7 | < 0.01 | ND | < 0.01 | |
| | | | | | | 14 | ND | ND | < 0.01 | |
| | | | | | | 21 | ND | ND | < 0.01 | |
| | | | | | | 28 | ND | ND | < 0.01 | |
| | | | | | | 36 | ND | ND | < 0.01 | |
| | | | | | | 42 | ND | ND | < 0.01 | |

| Location, year (Variety) | Form | Application | | | Total/ season, g ai/ha | PHI, days | Residue, mg/kg | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | Total [a] | |
| Twyford, Hawkes Bay, 2006 (Braeburn) 050074-01 | SC | 3.72 | 55- 58 | 4 | 226 | 0 | 0.035 | < 0.01 | 0.035 | 050074 |
| | | | | | | 3 | 0.031 | < 0.01 | 0.031 | |
| | | | | | | 7 | 0.013 | ND | 0.013 | |
| | | | | | | 14 | 0.011 | ND | 0.011 | |
| | | | | | | 21 | ND | ND | < 0.01 | |
| | | | | | | 28 | ND | ND | < 0.01 | |
| | | | | | | 36 | ND | ND | < 0.01 | |
| | | | | | | 42 | ND | ND | < 0.01 | |
| Twyford, Hawkes Bay, 2006 (Braeburn) 050074-01 | SC | 5 | 75- 79 | 4 | 307 | 0 | 0.037 | < 0.01 | 0.037 | 050074 |
| | | | | | | 3 | 0.038 | < 0.01 | 0.038 | |
| | | | | | | 7 | 0.016 | ND | 0.016 | |
| | | | | | | 14 | 0.011 | ND | 0.011 | |
| | | | | | | 21 | < 0.01 | ND | < 0.01 | |
| | | | | | | 28 | ND | ND | < 0.01 | |
| | | | | | | 36 | ND | ND | < 0.01 | |
| | | | | | | 42 | ND | ND | < 0.01 | |
| Twyford, Hawkes Bay, 2006 (Braeburn) 050074-01 | SC | 7.6 | 112- 118 | 4 | 461 | 0 | 0.068 | 0.018 | 0.086 | 050074 |
| | | | | | | 3 | 0.058 | 0.014 | 0.072 | |
| | | | | | | 7 | 0.028 | 0.008 | 0.036 | |
| | | | | | | 14 | 0.016 | ND | 0.016 | |
| | | | | | | 21 | < 0.01 | ND | < 0.01 | |
| | | | | | | 28 | ND | ND | < 0.01 | |
| | | | | | | 36 | ND | ND | < 0.01 | |
| | | | | | | 42 | ND | ND | < 0.01 | |
| Havelock North, Hawkes Bay, 2006 (Royal Gala) 050074-02 | SC | 2.5 | 36- 38 | 4 | 150 | 0 | 0.020 | < 0.01 | 0.02 | 050074 |
| | | | | | | 3 | < 0.01 | ND | < 0.01 | |
| | | | | | | 7 | < 0.01 | ND | < 0.01 | |
| | | | | | | 14 | ND | ND | < 0.01 | |
| | | | | | | 21 | ND | ND | < 0.01 | |
| | | | | | | 28 | ND | ND | < 0.01 | |
| | | | | | | 36 | ND | ND | < 0.01 | |
| | | | | | | 42 | ND | ND | < 0.01 | |

| Location, year (Variety) | Form | Application | | | Total/ season, g ai/ha | PHI, days | Residue, mg/kg | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | Total [a] | |
| Havelock North, Hawkes Bay, 2006 (Royal Gala) 050074-02 | SC | 3.7 | 54- 56 | 4 | 222 | 0 | 0.023 | < 0.01 | 0.023 | 050074 |
| | | | | | | 3 | < 0.01 | ND | < 0.01 | |
| | | | | | | 7 | ND | ND | < 0.01 | |
| | | | | | | 14 | ND | ND | < 0.01 | |
| | | | | | | 21 | ND | ND | < 0.01 | |
| | | | | | | 28 | ND | ND | < 0.01 | |
| | | | | | | 36 | ND | ND | < 0.01 | |
| | | | | | | 42 | ND | ND | < 0.01 | |
| Havelock North, Hawkes Bay, 2006 (Royal Gala) 050074-02 | SC | 5 | 73- 76 | 4 | 300 | 0 | 0.045 | 0.012 | 0.057 | 050074 |
| | | | | | | 3 | < 0.01 | ND | < 0.01 | |
| | | | | | | 7 | < 0.01 | ND | < 0.01 | |
| | | | | | | 14 | < 0.01 | ND | < 0.01 | |
| | | | | | | 21 | ND | ND | < 0.01 | |
| | | | | | | 28 | ND | ND | < 0.01 | |
| | | | | | | 36 | ND | ND | < 0.01 | |
| | | | | | | 42 | ND | ND | < 0.01 | |
| Havelock North, Hawkes Bay, 2006 (Royal Gala) 050074-02 | SC | 7.6 | 109- 115 | 4 | 450 | 0 | 0.074 | 0.019 | 0.093 | 050074 |
| | | | | | | 3 | 0.015 | ND | 0.015 | |
| | | | | | | 7 | < 0.01 | ND | < 0.01 | |
| | | | | | | 14 | < 0.01 | ND | < 0.01 | |
| | | | | | | 21 | ND | ND | < 0.01 | |
| | | | | | | 28 | ND | ND | < 0.01 | |
| | | | | | | 36 | ND | ND | < 0.01 | |
| | | | | | | 42 | ND | ND | < 0.01 | |
| Riwaka, Nelson, 2006 (Royal Gala) 050074-04 | SC | 2.5 | 38- 40 | 4 | 154 | 0 | 0.017 | ND | 0.017 | 050074 |
| | | | | | | 3 | ND | ND | < 0.01 | |
| | | | | | | 7 | ND | ND | < 0.01 | |
| | | | | | | 14 | ND | ND | < 0.01 | |
| | | | | | | 21 | ND | ND | < 0.01 | |
| | | | | | | 28 | ND | ND | < 0.01 | |
| | | | | | | 36 | ND | ND | < 0.01 | |
| | | | | | | 42 | ND | ND | < 0.01 | |
| Riwaka, Nelson, 2006 (Royal Gala) 050074-04 | SC | 3.72 | 56- 59 | 4 | 228 | 0 | 0.016 | ND | 0.016 | 050074 |
| | | | | | | 3 | < 0.01 | ND | < 0.01 | |
| | | | | | | 7 | ND | ND | < 0.01 | |
| | | | | | | 14 | ND | ND | < 0.01 | |
| | | | | | | 21 | ND | ND | < 0.01 | |
| | | | | | | 28 | ND | ND | < 0.01 | |
| | | | | | | 36 | ND | ND | < 0.01 | |
| | | | | | | 42 | ND | ND | < 0.01 | |

| Location, year (Variety) | Form | Application | | | Total/ season, g ai/ha | PHI, days | Residue, mg/kg | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | Total [a] | |
| Riwaka, Nelson, 2006 (Royal Gala) 050074-04 | SC | 5 | 76- 79 | 4 | 308 | 0 | 0.025 | < 0.01 | 0.025 | 050074 |
| | | | | | | 3 | ND | ND | < 0.01 | |
| | | | | | | 7 | < 0.01 | ND | < 0.01 | |
| | | | | | | 14 | ND | ND | < 0.01 | |
| | | | | | | 21 | ND | ND | < 0.01 | |
| | | | | | | 28 | ND | ND | < 0.01 | |
| | | | | | | 36 | ND | ND | < 0.01 | |
| | | | | | | 42 | ND | ND | < 0.01 | |
| Riwaka, Nelson, 2006 (Royal Gala) 050074-04 | SC | 7.6 | 113-119 | 4 | 461 | 0 | 0.060 | 0.018 | 0.078 | 050074 |
| | | | | | | 3 | 0.012 | ND | 0.012 | |
| | | | | | | 7 | < 0.01 | ND | < 0.01 | |
| | | | | | | 14 | < 0.01 | ND | < 0.01 | |
| | | | | | | 21 | ND | ND | < 0.01 | |
| | | | | | | 28 | ND | ND | < 0.01 | |
| | | | | | | 36 | ND | ND | < 0.01 | |
| | | | | | | 42 | ND | ND | < 0.01 | |

[a] Total residue = XDE-175-J + XDE-175-L.

[b] Samples on Days 0, 7, and 28 are from replacement trials in Cottonvale, QLD, referred to as 050062-06 in the study.

Table 24-2 Residues of spinetoram and metabolites from supervised trials on apple in Australia and New Zealand (for estimation of STMR).

| Location, year (Variety) | Form | Application | | | Total/ season, g ai/ha | PHI, days | Residue, mg/kg | | | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | ND-J | NF-J | Total [a] | |
| GAP, New Zealand Pome fruits | WG | 2.5 | | 4 | | 7 | | | | | | |
| AUSTRALIAN TRIALS, 2004/2005 SEASON | | | | | | | | | | | | |
| Shepparton East, VIC, 2005 (Sundowner) 040114-01 | SC | 2.5 | 37.5 | 4 | 150 | 0 | 0.051 | < 0.01 | - | ND | 0.061 | 040114 |
| | | | | | | 3 | 0.021 | ND | - | ND | 0.031 | |
| | | | | | | 7 | < 0.01 | ND | - | ND | < 0.02 | |
| | | | | | | 14 | < 0.01 | ND | - | ND | < 0.02 | |
| | | | | | | 21 | < 0.01 | ND | - | ND | < 0.02 | |
| | | | | | | 28 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 35 | ND | ND | - | ND | < 0.02 | |
| Shepparton East, VIC, 2005 (Sundowner) 040114-01 | SC | 3.75 | 56–57 | 4 | 225 | 0 | 0.120 | 0.023 | - | < 0.01 | 0.153 | 040114 |
| | | | | | | 3 | 0.050 | < 0.01 | - | < 0.01 | 0.060 | |
| | | | | | | 8 | 0.020 | ND | - | ND | 0.030 | |
| | | | | | | 14 | 0.011 | ND | - | ND | 0.021 | |
| | | | | | | 21 | < 0.01 | ND | - | ND | < 0.02 | |
| | | | | | | 28 | < 0.01 | ND | - | ND | < 0.02 | |

| Location, year (Variety) | Form | Application | | | Total/ season, g ai/ha | PHI, days | Residue, mg/kg | | | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | ND-J | NF-J | Total [a] | |
| | | | | | | 36 | ND | ND | - | ND | < 0.02 | |
| Shepparton East, VIC, 2005 (Sundowner) 040114-01 | SC | 5 | 75 | 4 | 300 | 0 | 0.120 | 0.026 | - | < 0.01 | 0.156 | 040114 |
| | | | | | | 3 | 0.094 | 0.012 | - | < 0.01 | 0.116 | |
| | | | | | | 8 | 0.042 | ND | - | < 0.01 | 0.052 | |
| | | | | | | 14 | 0.016 | ND | - | ND | 0.026 | |
| | | | | | | 21 | < 0.01 | ND | - | ND | < 0.02 | |
| | | | | | | 28 | < 0.01 | ND | - | ND | < 0.02 | |
| | | | | | | 36 | < 0.01 | ND | - | ND | < 0.02 | |
| Shepparton East, VIC, 2005 (Sundowner) 040114-01 | SC | 7.5 | 112–113 | 4 | 450 | 0 | 0.186 | 0.039 | - | 0.011 | 0.236 | 040114 |
| | | | | | | 3 | 0.101 | 0.017 | - | 0.012 | 0.130 | |
| | | | | | | 8 | 0.049 | ND | - | < 0.01 | 0.059 | |
| | | | | | | 14 | 0.024 | ND | - | < 0.01 | 0.034 | |
| | | | | | | 21 | < 0.01 | ND | - | ND | < 0.02 | |
| | | | | | | 28 | 0.024 | ND | - | < 0.01 | 0.034 | |
| | | | | | | 36 | < 0.01 | ND | - | ND | < 0.02 | |
| Shepparton East, VIC, 2005 (Sundowner) 040114-01 | SC | 2.5 | 37.5 | 1 | 37.5 | 0 | 0.030 | < 0.01 | - | ND | 0.04 | 040114 |
| | | | | | | 3 | 0.024 | ND | - | ND | 0.034 | |
| | | | | | | 8 | < 0.01 | ND | - | ND | < 0.02 | |
| | | | | | | 14 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 21 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 28 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 36 | ND | ND | - | ND | < 0.02 | |
| Shepparton East, VIC, 2005 (Sundowner) 040114-01 | SC | 3.75 | 56 | 1 | 56 | 0 | 0.053 | 0.012 | - | ND | 0.075 | 040114 |
| | | | | | | 3 | 0.026 | ND | - | ND | 0.036 | |
| | | | | | | 8 | < 0.01 | ND | - | ND | < 0.02 | |
| | | | | | | 14 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 21 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 28 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 36 | ND | ND | - | ND | < 0.02 | |
| Shepparton East, VIC, 2005 (Sundowner) 040114-01 | SC | 5 | 75 | 1 | 75 | 0 | 0.095 | 0.022 | - | ND | 0.127 | 040114 |
| | | | | | | 3 | 0.040 | < 0.01 | - | ND | 0.050 | |
| | | | | | | 8 | 0.017 | ND | - | ND | 0.027 | |
| | | | | | | 14 | 0.010 | ND | - | ND | 0.020 | |
| | | | | | | 21 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 28 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 36 | ND | ND | - | ND | < 0.02 | |
| Shepparton East, VIC, 2005 (Sundowner) 040114-01 | SC | 7.5 | 112.5 | 1 | 112.5 | 0 | 0.123 | 0.031 | - | ND | 0.164 | 040114 |
| | | | | | | 3 | 0.076 | 0.014 | - | < 0.01 | 0.100 | |
| | | | | | | 8 | 0.014 | ND | - | ND | 0.024 | |
| | | | | | | 14 | 0.034 | ND | - | ND | 0.044 | |
| | | | | | | 21 | < 0.01 | ND | - | ND | < 0.02 | |
| | | | | | | 28 | < 0.01 | ND | - | ND | < 0.02 | |
| | | | | | | 36 | ND | ND | - | ND | < 0.02 | |

| Location, year (Variety) | Form | Application | | | Total/ season, g ai/ha | PHI, days | Residue, mg/kg | | | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | ND-J | NF-J | Total [a] | |
| AUSTRALIAN TRIALS, 2005/2006 SEASON | | | | | | | | | | | | |
| Ardmona VIC, 2006 (Granny Smith) 050062-01 | SC | 5 | 62 | 4 | 248 | 0 | 0.04 | < 0.01 | ND | ND | 0.05 | 050062 |
| | | | | | | 7 | < 0.01 | ND | ND | ND | < 0.02 | |
| | | | | | | 14 | ND | ND | ND | ND | < 0.02 | |
| | | | | | | 28 | ND | ND | ND | ND | < 0.02 | |
| | | | | | | 35 | ND | ND | ND | ND | < 0.02 | |
| Ardmona VIC, 2006 (Granny Smith) 050062-01 | SC | 7.5 | 93 | 4 | 372 | 0 | 0.06 | 0.02 | ND | ND | 0.09 | 050062 |
| | | | | | | 7 | 0.01 | ND | < 0.01 | ND | 0.02 | |
| | | | | | | 14 | ND | ND | ND | ND | < 0.02 | |
| | | | | | | 28 | ND | ND | ND | ND | < 0.02 | |
| | | | | | | 35 | ND | ND | ND | ND | < 0.02 | |
| Poziers, QLD, 2005-06 (Summer Del) 050062-02 [b] | WG | 5 | 186-229 | 4 | 805 | 0 | 0.05 | 0.01 | < 0.01 | ND | 0.07 | 050062 |
| | | | | | | 7 | < 0.01 | ND | 0.01 | < 0.01 | 0.03 | |
| | | | | | | 28 | ND | ND | < 0.01 | < 0.01 | < 0.02 | |
| | | | | | | 35 | ND | ND | ND | ND | < 0.02 | |
| Poziers, QLD, 2005-06 (Summer Del) 050062-02 [b] | WG | 7.5 | 278–344 | 4 | 1207 | 0 | 0.04 | 0.02 | < 0.01 | < 0.01 | 0.07 | 050062 |
| | | | | | | 7 | 0.03 | ND | 0.03 | 0.01 | 0.07 | |
| | | | | | | 28 | 0.01 | ND | 0.02 | 0.01 | 0.04 | |
| | | | | | | 35 | ND | ND | < 0.01 | < 0.01 | < 0.02 | |
| Spreyton, Tasmania, 2006 (Fuji) 050062-03 | WG | 5 | 128–149 | 4 | 551 | 0 | 0.09 | 0.01 | < 0.01 | < 0.01 | 0.11 | 050062 |
| | | | | | | 7 | 0.03 | ND | < 0.01 | < 0.01 | 0.04 | |
| | | | | | | 14 | 0.02 | ND | ND | ND | 0.03 | |
| | | | | | | 28 | < 0.01 | ND | ND | ND | < 0.02 | |
| | | | | | | 35 | ND | ND | ND | ND | < 0.02 | |
| Spreyton, Tasmania, 2006 (Fuji) 050062-03 | WG | 7.5 | 188–207 | 4 | 797 | 0 | 0.11 | 0.02 | < 0.01 | < 0.01 | 0.14 | 050062 |
| | | | | | | 7 | 0.04 | ND | 0.010 | < 0.01 | 0.06 | |
| | | | | | | 14 | 0.02 | ND | ND | ND | 0.03 | |
| | | | | | | 28 | < 0.01 | ND | ND | ND | < 0.02 | |
| | | | | | | 35 | ND | ND | ND | ND | < 0.02 | |
| Ardmona VIC, 2006 (Granny Smith) 050062-01 | SC | 5 | 62 | 7 | 434 | 0 | 0.06 | 0.02 | < 0.01 | ND | 0.09 | 050062 |
| | | | | | | 7 | < 0.01 | ND | ND | ND | < 0.02 | |
| | | | | | | 14 | ND | ND | ND | ND | < 0.02 | |
| | | | | | | 28 | ND | ND | ND | ND | < 0.02 | |
| | | | | | | 35 | ND | ND | ND | ND | < 0.02 | |
| Ardmona VIC, 2006 (Granny Smith) 050062-01 | SC | 7.5 | 93 | 7 | 651 | 0 | 0.04 | 0.01 | ND | ND | 0.06 | 050062 |
| | | | | | | 7 | 0.01 | ND | < 0.01 | ND | 0.02 | |
| | | | | | | 14 | ND | ND | ND | ND | < 0.02 | |
| | | | | | | 28 | ND | ND | ND | ND | < 0.02 | |
| | | | | | | 35 | ND | ND | ND | ND | < 0.02 | |

| Location, year (Variety) | Form | Application | | | Total/ season, g ai/ha | PHI, days | Residue, mg/kg | | | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | ND-J | NF-J | Total [a] | |
| Poziers, QLD 2005-06 (Summer Del) 050062-02 | WG | 5 | 65–229 | 7 | 848 | 0 | ND | ND | ND | < 0.01 | < 0.02 | 050062 |
| | | | | | | 35 | ND | ND | ND | ND | < 0.02 | |
| Poziers, QLD, 2005-06 (Summer Del) 050062-02 | WG | 7.5 | 98–344 | 7 | 1549 | 0 | ND | ND | ND | < 0.01 | < 0.02 | 050062 |
| | | | | | | 35 | ND | ND | ND | < 0.01 | < 0.02 | |
| Spreyton, Tasmania, 2006 (Fuji) APPLE 050062-03 | WG | 5 | 70–149 | 7 | 785 | 0 | 0.05 | < 0.01 | ND | ND | 0.06 | 050062 |
| | | | | | | 7 | 0.03 | ND | < 0.01 | ND | 0.04 | |
| | | | | | | 14 | 0.02 | ND | ND | ND | 0.03 | |
| | | | | | | 28 | ND | ND | ND | ND | < 0.02 | |
| | | | | | | 35 | ND | ND | ND | ND | < 0.02 | |
| Spreyton, Tasmania, 2006 (Fuji) 050062-03 | WG | 7.5 | 101–207 | 7 | 1017 | 0 | 0.11 | 0.02 | 0.01 | < 0.01 | 0.15 | 050062 |
| | | | | | | 7 | 0.03 | ND | < 0.01 | < 0.01 | 0.04 | |
| | | | | | | 14 | 0.01 | ND | ND | ND | 0.02 | |
| | | | | | | 28 | 0.01 | ND | ND | ND | 0.02 | |
| | | | | | | 35 | ND | ND | ND | ND | < 0.02 | |
| NEW ZEALAND TRIALS, 2004/2005 SEASON | | | | | | | | | | | | |
| Hort Research, 2005 (Fuji) 040114-02 | SC | 2.5 | 52–62 | 4 | 233 | 0 | 0.032 | < 0.01 | - | < 0.01 | 0.042 | 040114 |
| | | | | | | 3 | < 0.01 | ND | - | < 0.01 | < 0.02 | |
| | | | | | | 8 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 14 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 21 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 28 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 36 | ND | ND | - | ND | < 0.02 | |
| Hort Research, 2005 (Fuji) 040114-02 | SC | 3.75 | 69–83 | 4 | 307 | 0 | 0.050 | 0.016 | - | < 0.01 | 0.076 | 040114 |
| | | | | | | 3 | < 0.01 | ND | - | < 0.01 | < 0.02 | |
| | | | | | | 8 | < 0.01 | ND | - | < 0.01 | < 0.02 | |
| | | | | | | 14 | ND | ND | - | < 0.01 | < 0.02 | |
| | | | | | | 21 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 28 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 36 | ND | ND | - | ND | < 0.02 | |
| Hort Research, 2005 (Fuji) 040114-02 | SC | 5 | 122–130 | 4 | 498 | 0 | 0.068 | 0.020 | - | < 0.01 | 0.098 | 040114 |
| | | | | | | 3 | 0.012 | ND | - | < 0.01 | 0.022 | |
| | | | | | | 8 | < 0.01 | ND | - | < 0.01 | < 0.02 | |
| | | | | | | 14 | ND | ND | - | < 0.01 | < 0.02 | |
| | | | | | | 21 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 28 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 36 | ND | ND | - | ND | < 0.02 | |

| Location, year (Variety) | Form | Application | | | Total/ season, g ai/ha | PHI, days | Residue, mg/kg | | | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | ND-J | NF-J | Total [a] | |
| Hort Research, 2005 (Fuji) 040114-02 | SC | 7.5 | 159-180 | 4 | 681 | 0 | 0.075 | 0.023 | - | < 0.01 | 0.108 | 040114 |
| | | | | | | 3 | 0.025 | < 0.01 | - | < 0.01 | 0.035 | |
| | | | | | | 8 | < 0.01 | ND | - | < 0.01 | < 0.02 | |
| | | | | | | 14 | < 0.01 | ND | - | < 0.01 | < 0.02 | |
| | | | | | | 21 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 28 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 36 | ND | ND | - | ND | < 0.02 | |
| Hort Research, 2005 (Fuji) 040114-02 | SC | 2.5 | 62 | 1 | 62 | 0 | 0.021 | < 0.01 | - | ND | 0.031 | 040114 |
| | | | | | | 3 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 8 | ND | ND | - | < 0.01 | < 0.02 | |
| | | | | | | 14 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 21 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 28 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 36 | ND | ND | - | ND | < 0.02 | |
| Hort Research, 2005 (Fuji) 040114-02 | SC | 3.75 | 81 | 1 | 81 | 0 | 0.046 | 0.015 | - | ND | 0.071 | 040114 |
| | | | | | | 3 | 0.015 | ND | - | < 0.01 | 0.025 | |
| | | | | | | 8 | < 0.01 | ND | - | ND | < 0.02 | |
| | | | | | | 14 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 21 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 28 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 36 | ND | ND | - | ND | < 0.02 | |
| Hort Research, 2005 (Fuji) 040114-02 | SC | 5 | 104 | 1 | 104 | 0 | 0.086 | 0.027 | - | ND | 0.123 | 040114 |
| | | | | | | 3 | 0.016 | ND | - | < 0.01 | 0.026 | |
| | | | | | | 8 | < 0.01 | ND | - | < 0.01 | < 0.02 | |
| | | | | | | 14 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 21 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 28 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 36 | ND | ND | - | ND | < 0.02 | |
| Hort Research, 2005 (Fuji) 040114-02 | SC | 7.5 | 147 | 1 | 147 | 0 | 0.127 | 0.041 | - | ND | 0.178 | 040114 |
| | | | | | | 3 | 0.026 | < 0.01 | - | < 0.01 | 0.036 | |
| | | | | | | 8 | 0.015 | ND | - | < 0.01 | 0.025 | |
| | | | | | | 14 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 21 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 28 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 36 | ND | ND | - | ND | < 0.02 | |
| NEW ZEALAND TRIALS, 2005/2006 SEASON | | | | | | | | | | | | |
| Twyford, Hawkes Bay, 2006 (Braeburn) 050074-01 | SC | 2.5 | 38-39 | 4 | 154 | 0 | 0.015 | ND | - | ND | 0.025 | 050074 |
| | | | | | | 3 | 0.012 | ND | - | ND | 0.022 | |
| | | | | | | 7 | < 0.01 | ND | - | ND | < 0.02 | |
| | | | | | | 14 | ND | ND | - | < 0.01 | < 0.02 | |
| | | | | | | 21 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 28 | ND | ND | - | ND | < 0.02 | |

| Location, year (Variety) | Form | Application | | | Total/ season, g ai/ha | PHI, days | Residue, mg/kg | | | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | ND-J | NF-J | Total [a] | |
| | | | | | | 36 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 42 | ND | ND | - | ND | < 0.02 | |
| Twyford, Hawkes Bay, 2006 (Braeburn) 050074-01 | SC | 5 | 75–79 | 4 | 307 | 0 | 0.037 | < 0.01 | - | < 0.01 | 0.047 | 050074 |
| | | | | | | 3 | 0.038 | < 0.01 | - | < 0.01 | 0.048 | |
| | | | | | | 7 | 0.016 | ND | - | 0.010 | 0.026 | |
| | | | | | | 14 | 0.011 | ND | - | < 0.01 | 0.021 | |
| | | | | | | 21 | < 0.01 | ND | - | < 0.01 | < 0.02 | |
| | | | | | | 28 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 36 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 42 | ND | ND | - | 0.008 | 0.018 | |
| Twyford, Hawkes Bay, 2006 (Braeburn) 050074-01 | SC | 3.72 | 55–58 | 4 | 226 | 0 | 0.035 | < 0.01 | - | < 0.01 | 0.045 | 050074 |
| | | | | | | 3 | 0.031 | < 0.01 | - | ND | 0.041 | |
| | | | | | | 7 | 0.013 | ND | - | < 0.01 | 0.023 | |
| | | | | | | 14 | 0.011 | ND | - | < 0.01 | 0.021 | |
| | | | | | | 21 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 28 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 36 | ND | ND | - | < 0.01 | < 0.02 | |
| | | | | | | 42 | ND | ND | - | ND | < 0.02 | |
| Twyford, Hawkes Bay, 2006 (Braeburn) 050074-01 | SC | 7.6 | 112-118 | 4 | 461 | 0 | 0.068 | 0.018 | - | < 0.01 | 0.096 | 050074 |
| | | | | | | 3 | 0.058 | 0.014 | - | 0.013 | 0.085 | |
| | | | | | | 7 | 0.028 | 0.008 | - | 0.012 | 0.048 | |
| | | | | | | 14 | 0.016 | ND | - | 0.011 | 0.027 | |
| | | | | | | 21 | < 0.01 | ND | - | < 0.01 | < 0.02 | |
| | | | | | | 28 | ND | ND | - | < 0.01 | < 0.02 | |
| | | | | | | 36 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 42 | ND | ND | - | ND | < 0.02 | |
| Havelock North, Hawkes Bay, 2006 (Royal Gala) 050074-02 | SC | 2.5 | 36–38 | 4 | 150 | 0 | 0.020 | < 0.01 | - | ND | 0.030 | 050074 |
| | | | | | | 3 | < 0.01 | ND | - | < 0.01 | < 0.02 | |
| | | | | | | 7 | < 0.01 | ND | - | < 0.01 | < 0.02 | |
| | | | | | | 14 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 21 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 28 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 36 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 42 | ND | ND | - | ND | < 0.02 | |
| Havelock North, Hawkes Bay, 2006 (Royal Gala) 050074-02 | SC | 3.7 | 54–56 | 4 | 222 | 0 | 0.023 | < 0.01 | - | < 0.01 | 0.033 | 050074 |
| | | | | | | 3 | < 0.01 | ND | - | < 0.01 | < 0.02 | |
| | | | | | | 7 | ND | ND | - | < 0.01 | < 0.02 | |
| | | | | | | 14 | ND | ND | - | < 0.01 | < 0.02 | |
| | | | | | | 21 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 28 | ND | ND | - | ND | < 0.02 | |

| Location, year (Variety) | Form | Application | | | Total/ season, g ai/ha | PHI, days | Residue, mg/kg | | | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | ND-J | NF-J | Total [a] | |
| | | | | | | 36 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 42 | ND | ND | - | ND | < 0.02 | |
| Havelock North, Hawkes Bay, 2006 (Royal Gala) 050074-02 | SC | 5 | 73–76 | 4 | 300 | 0 | 0.045 | 0.012 | - | < 0.01 | 0.067 | 050074 |
| | | | | | | 3 | < 0.01 | ND | - | < 0.01 | < 0.02 | |
| | | | | | | 7 | < 0.01 | ND | - | < 0.01 | < 0.02 | |
| | | | | | | 14 | < 0.01 | ND | - | < 0.01 | < 0.02 | |
| | | | | | | 21 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 28 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 36 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 42 | ND | ND | - | ND | < 0.02 | |
| Havelock North, Hawkes Bay, 2006 (Royal Gala) 050074-02 | SC | 7.6 | 109–115 | 4 | 450 | 0 | 0.074 | 0.019 | - | 0.012 | 0.105 | 050074 |
| | | | | | | 3 | 0.015 | ND | - | < 0.01 | 0.025 | |
| | | | | | | 7 | < 0.01 | ND | - | < 0.01 | < 0.02 | |
| | | | | | | 14 | < 0.01 | ND | - | < 0.01 | < 0.02 | |
| | | | | | | 21 | ND | ND | - | < 0.01 | < 0.02 | |
| | | | | | | 28 | ND | ND | - | < 0.01 | < 0.02 | |
| | | | | | | 36 | ND | ND | - | < 0.01 | < 0.02 | |
| | | | | | | 42 | ND | ND | - | ND | < 0.02 | |
| Riwaka, Nelson, 2006 (Royal Gala) 050074-04 | SC | 2.5 | 38–40 | 4 | 154 | 0 | 0.017 | ND | - | < 0.01 | 0.027 | 050074 |
| | | | | | | 3 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 7 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 14 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 21 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 28 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 36 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 42 | ND | ND | - | ND | < 0.02 | |
| Riwaka, Nelson, 2006 (Royal Gala) 050074-04 | SC | 3.72 | 56–59 | 4 | 228 | 0 | 0.016 | ND | - | < 0.01 | 0.026 | 050074 |
| | | | | | | 3 | < 0.01 | ND | - | ND | < 0.02 | |
| | | | | | | 7 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 14 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 21 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 28 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 36 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 42 | ND | ND | - | ND | < 0.02 | |
| Riwaka, Nelson, 2006 (Royal Gala) 050074-04 | SC | 5 | 76–79 | 4 | 308 | 0 | 0.025 | < 0.01 | - | ND | 0.035 | 050074 |
| | | | | | | 3 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 7 | < 0.01 | ND | - | < 0.01 | < 0.02 | |
| | | | | | | 14 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 21 | ND | ND | - | ND | < 0.02 | |

| Location, year (Variety) | Form | Application | | | Total/ season, g ai/ha | PHI, days | Residue, mg/kg | | | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | ND-J | NF-J | Total [a] | |
| | | | | | | 28 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 36 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 42 | ND | ND | - | ND | < 0.02 | |
| Riwaka, Nelson, 2006 (Royal Gala) 050074-04 | SC | 7.6 | 113–119 | 4 | 461 | 0 | 0.060 | 0.018 | - | < 0.01 | 0.088 | 050074 |
| | | | | | | 3 | 0.012 | ND | - | < 0.01 | 0.022 | |
| | | | | | | 7 | < 0.01 | ND | - | < 0.01 | < 0.02 | |
| | | | | | | 14 | < 0.01 | ND | - | < 0.01 | < 0.02 | |
| | | | | | | 21 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 28 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 36 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 42 | ND | ND | - | ND | < 0.02 | |

[a] Total residue = XDE-175-J + XDE-175-L + ND-J + NF-J.

[b] Samples on Days 0, 7, and 28 are from replacement trials in Cottonvale, QLD, referred to as 050062-06 in the study.

Table 25-1 Residues of spinetoram from supervised trials on pear in Australia and New Zealand (for estimation of maximum residue level)

| Location, year (Variety) | Form | Application | | | Total/ season, g ai/ha | PHI, days | Residue, mg/kg | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | Total [a] | |
| GAP, New Zealand Pome fruits | WG | 2.5 | | 4 | 200 | 7 | | | | |
| AUSTRALIAN TRIALS, 2005/2006 SEASON | | | | | | | | | | |
| Ardmona VIC, 2006 (Packham) 050062-04 | WG | 5 | 74 | 4 | 296 | 0 | 0.05 | 0.01 | 0.06 | 050062 |
| | | | | | | 7 | 0.01 | ND | 0.01 | |
| | | | | | | 14 | ND | ND | < 0.01 | |
| | | | | | | 28 | ND | ND | < 0.01 | |
| | | | | | | 35 | ND | ND | < 0.01 | |
| Ardmona VIC, 2006 (Packham) 050062-04 | WG | 7.5 | 111 | 4 | 444 | 0 | 0.11 | 0.02 | 0.13 | 050062 |
| | | | | | | 7 | 0.03 | ND | 0.03 | |
| | | | | | | 14 | 0.02 | ND | 0.02 | |
| | | | | | | 28 | < 0.01 | ND | < 0.01 | |
| | | | | | | 35 | ND | ND | < 0.01 | |
| Paracombe, SA, 2006 (Packham) 050062-05 | WG | 5 | 97–105 | 4 | 404 | 0 | 0.02 | ND | 0.02 | 050062 |
| | | | | | | 7 | ND | ND | < 0.01 | |
| | | | | | | 14 | ND | ND | < 0.01 | |
| | | | | | | 28 | ND | ND | < 0.01 | |
| | | | | | | 35 | ND | ND | < 0.01 | |
| Paracombe, SA, 2006 (Packham) 050062-05 | WG | 7.5 | 145–158 | 4 | 606 | 0 | 0.03 | ND | 0.03 | 050062 |
| | | | | | | 7 | < 0.01 | ND | < 0.01 | |
| | | | | | | 14 | < 0.01 | ND | < 0.01 | |
| | | | | | | 28 | ND | ND | < 0.01 | |

| Location, year (Variety) | Form | Application | | | Total/ season, g ai/ha | PHI, days | Residue, mg/kg | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | Total [a] | |
| | | | | | | 35 | ND | ND | < 0.01 | |
| Ardmona VIC, 2006 (Packham) 050062-04 | WG | 5 | 73–74 | 7 | 517 | 0 | 0.04 | < 0.01 | 0.04 | 050062 |
| | | | | | | 7 | 0.02 | ND | 0.02 | |
| | | | | | | 14 | 0.01 | ND | 0.01 | |
| | | | | | | 28 | ND | ND | < 0.01 | |
| | | | | | | 35 | ND | ND | < 0.01 | |
| Ardmona VIC, 2006 (Packham) 050062-04 | WG | 7.5 | 102–111 | 7 | 768 | 0 | 0.09 | 0.02 | 0.11 | 050062 |
| | | | | | | 7 | 0.02 | ND | 0.02 | |
| | | | | | | 14 | < 0.01 | ND | < 0.01 | |
| | | | | | | 28 | ND | ND | < 0.01 | |
| | | | | | | 35 | ND | ND | < 0.01 | |
| Paracombe, SA, 2006 (Packham) 050062-05 | WG | 5 | 91–105 | 7 | 677 | 0 | 0.01 | ND | 0.01 | 050062 |
| | | | | | | 7 | ND | ND | < 0.01 | |
| | | | | | | 14 | ND | ND | < 0.01 | |
| | | | | | | 28 | ND | ND | < 0.01 | |
| | | | | | | 35 | ND | ND | < 0.01 | |
| Paracombe, SA, 2006 (Packham) 050062-05 | WG | 7.5 | 136–158 | 7 | 1014 | 0 | 0.02 | ND | 0.02 | 050062 |
| | | | | | | 7 | ND | ND | < 0.01 | |
| | | | | | | 14 | ND | ND | < 0.01 | |
| | | | | | | 28 | ND | ND | < 0.01 | |
| | | | | | | 35 | ND | ND | < 0.01 | |
| NEW ZEALAND TRIALS, 2005/2006 SEASON | | | | | | | | | | |
| Hastings, Hawkes Bay, 2006 (Packham) 050074-03 | SC | 2.5 | 46–47 | 4 | 187 | 0 | 0.011 | ND | 0.011 | 050074 |
| | | | | | | 3 | < 0.01 | ND | < 0.01 | |
| | | | | | | 7 | ND | ND | < 0.01 | |
| | | | | | | 14 | ND | ND | < 0.01 | |
| | | | | | | 21 | ND | ND | < 0.01 | |
| | | | | | | 28 | ND | ND | < 0.01 | |
| | | | | | | 36 | ND | ND | < 0.01 | |
| | | | | | | 42 | ND | ND | < 0.01 | |
| Hastings, Hawkes Bay, 2006 (Packham) 050074-03 | SC | 3.72 | 68–70 | 4 | 275 | 0 | 0.021 | < 0.01 | 0.021 | 050074 |
| | | | | | | 3 | ND | ND | < 0.01 | |
| | | | | | | 7 | ND | ND | < 0.01 | |
| | | | | | | 14 | ND | ND | < 0.01 | |
| | | | | | | 21 | ND | ND | < 0.01 | |
| | | | | | | 28 | ND | ND | < 0.01 | |
| | | | | | | 36 | ND | ND | < 0.01 | |
| | | | | | | 42 | ND | ND | < 0.01 | |

| Location, year (Variety) | Form | Application | | | Total/ season, g ai/ha | PHI, days | Residue, mg/kg | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | Total [a] | |
| Hastings, Hawkes Bay, 2006 (Packham) 050074-03 | SC | 5 | 92–95 | 4 | 373 | 0 | 0.025 | < 0.01 | 0.025 | 050074 |
| | | | | | | 3 | 0.016 | ND | 0.016 | |
| | | | | | | 7 | < 0.01 | ND | < 0.01 | |
| | | | | | | 14 | < 0.01 | ND | < 0.01 | |
| | | | | | | 21 | ND | ND | < 0.01 | |
| | | | | | | 28 | ND | ND | < 0.01 | |
| | | | | | | 36 | ND | ND | < 0.01 | |
| | | | | | | 42 | ND | ND | < 0.01 | |
| Hastings, Hawkes Bay, 2006 (Packham) 050074-03 | SC | 7.6 | 139–142 | 4 | 561 | 0 | 0.037 | 0.011 | 0.048 | 050074 |
| | | | | | | 3 | 0.027 | ND | 0.027 | |
| | | | | | | 7 | ND | ND | < 0.01 | |
| | | | | | | 14 | < 0.01 | ND | < 0.01 | |
| | | | | | | 21 | ND | ND | < 0.01 | |
| | | | | | | 28 | ND | ND | < 0.01 | |
| | | | | | | 36 | ND | ND | < 0.01 | |
| | | | | | | 42 | ND | ND | < 0.01 | |
| Upper Moutere, Nelson, 2006 (Taylor's Gold) 050074-05 | SC | 2.5 | 38–41 | 4 | 154 | 0 | 0.032 | ND | 0.032 | 050074 |
| | | | | | | 3 | 0.026 | ND | 0.026 | |
| | | | | | | 7 | 0.015 | ND | 0.015 | |
| | | | | | | 14 | 0.015 | ND | 0.015 | |
| | | | | | | 21 | < 0.01 | ND | < 0.01 | |
| | | | | | | 28 | < 0.01 | ND | < 0.01 | |
| | | | | | | 36 | ND | ND | < 0.01 | |
| | | | | | | 42 | ND | ND | < 0.01 | |
| Upper Moutere, Nelson, 2006 (Taylor's Gold) 050074-05 | SC | 3.72 | 56–60 | 4 | 233 | 0 | 0.043 | < 0.01 | 0.043 | 050074 |
| | | | | | | 3 | 0.053 | < 0.01 | 0.053 | |
| | | | | | | 7 | 0.041 | ND | 0.041 | |
| | | | | | | 14 | 0.023 | ND | 0.023 | |
| | | | | | | 21 | 0.018 | ND | 0.018 | |
| | | | | | | 28 | 0.017 | ND | 0.017 | |
| | | | | | | 36 | 0.012 | ND | 0.012 | |
| | | | | | | 42 | < 0.01 | ND | < 0.01 | |
| Upper Moutere, Nelson, 2006 (Taylor's Gold) 050074-05 | SC | 5 | 76–81 | 4 | 315 | 0 | 0.064 | < 0.01 | 0.064 | 050074 |
| | | | | | | 3 | 0.062 | 0.011 | 0.073 | |
| | | | | | | 7 | 0.035 | ND | 0.035 | |
| | | | | | | 14 | 0.041 | < 0.01 | 0.041 | |
| | | | | | | 21 | 0.027 | ND | 0.027 | |
| | | | | | | 28 | 0.019 | ND | 0.019 | |
| | | | | | | 36 | 0.016 | ND | 0.016 | |
| | | | | | | 42 | < 0.01 | ND | < 0.01 | |

| Location, year (Variety) | Form | Application | | | Total/ season, g ai/ha | PHI, days | Residue, mg/kg | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | Total [a] | |
| Upper Moutere, Nelson, 2006 (Taylor's Gold) 050074-05 | SC | 7.6 | 115–121 | 4 | 472 | 0 | 0.096 | 0.017 | 0.113 | 050074 |
| | | | | | | 3 | 0.066 | 0.010 | 0.076 | |
| | | | | | | 7 | 0.056 | < 0.01 | 0.056 | |
| | | | | | | 14 | 0.037 | ND | 0.037 | |
| | | | | | | 21 | 0.049 | < 0.01 | 0.049 | |
| | | | | | | 28 | 0.027 | ND | 0.027 | |
| | | | | | | 36 | 0.013 | ND | 0.013 | |
| | | | | | | 42 | 0.010 | ND | 0.010 | |

[a] Total residue = XDE-175-J + XDE-175-L.

Table 25-2 Residues of spinetoram and metabolites from supervised trials on pear in Australia and New Zealand (for estimation of STMR)

| Location, year (Variety) | Form | Application | | | Total/ season, g ai/ha | PHI, days | Residue, mg/kg | | | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | ND-J a | NF-J | Total b | |
| GAP, New Zealand Pome fruit | WG | 2.5 | | 4 | 200 | 7 | | | | | | |
| AUSTRALIAN TRIALS, 2005/2006 SEASON | | | | | | | | | | | | |
| Ardmona VIC, 2006 (Packham) 050062-04 | WG | 5 | 74 | 4 | 296 | 0 | 0.05 | 0.01 | ND | ND | 0.07 | 050062 |
| | | | | | | 7 | 0.01 | ND | ND | ND | 0.02 | |
| | | | | | | 14 | ND | ND | ND | ND | < 0.02 | |
| | | | | | | 28 | ND | ND | ND | ND | < 0.02 | |
| | | | | | | 35 | ND | ND | ND | ND | < 0.02 | |
| Ardmona VIC, 2006 (Packham) 050062-04 | WG | 7.5 | 111 | 4 | 444 | 0 | 0.11 | 0.02 | < 0.01 | ND | 0.14 | 050062 |
| | | | | | | 7 | 0.03 | ND | < 0.01 | ND | 0.04 | |
| | | | | | | 14 | 0.02 | ND | < 0.01 | ND | 0.03 | |
| | | | | | | 28 | < 0.01 | ND | ND | ND | < 0.02 | |
| | | | | | | 35 | ND | ND | ND | ND | < 0.02 | |
| Paracombe, SA, 2006 (Packham) 050062-05 | WG | 5 | 97–105 | 4 | 404 | 0 | 0.02 | ND | ND | ND | 0.03 | 050062 |
| | | | | | | 7 | ND | ND | ND | ND | < 0.02 | |
| | | | | | | 14 | ND | ND | ND | ND | < 0.02 | |
| | | | | | | 28 | ND | ND | ND | ND | < 0.02 | |
| | | | | | | 35 | ND | ND | ND | ND | < 0.02 | |
| Paracombe, SA, 2006 (Packham) 050062-05 | WG | 7.5 | 145–158 | 4 | 606 | 0 | 0.03 | ND | ND | ND | 0.04 | 050062 |
| | | | | | | 7 | < 0.01 | ND | ND | ND | < 0.02 | |
| | | | | | | 14 | < 0.01 | ND | ND | ND | < 0.02 | |
| | | | | | | 28 | ND | ND | ND | ND | < 0.02 | |
| | | | | | | 35 | ND | ND | ND | ND | < 0.02 | |

| Location, year (Variety) | Form | Application | | | Total/ season, g ai/ha | PHI, days | Residue, mg/kg | | | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | ND-J a | NF-J | Total b | |
| Ardmona VIC, 2006 (Packham) 050062-04 | WG | 5 | 73–74 | 7 | 517 | 0 | 0.04 | < 0.01 | ND | ND | 0.05 | 050062 |
| | | | | | | 7 | 0.02 | ND | ND | ND | 0.03 | |
| | | | | | | 14 | 0.01 | ND | ND | ND | 0.02 | |
| | | | | | | 28 | ND | ND | ND | ND | < 0.02 | |
| | | | | | | 35 | ND | ND | ND | ND | < 0.02 | |
| Ardmona VIC, 2006 (Packham) 050062-04 | WG | 7.5 | 102–111 | 7 | 768 | 0 | 0.09 | 0.02 | ND | < 0.01 | 0.12 | 050062 |
| | | | | | | 7 | 0.02 | ND | ND | < 0.01 | 0.03 | |
| | | | | | | 14 | < 0.01 | ND | ND | ND | < 0.02 | |
| | | | | | | 28 | ND | ND | ND | ND | < 0.02 | |
| | | | | | | 35 | ND | ND | ND | ND | < 0.02 | |
| Paracombe, SA, 2006 (Packham) 050062-05 | WG | 5 | 91–105 | 7 | 677 | 0 | 0.01 | ND | ND | ND | 0.02 | 050062 |
| | | | | | | 7 | ND | ND | ND | ND | < 0.02 | |
| | | | | | | 14 | ND | ND | ND | ND | < 0.02 | |
| | | | | | | 28 | ND | ND | ND | ND | < 0.02 | |
| | | | | | | 35 | ND | ND | ND | ND | < 0.02 | |
| Paracombe, SA, 2006 (Packham) 050062-05 | WG | 7.5 | 136–158 | 7 | 1014 | 0 | 0.02 | ND | ND | ND | 0.03 | 050062 |
| | | | | | | 7 | ND | ND | ND | ND | < 0.02 | |
| | | | | | | 14 | ND | ND | ND | ND | < 0.02 | |
| | | | | | | 28 | ND | ND | ND | ND | < 0.02 | |
| | | | | | | 35 | ND | ND | ND | ND | < 0.02 | |
| NEW ZEALAND TRIALS, 2005/2006 SEASON | | | | | | | | | | | | |
| Hastings, Hawkes Bay, 2006 (Packham) 050074-03 | SC | 2.5 | 46–47 | 4 | 187 | 0 | 0.011 | ND | - | ND | 0.021 | 050074 |
| | | | | | | 3 | < 0.01 | ND | - | ND | < 0.02 | |
| | | | | | | 7 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 14 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 21 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 28 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 36 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 42 | ND | ND | - | ND | < 0.02 | |
| Hastings, Hawkes Bay, 2006 (Packham) 050074-03 | SC | 3.72 | 68–70 | 4 | 275 | 0 | 0.021 | < 0.01 | - | ND | 0.031 | 050074 |
| | | | | | | 3 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 7 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 14 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 21 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 28 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 36 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 42 | ND | ND | - | ND | < 0.02 | |

| Location, year (Variety) | Form | Application | | | Total/ season, g ai/ha | PHI, days | Residue, mg/kg | | | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | ND-J a | NF-J | Total b | |
| Hastings, Hawkes Bay, 2006 (Packham) 050074-03 | SC | 5 | 92–95 | 4 | 373 | 0 | 0.025 | < 0.01 | - | ND | 0.035 | 050074 |
| | | | | | | 3 | 0.016 | ND | - | ND | 0.026 | |
| | | | | | | 7 | < 0.01 | ND | - | ND | < 0.02 | |
| | | | | | | 14 | < 0.01 | ND | - | < 0.01 | < 0.02 | |
| | | | | | | 21 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 28 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 36 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 42 | ND | ND | - | ND | < 0.02 | |
| Hastings, Hawkes Bay, 2006 (Packham) 050074-03 | SC | 7.6 | 139–142 | 4 | 561 | 0 | 0.037 | 0.011 | - | ND | 0.058 | 050074 |
| | | | | | | 3 | 0.027 | ND | - | < 0.01 | 0.037 | |
| | | | | | | 7 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 14 | < 0.01 | ND | - | ND | < 0.02 | |
| | | | | | | 21 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 28 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 36 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 42 | ND | ND | - | ND | < 0.02 | |
| Upper Moutere, Nelson, 2006 (Taylor's Gold) 050074-05 | SC | 2.5 | 38–41 | 4 | 154 | 0 | 0.032 | ND | - | ND | 0.042 | 050074 |
| | | | | | | 3 | 0.026 | ND | - | ND | 0.036 | |
| | | | | | | 7 | 0.015 | ND | - | ND | 0.025 | |
| | | | | | | 14 | 0.015 | ND | - | ND | 0.025 | |
| | | | | | | 21 | < 0.01 | ND | - | ND | < 0.02 | |
| | | | | | | 28 | < 0.01 | ND | - | ND | < 0.02 | |
| | | | | | | 36 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 42 | ND | ND | - | ND | < 0.02 | |
| Upper Moutere, Nelson, 2006 (Taylor's Gold) 050074-05 | SC | 3.72 | 56–60 | 4 | 233 | 0 | 0.043 | < 0.01 | - | ND | 0.053 | 050074 |
| | | | | | | 3 | 0.053 | < 0.01 | - | ND | 0.063 | |
| | | | | | | 7 | 0.041 | ND | - | ND | 0.051 | |
| | | | | | | 14 | 0.023 | ND | - | ND | 0.033 | |
| | | | | | | 21 | 0.018 | ND | - | ND | 0.028 | |
| | | | | | | 28 | 0.017 | ND | - | ND | 0.027 | |
| | | | | | | 36 | 0.012 | ND | - | ND | 0.022 | |
| | | | | | | 42 | < 0.01 | ND | - | ND | < 0.02 | |
| Upper Moutere, Nelson, 2006 (Taylor's Gold) 050074-05 | SC | 5 | 76–81 | 4 | 315 | 0 | 0.064 | < 0.01 | - | ND | 0.074 | 050074 |
| | | | | | | 3 | 0.062 | 0.011 | - | ND | 0.083 | |
| | | | | | | 7 | 0.035 | ND | - | ND | 0.045 | |
| | | | | | | 14 | 0.041 | < 0.01 | - | ND | 0.051 | |
| | | | | | | 21 | 0.027 | ND | - | ND | 0.037 | |
| | | | | | | 28 | 0.019 | ND | - | ND | 0.029 | |
| | | | | | | 36 | 0.016 | ND | - | ND | 0.026 | |
| | | | | | | 42 | < 0.01 | ND | - | ND | < 0.02 | |

| Location, year (Variety) | Form | Application | | | Total/ season, g ai/ha | PHI, days | Residue, mg/kg | | | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | ND-J a | NF-J | Total b | |
| Upper Moutere, Nelson, 2006 (Taylor's Gold) 050074-05 | SC | 7.6 | 115–121 | 4 | 472 | 0 | 0.096 | 0.017 | - | ND | 0.123 | 050074 |
| | | | | | | 3 | 0.066 | 0.010 | - | ND | 0.086 | |
| | | | | | | 7 | 0.056 | < 0.01 | - | ND | 0.066 | |
| | | | | | | 14 | 0.037 | ND | - | ND | 0.047 | |
| | | | | | | 21 | 0.049 | < 0.01 | - | ND | 0.059 | |
| | | | | | | 28 | 0.027 | ND | - | ND | 0.037 | |
| | | | | | | 36 | 0.013 | ND | - | ND | 0.023 | |
| | | | | | | 42 | 0.010 | ND | - | ND | 0.020 | |

[a] Not determined.

[b] Total residue = XDE-175-J + XDE-175-L + ND-J + NF-J

*Stone Fruits*

*Supervised trials on cherry, peach, apricot and nectarine in Australia*

Trials on cherries (8), peaches (7), nectarines (4) and apricots (4) were conducted during the 2005 to 2006 season in Australia (Cowles, 2006; Report 050063).

Trials were conducted in six sites (two sites for cherries, two for peaches, and one each for apricots and nectarines), each site consisting of one untreated and four treated plots. Each plot, which had 1 to 4 trees ranging from flowering to 7 cm diameter fruits, was treated with 4 or 7 applications of a WG formulation of spinetoram containing 250 g ai/kg. Two treatment regimes were used. The first treatment schedule consisted of three applications at 5-day intervals over flowering followed by four applications at 14-day intervals commencing 63 days before harvest so that the last application was 21 days before harvest. The second treatment schedule consisted of four applications at 14-day intervals commencing 63 days before harvest so that the last application was 21 days before harvest. Treatments were made as foliar spray applications at rates of 5 g ai/hL or 7.5 g ai/hL of spinetoram.

Approximately 2 kg each of whole peaches, apricots, and nectarines and 0.5 kg of cherries were collected 0, 7, 14 or 15, 21 or 22, 28 and 35 days after the last application. Samples were placed in plastic bags, labelled, and frozen at -20°C until analysed in June 2006 to August 2006.

Samples were analysed for residues of spinetoram and the metabolites using the method GRM 05.03. The LOQ for the method was 0.01 mg/kg. The LOD was 0.005 mg/kg. Recoveries were within the acceptable range of 70–120% and relative standard deviation < 20%, with the exception of apricots. The mean recovery of XDE-175-J from apricots fortified at the LOQ (66%) was lower than 70%.

*Supervised trials on cherries, peach and apricot in New Zealand*

Trials on cherries (12), peach (8), and apricot (8) were conducted during the 2005 to 2006 season in New Zealand (Cowles, 2006; Report 050075).

Trials were conducted in seven sites (three sites for cherries, two for peaches, and two for apricots), each site consisting of one untreated and four treated plots. Each peach and apricot site had 4 trees while the cherry plots had 2 trees each. Four applications of a SC formulation of spinetoram containing 120 g ai/L were made to each plot using high volume spray applications at rates of 2.5 g ai/hL, 3.7 g ai/hL, 5 g ai/hL, or 7.6 g ai/ha. Treatments were made at intervals of 13–15 days to the stone fruit plots containing fruits ranging from 1.1 cm to 5 cm in diameter at the first application to 1.2 cm to 6.5 cm in diameter at the fourth application.

Approximately 2 kg each of whole peaches and apricots, and 1 kg of cherries were collected by hand 0, 1, 3, 7, 14, 21, and 28 days after the last application. Samples were placed in plastic bags, labelled, and frozen at -20 °C until analysed.

Spinetoram and its metabolites were analysed using method GRM 05.03 with an LOQ of 0.01 mg/kg. The LOD was determined to be 0.005 mg/kg.

Table 26-1 Residues of spinetoram from supervised trials on cherry in Australia and New Zealand (for estimation of maximum residue level.

| Location, year (Variety) | Form | Application | | | Total/ season, g ai/ha | PHI, days | Residue, mg/kg | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | Total [a] | |
| AUSTRALIAN TRIALS | | | | | | | | | | |
| Sheffield Rd, Spreyton TAS 2005 (Van) 050063-01 | WG | 5 | 54–92 | 4 | 271 | 0 | 0.05 | 0.01 | 0.06 | 050063 |
| | | | | | | 7 | < 0.01 | ND | < 0.01 | |
| | | | | | | 14 | < 0.01 | ND | < 0.01 | |
| | | | | | | 21 | < 0.01 | ND | < 0.01 | |
| | | | | | | 28 | < 0.01 | ND | < 0.01 | |
| Sheffield Rd, Spreyton TAS 2005 (Van) 050063-01 | WG | 7.5 | 93-125 | 4 | 432 | 0 | 0.09 | 0.03 | 0.12 | 050063 |
| | | | | | | 7 | 0.02 | ND | 0.02 | |
| | | | | | | 14 | ND | ND | < 0.01 | |
| | | | | | | 21 | < 0.01 | ND | < 0.01 | |
| | | | | | | 28 | < 0.01 | ND | < 0.01 | |
| Sheffield Rd, Spreyton TAS 2005 (Van) 050063-01 | WG | 5 | 54–92 | 7 | 471 | 0 | 0.14 | 0.04 | 0.18 | 050063 |
| | | | | | | 7 | 0.02 | ND | 0.02 | |
| | | | | | | 14 | 0.01 | ND | 0.01 | |
| | | | | | | 21 | ND | ND | < 0.01 | |
| | | | | | | 28 | < 0.01 | ND | < 0.01 | |
| Sheffield Rd, Spreyton TAS 2005 (Van) 050063-01 | WG | 7.5 | 93–125 | 7 | 758 | 0 | 0.13 | 0.04 | 0.17 | 050063 |
| | | | | | | 7 | 0.02 | ND | 0.02 | |
| | | | | | | 14 | < 0.01 | ND | < 0.01 | |
| | | | | | | 21 | < 0.01 | ND | < 0.01 | |
| | | | | | | 28 | < 0.01 | ND | < 0.01 | |
| Wilgro Orchards, Tumut Road, Batlow, NSW, 2005 (Stella) 050063-02 | WG | 5 | 70–90 | 4 | 330 | 7 | < 0.01 | ND | < 0.01 | 050063 |
| | | | | | | 14 | ND | ND | < 0.01 | |
| | | | | | | 21 | ND | ND | < 0.01 | |
| | | | | | | 28 | ND | ND | < 0.01 | |
| | | | | | | 35 | ND | ND | < 0.01 | |
| Wilgro Orchards, Tumut Road, Batlow, NSW, 2005 (Stella) 050063-02 | WG | 7.5 | 105–135 | 4 | 495 | 7 | 0.01 | ND | 0.01 | 050063 |
| | | | | | | 14 | < 0.01 | ND | < 0.01 | |
| | | | | | | 21 | ND | ND | < 0.01 | |
| | | | | | | 28 | ND | ND | < 0.01 | |
| | | | | | | 35 | ND | ND | < 0.01 | |
| Wilgro Orchards, Tumut Road, Batlow, NSW 2005 (Stella) 050063-02 | WG | 5 | 60–90 | 7 | 560 | 7 | < 0.01 | ND | < 0.01 | 050063 |
| | | | | | | 14 | ND | ND | < 0.01 | |
| | | | | | | 21 | ND | ND | < 0.01 | |
| | | | | | | 28 | ND | ND | < 0.01 | |
| | | | | | | 35 | ND | ND | < 0.01 | |

| Location, year (Variety) | Form | Application | | | Total/ season, g ai/ha | PHI, days | Residue, mg/kg | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | Total [a] | |
| Wilgro Orchards, Tumut Road, Batlow, NSW, 2005 (Stella) 050063-02 | WG | 7.5 | 90–135 | 7 | 694 | 7 | < 0.01 | ND | < 0.01 | 050063 |
| | | | | | | 14 | < 0.01 | ND | < 0.01 | |
| | | | | | | 21 | ND | ND | < 0.01 | |
| | | | | | | 28 | ND | ND | < 0.01 | |
| | | | | | | 35 | ND | ND | < 0.01 | |
| NEW ZEALAND TRIALS | | | | | | | | | | |
| Tukituki, Hawke's Bay, 2005 (Stella) 050075-01 | SC | 2.5 | 38–40 | 4 | 157 | 0 | 0.019 | < 0.01 | 0.019 | 050075 |
| | | | | | | 1 | 0.018 | ND | 0.018 | |
| | | | | | | 3 | < 0.01 | ND | < 0.01 | |
| | | | | | | 7 | < 0.01 | ND | < 0.01 | |
| | | | | | | 14 | ND | ND | < 0.01 | |
| Tukituki, Hawke's Bay, 2005 (Stella) 050075-01 | SC | 3.7 | 57–58 | 4 | 231 | 0 | 0.041 | 0.011 | 0.052 | 050075 |
| | | | | | | 1 | 0.025 | < 0.01 | 0.025 | |
| | | | | | | 3 | 0.018 | ND | 0.018 | |
| | | | | | | 7 | < 0.01 | ND | < 0.01 | |
| | | | | | | 14 | ND | ND | < 0.01 | |
| | | | | | | 21 | ND | ND | < 0.01 | |
| Tukituki, Hawke's Bay, 2005 (Stella) 050075-01 | SC | 5 | 78–79 | 4 | 313 | 0 | 0.069 | 0.019 | 0.088 | 050075 |
| | | | | | | 1 | 0.048 | 0.013 | 0.061 | |
| | | | | | | 3 | 0.036 | < 0.01 | 0.036 | |
| | | | | | | 7 | 0.018 | ND | 0.018 | |
| | | | | | | 14 | < 0.01 | ND | < 0.01 | |
| | | | | | | 21 | < 0.01 | ND | < 0.01 | |
| Tukituki, Hawke's Bay, 2005 (Stella) 050075-01 | SC | 7.6 | 116–119 | 4 | 469 | 0 | 0.136 | 0.037 | 0.173 | 050075 |
| | | | | | | 1 | 0.092 | 0.024 | 0.116 | |
| | | | | | | 3 | 0.029 | < 0.01 | 0.029 | |
| | | | | | | 7 | 0.045 | < 0.01 | 0.045 | |
| | | | | | | 14 | 0.028 | ND | 0.028 | |
| | | | | | | 21 | < 0.01 | ND | < 0.01 | |
| Alexandra, Central Otago, 2006 (Lapins) 050075-04 | SC | 2.5 | 35–41 | 4 | 156 | 0 | 0.030 | < 0.01 | 0.030 | 050075 |
| | | | | | | 1 | 0.028 | < 0.01 | 0.028 | |
| | | | | | | 3 | 0.019 | ND | 0.019 | |
| | | | | | | 7 | 0.019 | ND | 0.019 | |
| | | | | | | 14 | ND | ND | < 0.01 | |
| | | | | | | 21 | ND | ND | < 0.01 | |
| | | | | | | 28 | ND | ND | < 0.01 | |
| Alexandra, Central Otago, 2006 (Lapins) 050075-04 | SC | 3.7 | 52–61 | 4 | 231 | 0 | 0.060 | 0.015 | 0.075 | 050075 |
| | | | | | | 1 | 0.032 | < 0.01 | 0.032 | |
| | | | | | | 3 | 0.019 | < 0.01 | 0.019 | |
| | | | | | | 7 | 0.019 | ND | 0.019 | |
| | | | | | | 14 | < 0.01 | ND | < 0.01 | |
| | | | | | | 21 | < 0.01 | ND | < 0.01 | |
| | | | | | | 28 | < 0.01 | ND | < 0.01 | |

| Location, year (Variety) | Form | Application | | | Total/ season, g ai/ha | PHI, days | Residue, mg/kg | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | Total [a] | |
| Alexandra, Central Otago, 2006 (Lapins) 050075-04 | SC | 5 | 70–82 | 4 | 311 | 0 | 0.102 | 0.027 | 0.129 | 050075 |
| | | | | | | 1 | 0.064 | 0.016 | 0.080 | |
| | | | | | | 3 | 0.045 | 0.012 | 0.057 | |
| | | | | | | 7 | 0.054 | 0.013 | 0.067 | |
| | | | | | | 14 | 0.015 | ND | 0.015 | |
| | | | | | | 21 | 0.013 | ND | 0.013 | |
| | | | | | | 28 | < 0.01 | ND | < 0.01 | |
| Alexandra, Central Otago, 2006 (Lapins) 050075-04 | SC | 7.6 | 105–123 | 4 | 467 | 0 | 0.139 | 0.032 | 0.171 | 050075 |
| | | | | | | 1 | 0.100 | 0.026 | 0.126 | |
| | | | | | | 3 | 0.044 | 0.011 | 0.055 | |
| | | | | | | 7 | 0.059 | 0.013 | 0.072 | |
| | | | | | | 14 | 0.021 | < 0.01 | 0.021 | |
| | | | | | | 21 | 0.014 | ND | 0.014 | |
| | | | | | | 28 | < 0.01 | ND | < 0.01 | |
| Earnscleugh, Central Otago 2005 (Stella) 050075-05 | SC | 2.5 | 41–49 | 4 | 187 | 0 | 0.040 | 0.012 | 0.052 | 050075 |
| | | | | | | 1 | 0.039 | 0.011 | 0.050 | |
| | | | | | | 3 | 0.025 | < 0.01 | 0.025 | |
| | | | | | | 7 | 0.024 | < 0.01 | 0.024 | |
| | | | | | | 14 | 0.009 | ND | 0.009 | |
| | | | | | | 21 | 0.010 | ND | 0.01 | |
| | | | | | | 28 | ND | ND | < 0.01 | |
| Earnscleugh, Central Otago 2005 (Stella) 050075-05 | SC | 3.7 | 60–73 | 4 | 275 | 0 | 0.065 | 0.021 | 0.086 | 050075 |
| | | | | | | 1 | 0.055 | 0.019 | 0.074 | |
| | | | | | | 3 | 0.029 | < 0.01 | 0.029 | |
| | | | | | | 7 | 0.023 | < 0.01 | 0.023 | |
| | | | | | | 14 | < 0.01 | ND | < 0.01 | |
| | | | | | | 21 | < 0.01 | ND | < 0.01 | |
| | | | | | | 28 | < 0.01 | ND | < 0.01 | |
| Earnscleugh, Central Otago 2005 (Stella) 050075-05 | SC | 5 | 81–99 | 4 | 374 | 0 | 0.075 | 0.023 | 0.098 | 050075 |
| | | | | | | 1 | 0.089 | 0.028 | 0.117 | |
| | | | | | | 3 | 0.047 | 0.011 | 0.058 | |
| | | | | | | 7 | 0.025 | < 0.01 | 0.025 | |
| | | | | | | 14 | 0.024 | < 0.01 | 0.024 | |
| | | | | | | 21 | < 0.01 | ND | < 0.01 | |
| | | | | | | 28 | ND | ND | < 0.01 | |
| Earnscleugh, Central Otago 2005 (Stella) 050075-05 | SC | 7.6 | 122–148 | 4 | 560 | 0 | 0.103 | 0.035 | 0.138 | 050075 |
| | | | | | | 1 | 0.053 | 0.017 | 0.070 | |
| | | | | | | 3 | 0.059 | 0.017 | 0.076 | |
| | | | | | | 7 | 0.052 | 0.015 | 0.067 | |
| | | | | | | 14 | < 0.01 | < 0.01 | < 0.01 | |
| | | | | | | 21 | 0.013 | ND | 0.013 | |
| | | | | | | 28 | 0.010 | ND | 0.010 | |

[a] Total residue = XDE-175-J + XDE-175-L.

Table 26 Residues of spinetoram and metabolites from supervised trials on cherry in Australia and New Zealand (for estimation of STMR)

| Location, year (Variety) | Form | Application | | | Total/ season, g ai/ha | PHI, days | Residue, mg/kg | | | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | ND-J | NF-J | Total [a] | |
| AUSTRALIAN TRIALS | | | | | | | | | | | | |
| Sheffield Rd, Spreyton TAS 2005 (Van) 050063-01 | WG | 5 | 54–92 | 4 | 271 | 0 | 0.05 | 0.01 | ND | < 0.01 | 0.07 | 050063 |
| | | | | | | 7 | < 0.01 | ND | ND | < 0.01 | < 0.02 | |
| | | | | | | 14 | < 0.01 | ND | ND | < 0.01 | < 0.02 | |
| | | | | | | 21 | < 0.01 | ND | ND | < 0.01 | < 0.02 | |
| | | | | | | 28 | < 0.01 | ND | ND | 0.010 | 0.020 | |
| Sheffield Rd, Spreyton TAS 2005 (Van) 050063-01 | WG | 7.5 | 93-125 | 4 | 432 | 0 | 0.09 | 0.03 | ND | 0.02 | 0.14 | 050063 |
| | | | | | | 7 | 0.02 | ND | < 0.01 | 0.03 | 0.05 | |
| | | | | | | 14 | ND | ND | ND | < 0.01 | < 0.02 | |
| | | | | | | 21 | < 0.01 | ND | ND | < 0.01 | < 0.02 | |
| | | | | | | 28 | < 0.01 | ND | ND | < 0.01 | < 0.02 | |
| Sheffield Rd, Spreyton TAS 2005 (Van) 050063-01 | WG | 5 | 54–92 | 7 | 471 | 0 | 0.14 | 0.04 | < 0.01 | < 0.01 | 0.19 | 050063 |
| | | | | | | 7 | 0.02 | ND | ND | 0.01 | 0.03 | |
| | | | | | | 14 | 0.01 | ND | ND | 0.01 | 0.02 | |
| | | | | | | 21 | ND | ND | ND | ND | < 0.02 | |
| | | | | | | 28 | < 0.01 | ND | ND | ND | < 0.02 | |
| Sheffield Rd, Spreyton TAS 2005 (Van) 050063-01 | WG | 7.5 | 93–125 | 7 | 758 | 0 | 0.13 | 0.04 | < 0.01 | 0.02 | 0.19 | 050063 |
| | | | | | | 7 | 0.02 | ND | ND | 0.02 | 0.04 | |
| | | | | | | 14 | < 0.01 | ND | ND | < 0.01 | < 0.02 | |
| | | | | | | 21 | < 0.01 | ND | ND | < 0.01 | < 0.02 | |
| | | | | | | 28 | < 0.01 | ND | ND | < 0.01 | < 0.02 | |
| Wilgro Orchards, Tumut Road, Batlow, NSW, 2005 (Stella) 050063-02 | WG | 5 | 70–90 | 4 | 330 | 7 | < 0.01 | ND | ND | ND | < 0.02 | 050063 |
| | | | | | | 14 | ND | ND | ND | ND | < 0.02 | |
| | | | | | | 21 | ND | ND | ND | ND | < 0.02 | |
| | | | | | | 28 | ND | ND | ND | ND | < 0.02 | |
| | | | | | | 35 | ND | ND | ND | ND | < 0.02 | |
| Wilgro Orchards, Tumut Road, Batlow, NSW, 2005 (Stella) 050063-02 | WG | 7.5 | 105–135 | 4 | 495 | 7 | 0.01 | ND | ND | ND | 0.02 | 050063 |
| | | | | | | 14 | < 0.01 | ND | ND | ND | < 0.02 | |
| | | | | | | 21 | ND | ND | ND | ND | < 0.02 | |
| | | | | | | 28 | ND | ND | ND | ND | < 0.02 | |
| | | | | | | 35 | ND | ND | ND | ND | < 0.02 | |
| Wilgro Orchards, Tumut Road, Batlow, NSW 2005 (Stella) 050063-02 | WG | 5 | 60–90 | 7 | 560 | 7 | < 0.01 | ND | ND | ND | < 0.02 | 050063 |
| | | | | | | 14 | ND | ND | ND | ND | < 0.02 | |
| | | | | | | 21 | ND | ND | ND | ND | < 0.02 | |
| | | | | | | 28 | ND | ND | ND | ND | < 0.02 | |
| | | | | | | 35 | ND | ND | ND | ND | < 0.02 | |
| Wilgro Orchards, Tumut Road, Batlow, NSW, 2005 (Stella) 050063-02 | WG | 7.5 | 90–135 | 7 | 694 | 7 | < 0.01 | ND | < 0.01 | 0.01 | 0.02 | 050063 |
| | | | | | | 14 | < 0.01 | ND | ND | ND | < 0.02 | |
| | | | | | | 21 | ND | ND | ND | ND | < 0.02 | |
| | | | | | | 28 | ND | ND | ND | ND | < 0.02 | |
| | | | | | | 35 | ND | ND | ND | ND | < 0.02 | |

| Location, year (Variety) | Form | Application | | | Total/ season, g ai/ha | PHI, days | Residue, mg/kg | | | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | ND-J | NF-J | Total [a] | |
| NEW ZEALAND TRIALS | | | | | | | | | | | | |
| Tukituki, Hawke's Bay, 2005 (Stella) 050075-01 | SC | 2.5 | 38–40 | 4 | 157 | 0 | 0.019 | < 0.01 | - | < 0.01 | 0.029 | 050075 |
| | | | | | | 1 | 0.018 | ND | - | 0.012 | 0.030 | |
| | | | | | | 3 | < 0.01 | ND | - | 0.013 | 0.023 | |
| | | | | | | 7 | < 0.01 | ND | - | < 0.01 | < 0.02 | |
| | | | | | | 14 | ND | ND | - | < 0.01 | < 0.02 | |
| Tukituki, Hawke's Bay, 2005 (Stella) 050075-01 | SC | 3.7 | 57–58 | 4 | 231 | 0 | 0.041 | 0.011 | - | 0.013 | 0.065 | 050075 |
| | | | | | | 1 | 0.025 | < 0.01 | - | 0.014 | 0.039 | |
| | | | | | | 3 | 0.018 | ND | - | 0.018 | 0.036 | |
| | | | | | | 7 | < 0.01 | ND | - | 0.013 | 0.023 | |
| | | | | | | 14 | ND | ND | - | < 0.01 | < 0.02 | |
| | | | | | | 21 | ND | ND | - | ND | < 0.02 | |
| Tukituki, Hawke's Bay, 2005 (Stella) 050075-01 | SC | 5 | 78–79 | 4 | 313 | 0 | 0.069 | 0.019 | - | 0.024 | 0.112 | 050075 |
| | | | | | | 1 | 0.048 | 0.013 | - | 0.019 | 0.080 | |
| | | | | | | 3 | 0.036 | < 0.01 | - | 0.026 | 0.062 | |
| | | | | | | 7 | 0.018 | ND | - | 0.028 | 0.046 | |
| | | | | | | 14 | < 0.01 | ND | - | 0.019 | 0.029 | |
| | | | | | | 21 | < 0.01 | ND | - | < 0.01 | < 0.02 | |
| Tukituki, Hawke's Bay, 2005 (Stella) 050075-01 | SC | 7.6 | 116–119 | 4 | 469 | 0 | 0.136 | 0.037 | - | 0.027 | 0.200 | 050075 |
| | | | | | | 1 | 0.092 | 0.024 | - | 0.020 | 0.136 | |
| | | | | | | 3 | 0.029 | < 0.01 | - | 0.021 | 0.050 | |
| | | | | | | 7 | 0.045 | < 0.01 | - | 0.027 | 0.072 | |
| | | | | | | 14 | 0.028 | ND | - | 0.032 | 0.060 | |
| | | | | | | 21 | < 0.01 | ND | - | 0.010 | 0.020 | |
| Alexandra, Central Otago, 2006 (Lapins) 050075-04 | SC | 2.5 | 35–41 | 4 | 156 | 0 | 0.030 | < 0.01 | - | 0.015 | 0.045 | 050075 |
| | | | | | | 1 | 0.028 | < 0.01 | - | 0.018 | 0.046 | |
| | | | | | | 3 | 0.019 | ND | - | 0.025 | 0.044 | |
| | | | | | | 7 | 0.019 | ND | - | 0.022 | 0.041 | |
| | | | | | | 14 | ND | ND | - | < 0.01 | < 0.02 | |
| | | | | | | 21 | ND | ND | - | < 0.01 | < 0.02 | |
| | | | | | | 28 | ND | ND | - | < 0.01 | < 0.02 | |
| Alexandra, Central Otago, 2006 (Lapins) 050075-04 | SC | 3.7 | 52–61 | 4 | 231 | 0 | 0.060 | 0.015 | - | 0.024 | 0.099 | 050075 |
| | | | | | | 1 | 0.032 | < 0.01 | - | 0.019 | 0.051 | |
| | | | | | | 3 | 0.019 | < 0.01 | - | 0.020 | 0.039 | |
| | | | | | | 7 | 0.019 | ND | - | 0.018 | 0.037 | |
| | | | | | | 14 | < 0.01 | ND | - | 0.012 | 0.022 | |
| | | | | | | 21 | < 0.01 | ND | - | < 0.01 | < 0.02 | |
| | | | | | | 28 | < 0.01 | ND | - | < 0.01 | < 0.02 | |
| Alexandra, Central Otago, 2006 (Lapins) 050075-04 | SC | 5 | 70–82 | 4 | 311 | 0 | 0.102 | 0.027 | - | 0.029 | 0.158 | 050075 |
| | | | | | | 1 | 0.064 | 0.016 | - | 0.036 | 0.116 | |
| | | | | | | 3 | 0.045 | 0.012 | - | 0.032 | 0.089 | |
| | | | | | | 7 | 0.054 | 0.013 | - | 0.037 | 0.104 | |
| | | | | | | 14 | 0.015 | ND | - | 0.016 | 0.031 | |

| Location, year (Variety) | Form | Application | | | Total/ season, g ai/ha | PHI, days | Residue, mg/kg | | | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | ND-J | NF-J | Total [a] | |
| | | | | | | 21 | 0.013 | ND | - | 0.017 | 0.030 | |
| | | | | | | 28 | < 0.01 | ND | - | 0.013 | 0.023 | |
| Alexandra, Central Otago, 2006 (Lapins) 050075-04 | SC | 7.6 | 105–123 | 4 | 467 | 0 | 0.139 | 0.032 | - | 0.033 | 0.204 | 050075 |
| | | | | | | 1 | 0.100 | 0.026 | - | 0.027 | 0.153 | |
| | | | | | | 3 | 0.044 | 0.011 | - | 0.017 | 0.072 | |
| | | | | | | 7 | 0.059 | 0.013 | - | 0.037 | 0.109 | |
| | | | | | | 14 | 0.021 | < 0.01 | - | 0.026 | 0.047 | |
| | | | | | | 21 | 0.014 | ND | - | 0.018 | 0.032 | |
| | | | | | | 28 | < 0.01 | ND | - | 0.016 | 0.026 | |
| Earnscleugh, Central Otago 2005 (Stella) 050075-05 | SC | 2.5 | 41–49 | 4 | 187 | 0 | 0.040 | 0.012 | - | 0.018 | 0.070 | 050075 |
| | | | | | | 1 | 0.039 | 0.011 | - | 0.020 | 0.070 | |
| | | | | | | 3 | 0.025 | < 0.01 | - | 0.016 | 0.041 | |
| | | | | | | 7 | 0.024 | < 0.01 | - | 0.014 | 0.038 | |
| | | | | | | 14 | 0.009 | ND | - | 0.015 | 0.024 | |
| | | | | | | 21 | 0.010 | ND | - | 0.016 | 0.026 | |
| | | | | | | 28 | ND | ND | - | ND | < 0.02 | |
| Earnscleugh, Central Otago 2005 (Stella) 050075-05 | SC | 3.7 | 60–73 | 4 | 275 | 0 | 0.065 | 0.021 | - | 0.024 | 0.110 | 050075 |
| | | | | | | 1 | 0.055 | 0.019 | - | 0.026 | 0.100 | |
| | | | | | | 3 | 0.029 | < 0.01 | - | 0.019 | 0.048 | |
| | | | | | | 7 | 0.023 | < 0.01 | - | 0.034 | 0.057 | |
| | | | | | | 14 | < 0.01 | ND | - | 0.016 | 0.026 | |
| | | | | | | 21 | < 0.01 | ND | - | 0.019 | 0.029 | |
| | | | | | | 28 | < 0.01 | ND | - | < 0.01 | < 0.02 | |
| Earnscleugh, Central Otago 2005 (Stella) 050075-05 | SC | 5 | 81–99 | 4 | 374 | 0 | 0.075 | 0.023 | - | 0.022 | 0.120 | 050075 |
| | | | | | | 1 | 0.089 | 0.028 | - | 0.041 | 0.158 | |
| | | | | | | 3 | 0.047 | 0.011 | - | 0.020 | 0.078 | |
| | | | | | | 7 | 0.025 | < 0.01 | - | 0.023 | 0.048 | |
| | | | | | | 14 | 0.024 | < 0.01 | - | 0.023 | 0.047 | |
| | | | | | | 21 | < 0.01 | ND | - | 0.015 | 0.025 | |
| | | | | | | 28 | ND | ND | - | ND | < 0.02 | |
| Earnscleugh, Central Otago 2005 (Stella) 050075-05 | SC | 7.6 | 122–148 | 4 | 560 | 0 | 0.103 | 0.035 | - | 0.027 | 0.165 | 050075 |
| | | | | | | 1 | 0.053 | 0.017 | - | 0.024 | 0.094 | |
| | | | | | | 3 | 0.059 | 0.017 | - | 0.027 | 0.103 | |
| | | | | | | 7 | 0.052 | 0.015 | - | 0.024 | 0.091 | |
| | | | | | | 14 | < 0.01 | < 0.01 | - | 0.015 | 0.025 | |
| | | | | | | 21 | 0.013 | ND | - | 0.024 | 0.037 | |
| | | | | | | 28 | 0.010 | ND | - | 0.015 | 0.025 | |

[a] Total residue = XDE-175-J + XDE-175-L + ND-J + NF-J.

Table 27-1 Residues of spinetoram from supervised trials on peach in Australia and New Zealand (for estimation of maximum residue level)

| Location, year (Variety) | Form | Application | | | Total/ season, g ai/ha | PHI, days | Residue, mg/kg | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | Total [a] | |
| AUSTRALIAN TRIALS | | | | | | | | | | |
| McIssacs Rd, Ardmona VIC 2005 (Zea Lady) 050063-03 | SC | 5 | 62 | 4 | 248 | 0 | 0.05 | 0.01 | 0.06 | 050063 |
| | | | | | | 7 | 0.02 | < 0.01 | 0.02 | |
| | | | | | | 14 | < 0.01 | ND | < 0.01 | |
| | | | | | | 21 | ND | ND | < 0.01 | |
| | | | | | | 28 | ND | ND | < 0.01 | |
| | | | | | | 35 | ND | ND | < 0.01 | |
| McIssacs Rd, Ardmona VIC 2005 (Zea Lady) 050063-03 | SC | 7.5 | 93 | 4 | 372 | 0 | 0.03 | < 0.01 | 0.03 | 050063 |
| | | | | | | 7 | 0.02 | ND | 0.02 | |
| | | | | | | 14 | < 0.01 | ND | < 0.01 | |
| | | | | | | 21 | ND | ND | < 0.01 | |
| | | | | | | 28 | ND | ND | < 0.01 | |
| | | | | | | 35 | ND | ND | < 0.01 | |
| McIssacs Rd, Ardmona VIC 2005 (Zea Lady) 050063-03 | SC | 5 | 54–62 | 7 | 415 | 0 | 0.03 | < 0.01 | 0.03 | 050063 |
| | | | | | | 7 | 0.01 | ND | 0.01 | |
| | | | | | | 14 | ND | ND | < 0.01 | |
| | | | | | | 21 | ND | ND | < 0.01 | |
| | | | | | | 28 | ND | ND | < 0.01 | |
| McIssacs Rd, Ardmona VIC 2005 (Zea Lady) 050063-03 | SC | 7.5 | 86–93 | 7 | 637 | 0 | 0.05 | 0.01 | 0.06 | 050063 |
| | | | | | | 7 | 0.01 | ND | 0.01 | |
| | | | | | | 15 | ND | ND | < 0.01 | |
| | | | | | | 22 | ND | ND | < 0.01 | |
| | | | | | | 28 | ND | ND | < 0.01 | |
| | | | | | | 35 | ND | ND | < 0.01 | |
| Neale Road Upper Hermitage SA 2005/2006 (Tasty ee) 050063-04 | SC | 5 | 81–91 | 4 | 360 | 0 | 0.11 | 0.03 | 0.14 | 050063 |
| | | | | | | 7 | 0.02 | ND | 0.02 | |
| | | | | | | 15 | < 0.01 | ND | < 0.01 | |
| | | | | | | 22 | < 0.01 | ND | < 0.01 | |
| | | | | | | 28 | ND | ND | < 0.01 | |
| | | | | | | 35 | ND | ND | < 0.01 | |
| Neale Road Upper Hermitage SA 2005/2006 (Tasty ee) 050063-04 | WG | 7.5 | 121–136 | 4 | 538 | 0 | 0.12 | 0.03 | 0.15 | 050063 |
| | | | | | | 7 | 0.03 | ND | 0.03 | |
| | | | | | | 15 | 0.01 | ND | 0.01 | |
| | | | | | | 22 | 0.01 | ND | 0.01 | |
| | | | | | | 28 | < 0.01 | ND | < 0.01 | |
| | | | | | | 35 | ND | ND | < 0.01 | |
| Neale Road Upper Hermitage SA 2005/2006 (Tasty ee) 050063-04 | WG | 5 | 91–136 | 7 | 729 | 0 | 0.07 | 0.02 | 0.09 | 050063 |
| | | | | | | 7 | 0.02 | ND | 0.02 | |
| | | | | | | 15 | < 0.01 | ND | < 0.01 | |
| | | | | | | 22 | < 0.01 | ND | < 0.01 | |
| | | | | | | 28 | ND | ND | < 0.01 | |
| | | | | | | 35 | ND | ND | < 0.01 | |

| Location, year (Variety) | Form | Application | | | Total/ season, g ai/ha | PHI, days | Residue, mg/kg | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | Total [a] | |
| NEW ZEALAND TRIALS | | | | | | | | | | |
| Havelock North, Hawke's Bay, 2006 (Golden Queen) 050075-02 | SC | 2.5 | 46 | 4 | 184 | 0 | 0.020 | ND | 0.020 | 050075 |
| | | | | | | 1 | 0.010 | ND | 0.010 | |
| | | | | | | 3 | 0.011 | ND | 0.011 | |
| | | | | | | 7 | < 0.01 | ND | < 0.01 | |
| | | | | | | 14 | ND | ND | < 0.01 | |
| | | | | | | 21 | ND | ND | < 0.01 | |
| | | | | | | 28 | ND | ND | < 0.01 | |
| Havelock North, Hawke's Bay, 2006 (Golden Queen) 050075-02 | SC | 3.7 | 67–68 | 4 | 269 | 0 | 0.038 | < 0.01 | 0.038 | 050075 |
| | | | | | | 1 | 0.018 | ND | 0.018 | |
| | | | | | | 3 | 0.020 | ND | 0.020 | |
| | | | | | | 7 | < 0.01 | ND | < 0.01 | |
| | | | | | | 14 | < 0.01 | ND | < 0.01 | |
| | | | | | | 21 | < 0.01 | ND | < 0.01 | |
| | | | | | | 28 | ND | ND | < 0.01 | |
| Havelock North, Hawke's Bay, 2006 (Golden Queen) 050075-02 | SC | 5 | 91–92 | 4 | 365 | 0 | 0.049 | 0.012 | 0.061 | 050075 |
| | | | | | | 1 | 0.032 | < 0.01 | 0.032 | |
| | | | | | | 3 | 0.026 | < 0.01 | 0.026 | |
| | | | | | | 7 | 0.024 | < 0.01 | 0.024 | |
| | | | | | | 14 | < 0.01 | ND | < 0.01 | |
| | | | | | | 21 | ND | ND | < 0.01 | |
| | | | | | | 28 | ND | ND | < 0.01 | |
| Havelock North, Hawke's Bay, 2006 (Golden Queen) 050075-02 | SC | 7.6 | 137–138 | 4 | 549 | 0 | 0.058 | 0.016 | 0.074 | 050075 |
| | | | | | | 1 | 0.041 | < 0.01 | 0.041 | |
| | | | | | | 3 | 0.036 | < 0.01 | 0.036 | |
| | | | | | | 7 | 0.039 | < 0.01 | 0.039 | |
| | | | | | | 14 | 0.023 | ND | 0.023 | |
| | | | | | | 21 | 0.012 | ND | 0.012 | |
| | | | | | | 28 | 0.014 | ND | 0.014 | |
| Earnscleugh, Central Otago 2006 (Southern Ice) 050075-06 | SC | 2.5 | 49-52 | 4 | 199 | 0 | 0.060 | 0.014 | 0.074 | 050075 |
| | | | | | | 1 | 0.094 | 0.024 | 0.118 | |
| | | | | | | 3 | 0.032 | < 0.01 | 0.032 | |
| | | | | | | 7 | 0.013 | ND | 0.013 | |
| | | | | | | 14 | < 0.01 | ND | < 0.01 | |
| | | | | | | 21 | 0.019 | ND | 0.019 | |
| | | | | | | 28 | 0.027 | ND | 0.027 | |
| Earnscleugh, Central Otago 2006 (Southern Ice) 050075-06 | SC | 3.7 | 72–76 | 4 | 293 | 0 | 0.078 | 0.019 | 0.097 | 050075 |
| | | | | | | 1 | 0.053 | 0.012 | 0.065 | |
| | | | | | | 3 | 0.052 | 0.010 | 0.062 | |
| | | | | | | 7 | 0.030 | < 0.01 | 0.030 | |
| | | | | | | 14 | < 0.01 | ND | < 0.01 | |
| | | | | | | 21 | 0.014 | ND | 0.014 | |
| | | | | | | 28 | < 0.01 | ND | < 0.01 | |
| Earnscleugh, | SC | 5 | 98–103 | 4 | 397 | 0 | 0.154 | 0.032 | 0.186 | 050075 |

| Location, year (Variety) | Form | Application | | | Total/ season, g ai/ha | PHI, days | Residue, mg/kg | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | Total [a] | |
| Central Otago 2006 (Southern Ice) 050075-06 | | | | | | 1 | 0.053 | 0.012 | 0.065 | |
| | | | | | | 3 | 0.071 | 0.013 | 0.084 | |
| | | | | | | 7 | 0.039 | < 0.01 | 0.039 | |
| | | | | | | 14 | 0.015 | ND | 0.015 | |
| | | | | | | 21 | 0.014 | ND | 0.014 | |
| | | | | | | 28 | 0.020 | ND | 0.020 | |
| Earnscleugh, Central Otago 2006 (Southern Ice) 050075-06 | SC | 7.6 | 147-155 | 4 | 596 | 0 | 0.106 | 0.027 | 0.133 | 050075 |
| | | | | | | 1 | 0.111 | 0.029 | 0.140 | |
| | | | | | | 3 | 0.088 | 0.014 | 0.102 | |
| | | | | | | 7 | 0.027 | < 0.01 | 0.027 | |
| | | | | | | 14 | 0.019 | ND | 0.019 | |
| | | | | | | 21 | 0.020 | ND | 0.020 | |
| | | | | | | 28 | 0.034 | ND | 0.034 | |

[a] Total residue = XDE-175-J + XDE-175-L.

Table 27-2 Residues of spinetoram and metabolites from supervised trials on peach in Australia and New Zealand (for estimation of STMR)

| Location, year (Variety) | Form | Application | | | Total/ season, g ai/ha | PHI, days | Residue, mg/kg | | | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | ND-J | NF-J | Total [a] | |
| AUSTRALIAN TRIALS | | | | | | | | | | | | |
| McIssacs Rd, Ardmona VIC 2005 (Zea Lady) 050063-03 | SC | 5 | 62 | 4 | 248 | 0 | 0.05 | 0.01 | ND | ND | 0.07 | 050063 |
| | | | | | | 7 | 0.02 | < 0.01 | ND | ND | 0.03 | |
| | | | | | | 14 | < 0.01 | ND | ND | ND | < 0.02 | |
| | | | | | | 21 | ND | ND | ND | ND | < 0.02 | |
| | | | | | | 28 | ND | ND | ND | ND | < 0.02 | |
| | | | | | | 35 | ND | ND | ND | ND | < 0.02 | |
| McIssacs Rd, Ardmona VIC 2005 (Zea Lady) 050063-03 | SC | 7.5 | 93 | 4 | 372 | 0 | 0.03 | < 0.01 | ND | ND | 0.04 | 050063 |
| | | | | | | 7 | 0.02 | ND | ND | ND | 0.03 | |
| | | | | | | 14 | < 0.01 | ND | ND | ND | < 0.02 | |
| | | | | | | 21 | ND | ND | ND | ND | < 0.02 | |
| | | | | | | 28 | ND | ND | ND | ND | < 0.02 | |
| | | | | | | 35 | ND | ND | ND | ND | < 0.02 | |
| McIssacs Rd, Ardmona VIC 2005 (Zea Lady) 050063-03 | SC | 5 | 54–62 | 7 | 415 | 0 | 0.03 | < 0.01 | ND | ND | 0.04 | 050063 |
| | | | | | | 7 | 0.01 | ND | ND | ND | 0.02 | |
| | | | | | | 14 | ND | ND | ND | ND | < 0.02 | |
| | | | | | | 21 | ND | ND | ND | ND | < 0.02 | |
| | | | | | | 28 | ND | ND | ND | ND | < 0.02 | |

| Location, year (Variety) | Form | Application | | | Total/ season, g ai/ha | PHI, days | Residue, mg/kg | | | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | ND-J | NF-J | Total [a] | |
| McIssacs Rd, Ardmona VIC 2005 (Zea Lady) 050063-03 | SC | 7.5 | 86–93 | 7 | 637 | 0 | 0.05 | 0.01 | < 0.01 | < 0.01 | 0.07 | 050063 |
| | | | | | | 7 | 0.01 | ND | ND | ND | 0.02 | |
| | | | | | | 15 | ND | ND | ND | ND | < 0.02 | |
| | | | | | | 22 | ND | ND | ND | ND | < 0.02 | |
| | | | | | | 28 | ND | ND | ND | ND | < 0.02 | |
| | | | | | | 35 | ND | ND | ND | ND | < 0.02 | |
| Neale Road Upper Hermitage SA 2005/2006 (Tasty ee) 050063-04 | SC | 5 | 81–91 | 4 | 360 | 0 | 0.11 | 0.03 | < 0.01 | < 0.01 | 0.15 | 050063 |
| | | | | | | 7 | 0.02 | ND | ND | < 0.01 | 0.03 | |
| | | | | | | 15 | < 0.01 | ND | ND | ND | < 0.02 | |
| | | | | | | 22 | < 0.01 | ND | ND | ND | < 0.02 | |
| | | | | | | 28 | ND | ND | ND | ND | < 0.02 | |
| | | | | | | 35 | ND | ND | ND | ND | < 0.02 | |
| Neale Road Upper Hermitage SA 2005/2006 (Tasty ee) 050063-04 | WG | 7.5 | 121–136 | 4 | 538 | 0 | 0.12 | 0.03 | 0.01 | 0.02 | 0.18 | 050063 |
| | | | | | | 7 | 0.03 | ND | ND | 0.01 | 0.04 | |
| | | | | | | 15 | 0.01 | ND | ND | < 0.01 | 0.02 | |
| | | | | | | 22 | 0.01 | ND | ND | < 0.01 | 0.02 | |
| | | | | | | 28 | < 0.01 | ND | ND | < 0.01 | < 0.02 | |
| | | | | | | 35 | ND | ND | ND | ND | < 0.02 | |
| Neale Road Upper Hermitage SA 2005/2006 (Tasty ee) 050063-04 | WG | 5 | 91–136 | 7 | 729 | 0 | 0.07 | 0.02 | < 0.01 | < 0.01 | 0.010 | 050063 |
| | | | | | | 7 | 0.02 | ND | ND | 0.01 | 0.03 | |
| | | | | | | 15 | < 0.01 | ND | ND | ND | < 0.02 | |
| | | | | | | 22 | < 0.01 | ND | ND | ND | < 0.02 | |
| | | | | | | 28 | ND | ND | ND | ND | < 0.02 | |
| | | | | | | 35 | ND | ND | ND | ND | < 0.02 | |
| NEW ZEALAND TRIALS | | | | | | | | | | | | |
| Havelock North, Hawke's Bay, 2006 (Golden Queen) 050075-02 | SC | 2.5 | 46 | 4 | 184 | 0 | 0.020 | ND | - | 0.011 | 0.031 | 050075 |
| | | | | | | 1 | 0.010 | ND | - | < 0.01 | 0.020 | |
| | | | | | | 3 | 0.011 | ND | - | 0.011 | 0.022 | |
| | | | | | | 7 | < 0.01 | ND | - | < 0.01 | < 0.02 | |
| | | | | | | 14 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 21 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 28 | ND | ND | - | ND | < 0.02 | |
| Havelock North, Hawke's Bay, 2006 (Golden Queen) 050075-02 | SC | 3.7 | 67–68 | 4 | 269 | 0 | 0.038 | < 0.01 | - | 0.013 | 0.051 | 050075 |
| | | | | | | 1 | 0.018 | ND | - | 0.011 | 0.029 | |
| | | | | | | 3 | 0.020 | ND | - | 0.016 | 0.036 | |
| | | | | | | 7 | < 0.01 | ND | - | < 0.01 | < 0.02 | |
| | | | | | | 14 | < 0.01 | ND | - | < 0.01 | < 0.02 | |
| | | | | | | 21 | < 0.01 | ND | - | < 0.01 | < 0.02 | |
| | | | | | | 28 | ND | ND | - | < 0.01 | < 0.02 | |

| Location, year (Variety) | Form | Application | | | Total/ season, g ai/ha | PHI, days | Residue, mg/kg | | | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | ND-J | NF-J | Total [a] | |
| Havelock North, Hawke's Bay, 2006 (Golden Queen) 050075-02 | SC | 5 | 91–92 | 4 | 365 | 0 | 0.049 | 0.012 | - | 0.017 | 0.078 | 050075 |
| | | | | | | 1 | 0.032 | < 0.01 | - | 0.013 | 0.045 | |
| | | | | | | 3 | 0.026 | < 0.01 | - | 0.015 | 0.041 | |
| | | | | | | 7 | 0.024 | < 0.01 | - | 0.015 | 0.039 | |
| | | | | | | 14 | < 0.01 | ND | - | 0.011 | 0.021 | |
| | | | | | | 21 | ND | ND | - | ND | < 0.02 | |
| | | | | | | 28 | ND | ND | - | < 0.01 | < 0.02 | |
| Havelock North, Hawke's Bay, 2006 (Golden Queen) 050075-02 | SC | 7.6 | 137–138 | 4 | 549 | 0 | 0.058 | 0.016 | - | 0.018 | 0.092 | 050075 |
| | | | | | | 1 | 0.041 | < 0.01 | - | 0.016 | 0.057 | |
| | | | | | | 3 | 0.036 | < 0.01 | - | 0.027 | 0.063 | |
| | | | | | | 7 | 0.039 | < 0.01 | - | 0.025 | 0.064 | |
| | | | | | | 14 | 0.023 | ND | - | 0.017 | 0.040 | |
| | | | | | | 21 | 0.012 | ND | - | 0.017 | 0.029 | |
| | | | | | | 28 | 0.014 | ND | - | 0.020 | 0.034 | |
| Earnscleugh, Central Otago 2006 (Southern Ice) 050075-06 | SC | 2.5 | 49-52 | 4 | 199 | 0 | 0.060 | 0.014 | - | 0.022 | 0.096 | 050075 |
| | | | | | | 1 | 0.094 | 0.024 | - | 0.038 | 0.156 | |
| | | | | | | 3 | 0.032 | < 0.01 | - | 0.023 | 0.055 | |
| | | | | | | 7 | 0.013 | ND | - | < 0.01 | 0.023 | |
| | | | | | | 14 | < 0.01 | ND | - | < 0.01 | < 0.02 | |
| | | | | | | 21 | 0.019 | ND | - | < 0.01 | 0.029 | |
| | | | | | | 28 | 0.027 | ND | - | 0.010 | 0.037 | |
| Earnscleugh, Central Otago 2006 (Southern Ice) 050075-06 | SC | 3.7 | 72–76 | 4 | 293 | 0 | 0.078 | 0.019 | - | 0.013 | 0.11 | 050075 |
| | | | | | | 1 | 0.053 | 0.012 | - | 0.020 | 0.085 | |
| | | | | | | 3 | 0.052 | 0.010 | - | 0.025 | 0.087 | |
| | | | | | | 7 | 0.030 | < 0.01 | - | 0.017 | 0.047 | |
| | | | | | | 14 | < 0.01 | ND | - | < 0.01 | < 0.02 | |
| | | | | | | 21 | 0.014 | ND | - | 0.011 | 0.025 | |
| | | | | | | 28 | < 0.01 | ND | - | < 0.01 | < 0.02 | |
| Earnscleugh, Central Otago 2006 (Southern Ice) 050075-06 | SC | 5 | 98–103 | 4 | 397 | 0 | 0.154 | 0.032 | - | 0.041 | 0.227 | 050075 |
| | | | | | | 1 | 0.053 | 0.012 | - | 0.026 | 0.091 | |
| | | | | | | 3 | 0.071 | 0.013 | - | 0.033 | 0.117 | |
| | | | | | | 7 | 0.039 | < 0.01 | - | 0.036 | 0.075 | |
| | | | | | | 14 | 0.015 | ND | - | 0.014 | 0.029 | |
| | | | | | | 21 | 0.014 | ND | - | 0.017 | 0.031 | |
| | | | | | | 28 | 0.020 | ND | - | 0.020 | 0.04 | |
| Earnscleugh, Central Otago 2006 (Southern Ice) 050075-06 | SC | 7.6 | 147-155 | 4 | 596 | 0 | 0.106 | 0.027 | - | 0.017 | 0.150 | 050075 |
| | | | | | | 1 | 0.111 | 0.029 | - | 0.035 | 0.175 | |
| | | | | | | 3 | 0.088 | 0.014 | - | 0.041 | 0.143 | |
| | | | | | | 7 | 0.027 | < 0.01 | - | 0.019 | 0.046 | |
| | | | | | | 14 | 0.019 | ND | - | 0.014 | 0.033 | |
| | | | | | | 21 | 0.020 | ND | - | 0.012 | 0.032 | |
| | | | | | | 28 | 0.034 | ND | - | 0.021 | 0.055 | |

[a] Total residue = XDE-175-J + XDE-175-L + ND-J + NF-J.

Table 28-1 Residues of spinetoram from supervised trials on nectarine in Australia (for estimation of maximum residue level)

| Location, year (Variety) | Form | Application | | | Total/ season, g ai/ha | PHI, days | Residue, mg/kg | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | Total [a] | |
| NECTARINES–AUSTRALIAN TRIALS | | | | | | | | | | |
| McIssacs Rd, Ardmona VIC 2005 (Fire Pearl) 050063-06 | WG | 5 | 62 | 4 | 248 | 0 | 0.01 | ND | 0.02 | 050063 |
| | | | | | | 7 | ND | ND | < 0.02 | |
| | | | | | | 14 | ND | ND | < 0.02 | |
| | | | | | | 21 | ND | ND | < 0.02 | |
| | | | | | | 28 | ND | ND | < 0.02 | |
| | | | | | | 35 | ND | ND | < 0.02 | |
| McIssacs Rd, Ardmona VIC 2005 (Fire Pearl) 050063-06 | WG | 7.5 | 93 | 4 | 372 | 0 | 0.02 | ND | 0.03 | 050063 |
| | | | | | | 7 | < 0.01 | ND | < 0.02 | |
| | | | | | | 14 | ND | ND | < 0.02 | |
| | | | | | | 21 | ND | ND | < 0.02 | |
| | | | | | | 28 | ND | ND | < 0.02 | |
| | | | | | | 35 | ND | ND | < 0.02 | |
| McIssacs Rd, Ardmona VIC 2005 (Fire Pearl) 050063-06 | WG | 5 | 59–62 | 7 | 427 | 0 | 0.02 | < 0.01 | 0.03 | 050063 |
| | | | | | | 7 | < 0.01 | ND | < 0.02 | |
| | | | | | | 14 | ND | ND | < 0.02 | |
| | | | | | | 21 | ND | ND | < 0.02 | |
| | | | | | | 28 | ND | ND | < 0.02 | |
| | | | | | | | | | | |
| McIssacs Rd, Ardmona VIC 2005 (Fire Pearl) 050063-06 | WG | 7.5 | 77–93 | 7 | 629 | 0 | 0.01 | ND | 0.03 | 050063 |
| | | | | | | 7 | < 0.01 | ND | < 0.02 | |
| | | | | | | 14 | ND | ND | < 0.02 | |
| | | | | | | 21 | ND | ND | < 0.02 | |
| | | | | | | 28 | ND | ND | < 0.02 | |
| | | | | | | 35 | ND | ND | < 0.02 | |

[a] Total residue = XDE-175-J + XDE-175-L.

Table 28-2 Residues of spinetoram and metabolites from supervised trials on nectarine in Australia (for estimation of STMR)

| Location, year (Variety) | Form | Application | | | Total/ season, g ai/ha | PHI, days | Residue, mg/kg | | | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | ND-J | NF-J | Total [a] | |
| NECTARINES–AUSTRALIAN TRIALS | | | | | | | | | | | | |
| McIssacs Rd, Ardmona VIC 2005 (Fire Pearl) 050063-06 | WG | 5 | 62 | 4 | 248 | 0 | 0.01 | ND | ND | ND | 0.02 | 050063 |
| | | | | | | 7 | ND | ND | ND | ND | < 0.02 | |
| | | | | | | 14 | ND | ND | ND | ND | < 0.02 | |
| | | | | | | 21 | ND | ND | ND | ND | < 0.02 | |
| | | | | | | 28 | ND | ND | ND | ND | < 0.02 | |

| Location, year (Variety) | Form | Application | | | Total/ season, g ai/ha | PHI, days | Residue, mg/kg | | | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | ND-J | NF-J | Total [a] | |
| NECTARINES–AUSTRALIAN TRIALS | | | | | | | | | | | | |
| | | | | | | 35 | ND | ND | ND | ND | < 0.02 | |
| McIssacs Rd, Ardmona VIC 2005 (Fire Pearl) 050063-06 | WG | 7.5 | 93 | 4 | 372 | 0 | 0.02 | ND | ND | ND | 0.03 | 050063 |
| | | | | | | 7 | < 0.01 | ND | ND | ND | < 0.02 | |
| | | | | | | 14 | ND | ND | ND | ND | < 0.02 | |
| | | | | | | 21 | ND | ND | ND | ND | < 0.02 | |
| | | | | | | 28 | ND | ND | ND | ND | < 0.02 | |
| | | | | | | 35 | ND | ND | ND | ND | < 0.02 | |
| McIssacs Rd, Ardmona VIC 2005 (Fire Pearl) 050063-06 | WG | 5 | 59–62 | 7 | 427 | 0 | 0.02 | < 0.01 | ND | ND | 0.03 | 050063 |
| | | | | | | 7 | < 0.01 | ND | ND | ND | < 0.02 | |
| | | | | | | 14 | ND | ND | ND | ND | < 0.02 | |
| | | | | | | 21 | ND | ND | ND | ND | < 0.02 | |
| | | | | | | 28 | ND | ND | ND | ND | < 0.02 | |
| | | | | | | | | | | | | |
| McIssacs Rd, Ardmona VIC 2005 (Fire Pearl) 050063-06 | WG | 7.5 | 77–93 | 7 | 629 | 0 | 0.01 | ND | ND | ND | 0.03 | 050063 |
| | | | | | | 7 | < 0.01 | ND | ND | ND | < 0.02 | |
| | | | | | | 14 | ND | ND | ND | ND | < 0.02 | |
| | | | | | | 21 | ND | ND | ND | ND | < 0.02 | |
| | | | | | | 28 | ND | ND | ND | ND | < 0.02 | |
| | | | | | | 35 | ND | ND | ND | ND | < 0.02 | |

[a] Total residue = XDE-175-J + XDE-175-L + ND-J + NF-J.

Table 29-1 Residues of spinetoram from supervised trials on apricot in Australia and New Zealand (for estimation of maximum residue level)

| Location, year (Variety) | Form | Application | | | Total/ season, g ai/ha | PHI, days | Residue, mg/kg | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | Total [a] | |
| AUSTRALIAN TRIALS | | | | | | | | | | |
| McIssacs Rd, Ardmona VIC 2005 (Francesco) 050063-05 | WG | 5 | 51 | 4 | 204 | 0 | 0.08 | 0.02 | 0.10 | 050063 |
| | | | | | | 7 | 0.02 | ND | 0.02 | |
| | | | | | | 14 | < 0.01 | < 0.01 | < 0.01 | |
| | | | | | | 21 | < 0.01 | ND | < 0.01 | |
| | | | | | | 28 | ND | ND | < 0.01 | |
| | | | | | | 35 | ND | ND | < 0.01 | |
| McIssacs Rd, Ardmona VIC 2005 (Francesco) 050063-05 | WG | 7.5 | 76 | 4 | 304 | 0 | 0.08 | 0.02 | 0.10 | 050063 |
| | | | | | | 7 | 0.02 | ND | 0.02 | |
| | | | | | | 14 | 0.01 | ND | 0.01 | |
| | | | | | | 21 | < 0.01 | ND | < 0.01 | |
| | | | | | | 28 | < 0.01 | ND | < 0.01 | |
| | | | | | | 35 | ND | ND | < 0.01 | |

| Location, year (Variety) | Form | Application | | | Total/ season, g ai/ha | PHI, days | Residue, mg/kg | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | Total [a] | |
| McIssacs Rd, Ardmona VIC 2005 (Francesco) 050063-05 | WG | 5 | 51–52 | 7 | 358 | 0 | 0.07 | 0.02 | 0.09 | 050063 |
| | | | | | | 7 | 0.02 | ND | 0.02 | |
| | | | | | | 14 | 0.01 | ND | 0.01 | |
| | | | | | | 21 | ND | ND | < 0.01 | |
| | | | | | | 28 | ND | ND | < 0.01 | |
| | | | | | | 35 | ND | ND | < 0.01 | |
| McIssacs Rd, Ardmona VIC 2005 (Francesco) 050063-05 | WG | 7.5 | 76–79 | 7 | 536 | 0 | 0.16 | 0.04 | 0.20 | 050063 |
| | | | | | | 7 | 0.02 | ND | 0.02 | |
| | | | | | | 14 | 0.02 | ND | 0.02 | |
| | | | | | | 21 | 0.01 | ND | 0.01 | |
| | | | | | | 28 | < 0.01 | ND | < 0.01 | |
| | | | | | | 35 | < 0.01 | ND | < 0.01 | |
| NEW ZEALAND TRIALS | | | | | | | | | | |
| Bay View, Hawke's Bay, 2005 (Castlebright) 050075-03 | SC | 2.5 | 31–33 | 4 | 129 | 0 | 0.038 | < 0.01 | 0.038 | 050075 |
| | | | | | | 1 | 0.048 | 0.012 | 0.06 | |
| | | | | | | 3 | 0.031 | < 0.01 | 0.031 | |
| | | | | | | 7 | 0.016 | ND | 0.016 | |
| | | | | | | 14 | 0.010 | ND | 0.01 | |
| Bay View, Hawke's Bay, 2005 (Castlebright) 050075-03 | SC | 3.7 | 46–48 | 4 | 189 | 0 | 0.057 | 0.015 | 0.072 | 050075 |
| | | | | | | 1 | 0.048 | 0.012 | 0.06 | |
| | | | | | | 3 | 0.032 | < 0.01 | 0.032 | |
| | | | | | | 7 | 0.041 | < 0.01 | 0.041 | |
| | | | | | | 14 | 0.014 | ND | 0.014 | |
| Bay View, Hawke's Bay, 2005 (Castlebright) 050075-03 | SC | 5 | 62–65 | 4 | 256 | 0 | 0.080 | 0.019 | 0.099 | 050075 |
| | | | | | | 1 | 0.091 | 0.022 | 0.113 | |
| | | | | | | 3 | 0.063 | 0.015 | 0.078 | |
| | | | | | | 7 | 0.055 | 0.012 | 0.067 | |
| | | | | | | 14 | 0.017 | ND | 0.017 | |
| Bay View, Hawke's Bay, 2005 (Castlebright) 050075-03 | SC | 7.6 | 93–98 | 4 | 385 | 0 | 0.185 | 0.043 | 0.228 | 050075 |
| | | | | | | 1 | 0.208 | 0.052 | 0.26 | |
| | | | | | | 3 | 0.153 | 0.033 | 0.186 | |
| | | | | | | 7 | 0.080 | 0.017 | 0.097 | |
| | | | | | | 14 | 0.030 | < 0.01 | 0.03 | |
| Earnscleugh, Central Otago 2006 (Sundrop) 050075-07 | SC | 2.5 | 36–40 | 4 | 150 | 0 | 0.035 | 0.055 | 0.09 | 050075 |
| | | | | | | 1 | 0.039 | 0.012 | 0.051 | |
| | | | | | | 3 | 0.018 | ND | 0.018 | |
| | | | | | | 7 | 0.019 | ND | 0.019 | |
| | | | | | | 14 | < 0.01 | ND | < 0.01 | |
| Earnscleugh, Central Otago 2006 (Sundrop) 050075-07 | SC | 3.7 | 53–59 | 4 | 221 | 0 | 0.126 | 0.039 | 0.165 | 050075 |
| | | | | | | 1 | 0.124 | 0.037 | 0.161 | |
| | | | | | | 3 | 0.063 | 0.018 | 0.081 | |
| | | | | | | 7 | 0.041 | 0.011 | 0.052 | |
| | | | | | | 14 | 0.022 | < 0.01 | 0.022 | |

| Location, year (Variety) | Form | Application | | | Total/ season, g ai/ha | PHI, days | Residue, mg/kg | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | Total [a] | |
| Earnscleugh, Central Otago 2006 (Sundrop) 050075-07 | SC | 5 | 72–79 | 4 | 298 | 0 | 0.101 | 0.031 | 0.132 | 050075 |
| | | | | | | 1 | 0.080 | 0.023 | 0.103 | |
| | | | | | | 3 | 0.081 | 0.025 | 0.106 | |
| | | | | | | 7 | 0.064 | 0.017 | 0.081 | |
| | | | | | | 14 | 0.024 | ND | 0.024 | |
| Earnscleugh, Central Otago 2006 (Sundrop) 050075-07 | SC | 7.6 | 107–119 | 4 | 447 | 0 | 0.222 | 0.067 | 0.289 | 050075 |
| | | | | | | 1 | 0.213 | 0.062 | 0.275 | |
| | | | | | | 3 | 0.139 | 0.038 | 0.177 | |
| | | | | | | 7 | 0.084 | 0.022 | 0.106 | |
| | | | | | | 14 | 0.034 | < 0.01 | 0.034 | |

[a] Total residue = XDE-175-J + XDE-175-L.

Table 29-2 Residues of spinetoram and metabolites from supervised trials on apricot in Australia and New Zealand (for estimation of STMR)

| Location, year (Variety) | Form | Application | | | Total/ season, g ai/ha | PHI, days | Residue, mg/kg | | | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | ND-J | NF-J | Total [a] | |
| AUSTRALIAN TRIALS | | | | | | | | | | | | |
| McIssacs Rd, Ardmona VIC 2005 (Francesco) 050063-05 | WG | 5 | 51 | 4 | 204 | 0 | 0.08 | 0.02 | 0.01 | < 0.01 | 0.12 | 050063 |
| | | | | | | 7 | 0.02 | ND | 0.01 | < 0.01 | 0.04 | |
| | | | | | | 14 | < 0.01 | < 0.01 | ND | ND | < 0.02 | |
| | | | | | | 21 | < 0.01 | ND | ND | ND | < 0.02 | |
| | | | | | | 28 | ND | ND | ND | ND | < 0.02 | |
| | | | | | | 35 | ND | ND | ND | ND | < 0.02 | |
| McIssacs Rd, Ardmona VIC 2005 (Francesco) 050063-05 | WG | 7.5 | 76 | 4 | 304 | 0 | 0.08 | 0.02 | 0.02 | < 0.01 | 0.13 | 050063 |
| | | | | | | 7 | 0.02 | ND | < 0.01 | < 0.01 | 0.03 | |
| | | | | | | 14 | 0.01 | ND | < 0.01 | < 0.01 | 0.02 | |
| | | | | | | 21 | < 0.01 | ND | ND | ND | < 0.02 | |
| | | | | | | 28 | < 0.01 | ND | ND | ND | < 0.02 | |
| | | | | | | 35 | ND | ND | ND | ND | < 0.02 | |
| McIssacs Rd, Ardmona VIC 2005 (Francesco) 050063-05 | WG | 5 | 51–52 | 7 | 358 | 0 | 0.07 | 0.02 | 0.01 | < 0.01 | 0.11 | 050063 |
| | | | | | | 7 | 0.02 | ND | 0.01 | 0.010 | 0.04 | |
| | | | | | | 14 | 0.01 | ND | < 0.01 | < 0.01 | 0.02 | |
| | | | | | | 21 | ND | ND | ND | ND | < 0.02 | |
| | | | | | | 28 | ND | ND | ND | ND | < 0.02 | |
| | | | | | | 35 | ND | ND | ND | ND | < 0.02 | |

| Location, year (Variety) | Form | Application | | | Total/ season, g ai/ha | PHI, days | Residue, mg/kg | | | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | ND-J | NF-J | Total [a] | |
| McIssacs Rd, Ardmona VIC 2005 (Francesco) 050063-05 | WG | 7.5 | 76–79 | 7 | 536 | 0 | 0.16 | 0.04 | 0.02 | 0.01 | 0.23 | 050063 |
| | | | | | | 7 | 0.02 | ND | 0.010 | < 0.01 | 0.04 | |
| | | | | | | 14 | 0.02 | ND | 0.010 | < 0.01 | 0.04 | |
| | | | | | | 21 | 0.01 | ND | < 0.01 | < 0.01 | 0.02 | |
| | | | | | | 28 | < 0.01 | ND | < 0.01 | < 0.01 | < 0.02 | |
| | | | | | | 35 | < 0.01 | ND | ND | ND | < 0.02 | |
| NEW ZEALAND TRIALS | | | | | | | | | | | | |
| Bay View, Hawke's Bay, 2005 (Castlebright) 050075-03 | SC | 2.5 | 31–33 | 4 | 129 | 0 | 0.038 | < 0.01 | - | 0.018 | 0.056 | 050075 |
| | | | | | | 1 | 0.048 | 0.012 | - | 0.023 | 0.083 | |
| | | | | | | 3 | 0.031 | < 0.01 | - | 0.023 | 0.054 | |
| | | | | | | 7 | 0.016 | ND | - | 0.015 | 0.031 | |
| | | | | | | 14 | 0.010 | ND | - | 0.013 | 0.023 | |
| Bay View, Hawke's Bay, 2005 (Castlebright) 050075-03 | SC | 3.7 | 46–48 | 4 | 189 | 0 | 0.057 | 0.015 | - | 0.020 | 0.092 | 050075 |
| | | | | | | 1 | 0.048 | 0.012 | - | 0.021 | 0.081 | |
| | | | | | | 3 | 0.032 | < 0.01 | - | 0.021 | 0.053 | |
| | | | | | | 7 | 0.041 | < 0.01 | - | 0.028 | 0.069 | |
| | | | | | | 14 | 0.014 | ND | - | 0.017 | 0.031 | |
| Bay View, Hawke's Bay, 2005 (Castlebright) 050075-03 | SC | 5 | 62–65 | 4 | 256 | 0 | 0.080 | 0.019 | - | 0.026 | 0.125 | 050075 |
| | | | | | | 1 | 0.091 | 0.022 | - | 0.036 | 0.149 | |
| | | | | | | 3 | 0.063 | 0.015 | - | 0.032 | 0.11 | |
| | | | | | | 7 | 0.055 | 0.012 | - | 0.042 | 0.109 | |
| | | | | | | 14 | 0.017 | ND | - | 0.020 | 0.037 | |
| Bay View, Hawke's Bay, 2005 (Castlebright) 050075-03 | SC | 7.6 | 93–98 | 4 | 385 | 0 | 0.185 | 0.043 | - | 0.045 | 0.273 | 050075 |
| | | | | | | 1 | 0.208 | 0.052 | - | 0.058 | 0.318 | |
| | | | | | | 3 | 0.153 | 0.033 | - | 0.065 | 0.251 | |
| | | | | | | 7 | 0.080 | 0.017 | - | 0.055 | 0.152 | |
| | | | | | | 14 | 0.030 | < 0.01 | - | 0.030 | 0.06 | |
| Earnscleugh,Central Otago 2006 (Sundrop) 050075-07 | SC | 2.5 | 36–40 | 4 | 150 | 0 | 0.035 | 0.055 | - | 0.010 | 0.1 | 050075 |
| | | | | | | 1 | 0.039 | 0.012 | - | 0.013 | 0.064 | |
| | | | | | | 3 | 0.018 | ND | - | < 0.01 | 0.028 | |
| | | | | | | 7 | 0.019 | ND | - | < 0.01 | 0.029 | |
| | | | | | | 14 | < 0.01 | ND | - | 0.004 | 0.014 | |
| Earnscleugh,Central Otago 2006 (Sundrop) 050075-07 | SC | 3.7 | 53–59 | 4 | 221 | 0 | 0.126 | 0.039 | - | 0.024 | 0.189 | 050075 |
| | | | | | | 1 | 0.124 | 0.037 | - | 0.036 | 0.197 | |
| | | | | | | 3 | 0.063 | 0.018 | - | 0.028 | 0.109 | |
| | | | | | | 7 | 0.041 | 0.011 | - | 0.023 | 0.075 | |
| | | | | | | 14 | 0.022 | < 0.01 | - | 0.016 | 0.038 | |
| Earnscleugh,Central Otago 2006 (Sundrop) 050075-07 | SC | 5 | 72–79 | 4 | 298 | 0 | 0.101 | 0.031 | - | 0.025 | 0.157 | 050075 |
| | | | | | | 1 | 0.080 | 0.023 | - | 0.019 | 0.122 | |
| | | | | | | 3 | 0.081 | 0.025 | - | 0.031 | 0.137 | |
| | | | | | | 7 | 0.064 | 0.017 | - | 0.034 | 0.115 | |
| | | | | | | 14 | 0.024 | ND | - | 0.021 | 0.045 | |

| Location, year (Variety) | Form | Application | | | Total/ season, g ai/ha | PHI, days | Residue, mg/kg | | | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | ND-J | NF-J | Total [a] | |
| Earnscleugh,Central Otago 2006 (Sundrop) 050075-07 | SC | 7.6 | 107–119 | 4 | 447 | 0 | 0.222 | 0.067 | - | 0.038 | 0.327 | 050075 |
| | | | | | | 1 | 0.213 | 0.062 | - | 0.046 | 0.321 | |
| | | | | | | 3 | 0.139 | 0.038 | - | 0.038 | 0.215 | |
| | | | | | | 7 | 0.084 | 0.022 | - | 0.036 | 0.142 | |
| | | | | | | 14 | 0.034 | < 0.01 | - | 0.017 | 0.051 | |

[a] Total residue = XDE-175-J + XDE-175-L + ND-J + NF-J.

*Fruiting Vegetables other than Cucurbits*

*Supervised trials on tomato in the USA*

Six supervised trials on tomatoes were conducted in major tomato growing areas in the USA in 2004 and 2007 (Dolder and Wendelburg, 2005; Report 040063; McKellar, 2008; Report ARAP 07D-001).

Trials were conducted in six sites, each site consisting of a control plot and a treated plot. Each treated plot received five applications of a suspension concentrate (SC) formulation containing either 100 g/L or 120 g/L of spinetoram. The initial application was made at a nominal rate of 20 g ai/ha, followed by four additional applications at the higher rate of 70 g ai/ha for a seasonal maximum of approximately 300 g ai/ha. The applications were made as low volume spray applications at 4-day intervals. Mature tomatoes were collected at the PHI of 1 day. In one trial, samples were also taken on days 0, 1, 3, 7, and 14 days after the last treatment.

Approximately 2 kg of mature whole tomato fruits were collected by hand. At each sampling, a single composite sample was taken from the control plot while duplicate, composite samples were taken independently from each treated plot. Samples were frozen within 4 hours of collection, placed in plastic bags, labelled, shipped frozen, and kept in temperature-monitored freezers at -20 °C until analysis.

Spinetoram and its metabolites were analysed using the method GRM 05.04 with an LOQ and LOD of 0.01 mg/kg and 0.003 mg/kg, respectively. Samples from the 2007 trials were analysed using method GRM 05.03 with the same LOQ and LOD as GRM 05.04.

Table 30-1 Residues of spinetoram from supervised trials on tomato in the USA (for estimation of maximum residue level)

| Location, year (Variety) | Form | Application | | | Total/ season, g ai/ha | PHI, days | Residue, mg/kg | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | Total [a] | |
| GAP, USA Fruiting vegetables | SC | | 44-88 | 6 | 298 | 1 | | | | |
| North Rose, NY, 2004 (Floradade) | SC | 5, initial, + 21, 4 appl. | 21, initial, + 70-71, 4 appl. | 5 | 304 | 1 | 0.010 | ND | 0.010 | 040063 |
| | | | | | | | < 0.01 | ND | < 0.01 | |
| Lake Park, GA, 2004 (Sonoma Roam) | SC | 5, initial, + 17-19, 4 appl. | 20, initial, + 71-72, 4 appl. | 5 | 306 | 1 | ND | ND | < 0.01 | 040063 |
| | | | | | | | ND | ND | < 0.01 | |

| Location, year (Variety) | Form | Application | | | Total/ season, g ai/ha | PHI, days | Residue, mg/kg | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | Total [a] | |
| Jennings, FL, 2004 (Florida) | SC | 5, initial, + 17-19, 4 appl. | 20, initial, + 70-76, 4 appl. | 5 | 308 | 1 | < 0.01 | ND | < 0.01 | 040063 |
| | | | | | | | ND | ND | < 0.01 | |
| Paynesville, MN, 2004 (Olinda) | SC | 7, initial, +24, 4 appl. | 20, initial, + 70-71, 4 appl. | 5 | 301 | 1 | 0.025 | < 0.01 | 0.025 | 040063 |
| | | | | | | | 0.022 | < 0.01 | 0.022 | |
| Porterville, CA, 2004 (Early Girl) | SC | 22-25 | 70-71 | 4 | 281 | 0 | 0.043 | 0.015 | 0.058 | 040063 |
| | | | | | | | 0.020 | < 0.01 | 0.02 | |
| | | | | | | 1 | 0.015 | < 0.01 | 0.015 | |
| | | | | | | | 0.012 | < 0.01 | 0.012 | |
| | | | | | | 3 | < 0.01 | ND | < 0.01 | |
| | | | | | | | < 0.01 | ND | < 0.01 | |
| | | | | | | 7 | ND | ND | < 0.01 | |
| | | | | | | | < 0.01 | ND | < 0.01 | |
| | | | | | | 14 | ND | ND | < 0.01 | |
| | | | | | | | ND | ND | < 0.01 | |
| GA, 2007 (Not specified) | SC | 5, initial, + 14-16, 4 appl. | 21, initial, + 68-70, 4 appl. | 5 | 295 | 1 | 0.022 | < 0.01 | 0.022 | 040063 |
| | | | | | | | 0.024 | < 0.01 | 0.024 | |

[a] Total residue = XDE-175-J + XDE-175-L.

Table 30-2 Residues of spinetoram and metabolites from supervised trials on tomato in the USA (for estimation of STMR)

| Location, year (Variety) | Form | Application | | | Total/ season, g ai/ha | PHI, days | Residue, mg/kg | | | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | ND-J | NF-J | Total [a] | |
| GAP, USA Fruiting vegetables | SC | | 44-88 | 6 | 298 | 1 | | | | | | |
| North Rose, NY, 2004 (Floradade) | SC | 5, initial, + 21, 4 appl. | 21, initial, + 70-71, 4 appl. | 5 | 304 | 1 | 0.010 | ND | < 0.01 | 0.010 | 0.020 | 040063 |
| | | | | | | | < 0.01 | ND | < 0.01 | < 0.01 | < 0.02 | |
| Lake Park, GA, 2004 (Sonoma Roam) | SC | 5, initial, + 17-19, 4 appl. | 20, initial, + 71-72, 4 appl. | 5 | 306 | 1 | ND | ND | ND | < 0.01 | < 0.02 | 040063 |
| | | | | | | | ND | ND | ND | < 0.01 | < 0.02 | |
| Jennings, FL, 2004 (Florida) | SC | 5, initial, + 17-19, 4 appl. | 20, initial, + 70-76, 4 appl. | 5 | 308 | 1 | < 0.01 | ND | ND | < 0.01 | < 0.02 | 040063 |
| | | | | | | | ND | ND | ND | < 0.01 | < 0.02 | |

| Location, year (Variety) | Form | Application | | | Total/ season, g ai/ha | PHI, days | Residue, mg/kg | | | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | ND-J | NF-J | Total [a] | |
| Paynesville, MN, 2004 (Olinda) | SC | 7, initial, +24, 4 appl. | 20, initial, + 70-71, 4 appl. | 5 | 301 | 1 | 0.025 | < 0.01 | < 0.01 | < 0.01 | 0.035 | 040063 |
| | | | | | | | 0.022 | < 0.01 | < 0.01 | < 0.01 | 0.032 | |
| Porterville, CA, 2004 (Early Girl) | SC | 22-25 | 70-71 | 4 | 281 | 0 | 0.043 | 0.015 | < 0.01 | 0.011 | 0.069 | 040063 |
| | | | | | | | 0.020 | < 0.01 | < 0.01 | < 0.01 | 0.030 | |
| | | | | | | 1 | 0.015 | < 0.01 | < 0.01 | < 0.01 | 0.025 | |
| | | | | | | | 0.012 | < 0.01 | < 0.01 | < 0.01 | 0.022 | |
| | | | | | | 3 | < 0.01 | ND | ND | < 0.01 | < 0.02 | |
| | | | | | | | < 0.01 | ND | < 0.01 | < 0.01 | < 0.02 | |
| | | | | | | 7 | ND | ND | ND | < 0.01 | < 0.02 | |
| | | | | | | | < 0.01 | ND | < 0.01 | < 0.01 | < 0.02 | |
| | | | | | | 14 | ND | ND | ND | ND | < 0.02 | |
| | | | | | | | ND | ND | ND | < 0.01 | < 0.02 | |
| GA, 2007 (Not specified) | SC | 5, initial, + 14-16, 4 appl. | 21, initial, + 68-70, 4 appl. | 5 | 295 | 1 | 0.022 | < 0.01 | ND | < 0.01 | 0.032 | 040063 |
| | | | | | | | 0.024 | < 0.01 | ND | < 0.01 | 0.034 | |

[a] Total residue = XDE-175-J + XDE-175-L + ND-J + NF-J.

*Leafy Vegetables*

*Supervised trials on lettuce in the US*

Six supervised trials on leaf lettuce were conducted in major lettuce growing areas in the US in 2004 and 2007 (Dolder and Wendelburg, 2005; Report 040063; McKellar, 2008; Report ARAP 07D-001).

Trials were conducted in six sites, each site consisting of a control plot and a treated plot. Each treated plot received five applications of a suspension concentrate (SC) formulation containing either 100 g/L or 120 g/L of spinetoram. The initial application was made at a nominal rate of 20 g ai/ha, followed by four additional applications at the higher rate of 70 g ai/ha for a seasonal total of approximately 300 g ai/ha. The applications were made as low volume spray applications at 4-day intervals. Mature lettuce samples were collected at the PHI of 1 day. In one trial, samples were also taken on days 0, 1, 3, 7, and 14 after the last application.

Approximately 1 kg of mature lettuce leaves was collected by hand. At each sampling, a single composite sample was taken from the control plot while duplicate, composite samples were taken independently from each treated plot. Samples were frozen within 4 h of collection, placed in plastic bags, labelled, shipped frozen, and kept in temperature-monitored freezers at –20 °C until analysis.

Spinetoram and its metabolites were analysed using the method GRM 05.04 with an LOQ and limit of detection LOD of 0.01 mg/kg and 0.003 mg/kg, respectively. Samples from the 2007 trials were analysed using method GRM 05.03 with the same LOQ and LOD values as GRM 05.04.

Table 31-1 Residues of spinetoram from supervised trials on leaf lettuce in the USA (for estimation of maximum residue level)

| Location, year (Variety) | Form | Application | | | Total/ season, g ai/ha | PHI, days | Residue, mg/kg | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | Total [a] | |
| GAP, USA Leafy vegetables | WG or SC | | 44–88 | 6 | 298 | 1 | | | | |
| North Rose, NY, 2004 (Black Seeded Simpson) | WG | 6 initial and 22, 4 appl. | 21 initial and 71-73, 4 appl. | 5 | 309 | 1 | 0.226 | 0.057 | 0.283 | 040063 |
| | | | | | | | 0.247 | 0.067 | 0.314 | |
| Chula, GA, 2004 (Tehama) | WG | 7 initial and 23, 4 appl. | 21 initial and 70-71, 4 appl. | 5 | 305 | 1 | 0.120 | 0.010 | 0.130 | 040063 |
| | | | | | | | 0.142 | 0.012 | 0.154 | |
| Jennings, FL, 2004 (Tehama) | WG | 6 initial and 21-22, 4 appl. | 20 initial and 70-74, 4 appl. | 5 | 305 | 1 | 0.222 | 0.060 | 0.282 | 040063 |
| | | | | | | | 0.265 | 0.076 | 0.341 | |
| King City, CA, 2004 (Sunbelt) | WG | 7 initial and 22-24, 4 appl. | 21 initial and 67-71, 4 appl. | 5 | 299 | 1 | 0.153 | 0.043 | 0.196 | 040063 |
| | | | | | | | 0.245 | 0.075 | 0.320 | |
| Porterville, CA, 2004 (Shining Star) | WG | 6 initial and 19-20, 4 appl. | 20 initial and 70, 4 appl. | 5 | 300 | 0 | 0.97 | 0.31 | 1.28 | 040063 |
| | | | | | | | 1.42 | 0.44 | 1.86 | |
| | | | | | | 1 | 0.42 | 0.12 | 0.54 | 040063 |
| | | | | | | | 0.43 | 0.12 | 0.55 | |
| | | | | | | 3 | 0.11 | 0.03 | 0.14 | 040063 |
| | | | | | | | 0.12 | 0.03 | 0.15 | |
| | | | | | | 7 | 0.05 | 0.01 | 0.06 | 040063 |
| | | | | | | | 0.07 | 0.02 | 0.09 | |
| | | | | | | 14 | 0.02 | < 0.01 | 0.02 | 040063 |
| | | | | | | | 0.02 | < 0.01 | 0.02 | |
| GA, 2007 (Not specified) | WG | 4 initial and 15, 4 appl. | 21 initial and 70, 4 appl. | 5 | 305 | 1 | 5.62 | 1.40 | 7.02 | 040063 |
| | | | | | | | 6.26 | 1.54 | 7.80 | |

[a] Total residue = XDE-175-J + XDE-175-L.

Table 31-2 Residues of spinetoram and metabolites from supervised trials on leaf lettuce in the USA (for estimation of STMR)

| Location, year (Variety) | Form | Application | | | Total/ season, g ai/ha | PHI, days | Residue, mg/kg | | | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | ND-J | NF-J | Total [a] | |
| GAP, USA Leafy vegetables | WG or SC | | 44–88 | 6 | 298 | 1 | | | | | | |
| North Rose, NY, 2004 (Black Seeded Simpson) | WG | 6 initial and 22, 4 appl. | 21 initial and 71-73, 4 appl. | 5 | 309 | 1 | 0.226 | 0.057 | 0.183 | 0.094 | 0.560 | 040063 |
| | | | | | | | 0.247 | 0.067 | 0.208 | 0.121 | 0.643 | |
| Chula, GA, 2004 (Tehama) | WG | 7 initial and 23, 4 appl. | 21 initial and 70-71, 4 appl. | 5 | 305 | 1 | 0.120 | 0.010 | 0.056 | 0.077 | 0.263 | 040063 |
| | | | | | | | 0.142 | 0.012 | 0.055 | 0.070 | 0.279 | |
| Jennings, FL, 2004 (Tehama) | WG | 6 initial and 21-22, 4 appl. | 20 initial and 70-74, 4 appl. | 5 | 305 | 1 | 0.222 | 0.060 | 0.141 | 0.700 | 1.123 | 040063 |
| | | | | | | | 0.265 | 0.076 | 0.151 | 0.858 | 1.350 | |
| King City, CA, 2004 (Sunbelt) | WG | 7 initial and 22-24, 4 appl. | 21 initial and 67-71, 4 appl. | 5 | 299 | 1 | 0.153 | 0.043 | 0.076 | 0.056 | 0.328 | 040063 |
| | | | | | | | 0.245 | 0.075 | 0.146 | 0.092 | 0.558 | |
| Porterville, CA, 2004 (Shining Star) | WG | 6 initial and 19-20, 4 appl. | 20 initial and 70, 4 appl. | 5 | 300 | 0 | 0.97 | 0.31 | 0.31 | 0.19 | 1.78 | 040063 |
| | | | | | | | 1.42 | 0.44 | 0.42 | 0.22 | 2.50 | |
| | | | | | | 1 | 0.42 | 0.12 | 0.32 | 0.25 | 1.11 | 040063 |
| | | | | | | | 0.43 | 0.12 | 0.35 | 0.26 | 1.16 | |
| | | | | | | 3 | 0.11 | 0.03 | 0.13 | 0.10 | 0.37 | 040063 |
| | | | | | | | 0.12 | 0.03 | 0.16 | 0.12 | 0.43 | |
| | | | | | | 7 | 0.05 | 0.01 | 0.08 | 0.08 | 0.22 | 040063 |
| | | | | | | | 0.07 | 0.02 | 0.09 | 0.09 | 0.27 | |
| | | | | | | 14 | 0.02 | < 0.01 | 0.02 | 0.02 | 0.06 | 040063 |
| | | | | | | | 0.02 | < 0.01 | 0.02 | 0.03 | 0.07 | |
| GA, 2007 (Not specified) | WG | 4 initial and 15, 4 appl. | 21 initial and 70, 4 appl. | 5 | 305 | 1 | 5.62 | 1.40 | 0.66 | 0.94 | 8.62 | 040063 |
| | | | | | | | 6.26 | 1.54 | 0.75 | 1.00 | 9.55 | |

[a] Total residue = XDE-175-J + XDE-175-L + ND-J + NF-J.

*Root and Tuber Vegetables*

*Supervised trials sugar beet in the USA*

Six supervised trials on sugar beet were conducted in major sugar beet growing areas in the US in 2004 and 2007 (Dolder and Wendelburg, 2005; Report 040063; McKellar, 2008; Report ARAP 07D-001).

Trials were conducted in six sites, each site consisting of a control plot and a treated plot. Each treated plot received four applications of a suspension concentrate (SC) formulation containing either 100 g/L or 120 g/L of spinetoram at a nominal rate of 70 g ai/ha for a seasonal maximum of 280 g ai/ha. The applications were made as low volume spray applications at 4-day intervals. Mature roots and tops were collected 3 days after the last application. In one trial, samples were also taken on days 0, 1, 3, 7, and 14 days after the last treatment.

Approximately 2.3 kg of mature roots and 2.8 kg of tops were collected by hand. At each sampling, a single composite sample was taken from the control plot while duplicate, composite samples were taken independently from each treated plot. Due to the large size of the sugar beet, the root portion was quartered longitudinally and only one quarter from each of 12 beets were retained as a sample. Samples were frozen within 4 h of collection, placed in plastic bags, labelled, shipped frozen, and kept in temperature-monitored freezers at –20 °C until analysis.

Spinetoram and its metabolites were analysed using the method GRM 05.04 with an LOQ and LOD of 0.01 mg/kg and 0.003 mg/kg, respectively. Samples from the 2007 trials were analysed using method GRM 05.03 with the same LOQ and LOD values as GRM 05.04.

Table 32-1 Residues of spinetoram from supervised trials on sugar beet in the USA (for estimation of maximum residue level)

| Location, year (Variety) | Form | Application | | | Total/ season, g ai/ha | PHI, days | Residue, mg/kg | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | Total [a] | |
| GAP, US, Tuberous vegetables | SC | | 53-70 | 4 | 281 | 7 | | | | |
| SUGAR BEET ROOTS | | | | | | | | | | |
| Olivia, MN, 2004 (Beta) | SC | 24–25 | 70–71 | 4 | 282 | 3 | ND | ND | < 0.01 | 040063 |
| | | | | | | | ND | ND | < 0.01 | |
| Edgeley, ND, 2004 (Beta) | SC | 24–25 | 70–71 | 4 | 281 | 3 | ND | ND | < 0.01 | 040063 |
| | | | | | | | ND | ND | < 0.01 | |
| Levelland, TX, 2004 (Wrangler) | SC | 19 | 70–72 | 4 | 284 | 3 | ND | ND | < 0.01 | 040063 |
| | | | | | | | ND | ND | < 0.01 | |
| Porterville, CA, 2004 (Beta 4030R) | SC | 19 | 70–71 | 4 | 283 | 3 | ND | ND | < 0.01 | 040063 |
| | | | | | | | ND | ND | < 0.01 | |
| Ephrata, WA, 2004 (Blazer) | SC | 21 | 70–71 | 4 | 283 | 0 | < 0.01 | ND | < 0.01 | 040063 |
| | | | | | | | < 0.01 | ND | < 0.01 | |
| | | | | | | 1 | ND | ND | < 0.01 | |
| | | | | | | | ND | ND | < 0.01 | |
| | | | | | | 3 | < 0.01 | ND | < 0.01 | |
| | | | | | | | ND | ND | < 0.01 | |
| | | | | | | 7 | ND | ND | < 0.01 | |
| | | | | | | | ND | ND | < 0.01 | |

| Location, year (Variety) | Form | Application | | | Total/ season, g ai/ha | PHI, days | Residue, mg/kg | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | Total [a] | |
| | | | | | | 14 | ND | ND | < 0.01 | |
| | | | | | | | ND | ND | < 0.01 | |
| GA, 2007 (Not specified) | SC | 21-22 | 69-72 | 4 | 283 | 3 | < 0.01 | ND | < 0.01 | ARAP 07D-001 |
| | | | | | | | < 0.01 | ND | < 0.01 | |

[a] Total residue = XDE-175-J + XDE-175-L.

Table 32-2 Residues of spinetoram and metabolites from supervised trials on sugar beet in the USA (for estimation of STMR)

| Location, year (Variety) | Form | Application | | | Total/ season, g ai/ha | PHI, days | Residue, mg/kg | | | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | ND-J | NF-J | Total [a] | |
| GAP, US, Root vegetables | SC | | 53-70 | 4 | 281 | 7 | | | | | | |
| SUGAR BEET ROOTS | | | | | | | | | | | | |
| Olivia, MN, 2004 (Beta) | SC | 24–25 | 70–71 | 4 | 282 | 3 | ND | ND | ND | ND | < 0.02 | 040063 |
| | | | | | | | ND | ND | ND | ND | < 0.02 | |
| Edgeley, ND, 2004 (Beta) | SC | 24–25 | 70–71 | 4 | 281 | 3 | ND | ND | ND | ND | < 0.02 | 040063 |
| | | | | | | | ND | ND | ND | ND | < 0.02 | |
| Levelland, TX, 2004 (Wrangler) | SC | 19 | 70–72 | 4 | 284 | 3 | ND | ND | ND | < 0.01 | < 0.02 | 040063 |
| | | | | | | | ND | ND | ND | ND | < 0.02 | |
| Porterville, CA, 2004 (Beta 4030R) | SC | 19 | 70–71 | 4 | 283 | 3 | ND | ND | < 0.01 | < 0.01 | < 0.02 | 040063 |
| | | | | | | | ND | ND | ND | ND | < 0.02 | |
| Ephrata, WA, 2004 (Blazer) | SC | 21 | 70–71 | 4 | 283 | 0 | < 0.01 | ND | < 0.01 | < 0.01 | < 0.02 | 040063 |
| | | | | | | | < 0.01 | ND | < 0.01 | < 0.01 | < 0.02 | |
| | | | | | | 1 | ND | ND | ND | ND | < 0.02 | |
| | | | | | | | ND | ND | ND | < 0.01 | < 0.02 | |
| | | | | | | 3 | < 0.01 | ND | < 0.01 | < 0.01 | < 0.02 | |
| | | | | | | | ND | ND | ND | ND | < 0.02 | |
| | | | | | | 7 | ND | ND | ND | < 0.01 | < 0.02 | |
| | | | | | | | ND | ND | ND | ND | < 0.02 | |
| | | | | | | 14 | ND | ND | < 0.01 | < 0.01 | < 0.02 | |
| | | | | | | | ND | ND | < 0.01 | ND | < 0.02 | |
| GA, 2007 (Not specified) | SC | 21-22 | 69-72 | 4 | 283 | 3 | < 0.01 | ND | < 0.01 | ND | < 0.02 | ARAP 07D-001 |
| | | | | | | | < 0.01 | ND | < 0.01 | ND | < 0.02 | |

[a] Total residue = XDE-175-J + XDE-175-L + ND-J + NF-J.

*Tree Nuts*

*Supervised trials on almond and pecan in the USA*

Six supervised trials on almond and six on pecan were conducted in the USA in 2007 (Dolder and Schelle, 2008; Report 070069).

Trials on almond were conducted in six sites in California, while trials on pecan were conducted in six different States in the USA. Each site consisted of a control plot and a treated plot, with at least six trees in each plot. Each treated plot received four applications of WG formulation containing either 250 g/kg of spinetoram at a nominal rate of 123 g ai/ha for a seasonal maximum of 500g ai/ha. The applications were made at intervals of 7 days, using airblast sprayers with four to seven nozzles. Spray volumes ranged from 889 to 1074 L/ha. Whole nuts were collected 7 days after the last application. In decline trials (one each for almond and pecan), samples were taken on days 0, 1, 3, 7 and 14 days after the last treatment.

For each commodity at each site, a single, composite sample was taken from the control plot while duplicate, composite samples were taken independently from the treated plot. At each sampling the nuts were knocked or shaken from the trees. Trees at the ends of plots were not sampled. The whole nuts were collected and, within approximately 24 hours, were cracked using a nut cracker and the nutmeat (and hulls in the case of almonds) was collected by hand or with a sheller. A minimum of 1 kg was collected for each sample. Samples were placed in sample bags, labelled and frozen within approximately 24 h of sampling and remained frozen up through shipment overnight to the analytical laboratory. All samples were received frozen and were stored in temperature-monitored freezers at approximately –20 °C.

Spinetoram and its metabolites were analysed using the method GRM 05.04 with an LOQ and LOD of 0.01 mg/kg and 0.003 mg/kg, respectively.

Table 33-1 Residues of spinetoram from supervised trials on almonds in the USA (for estimation of maximum residue level)

| Location, year (Variety) | Form | Application | | | Total/ year g ai/ha | PHI, days | Residue, mg/kg | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | Total [a] | |
| GAP, USA, Tree Nuts | WG | | 26–123 | 4 | 490 | 14 | | | | |
| Terra Bella, CA, 2007 070069CA1 (Carmel) | WG | 13 | 117–144 | 4 | 532 | 7 | < 0.01 | ND | < 0.01 | 070069 |
| | | | | | | | < 0.01 | ND | < 0.01 | |
| Wasco, CA, 2007 070069CA2 (Price) | WG | 13 | 117–130 | 4 | 501 | 7 | < 0.01 | ND | < 0.01 | 070069 |
| | | | | | | | < 0.01 | ND | < 0.01 | |
| Earlimart, CA, 2007 070069CA3 (Fritz) | WG | 13 | 117–133 | 4 | 491 | 7 | ND | ND | < 0.01 | 070069 |
| | | | | | | | ND | ND | < 0.01 | |
| Sultana, CA, 2007 070069CA4 (Carmel) | WG | 13 | 123–124 | 4 | 494 | 7 | < 0.01 | ND | < 0.01 | 070069 |
| | | | | | | | < 0.01 | ND | < 0.01 | |
| Sanger, CA, 2007 070069CA5 (Neplus) | WG | 13 | 110–124 | 4 | 480 | 7 | ND | ND | < 0.01 | 070069 |
| | | | | | | | ND | ND | < 0.01 | |

| Location, year (Variety) | Form | Application | | | Total/ year g ai/ha | PHI, days | Residue, mg/kg | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | Total [a] | |
| Sanger, CA, 2007 070069CA6 (Nonpareil) | WG | 13 | 123–123 | 4 | 495 | 0 | 0.011 | ND | 0.011 | 070069 |
| | | | | | | | 0.011 | < 0.01 | 0.011 | |
| | | | | | | 1 | < 0.01 | ND | < 0.01 | |
| | | | | | | | 0.011 | ND | 0.011 | |
| | | | | | | 3 | < 0.01 | ND | < 0.01 | |
| | | | | | | | 0.011 | ND | 0.011 | |
| | | | | | | 7 | < 0.01 | ND | < 0.01 | 070069 |
| | | | | | | | < 0.01 | ND | < 0.01 | |
| | | | | | | 14 | < 0.01 | ND | < 0.01 | 070069 |
| | | | | | | | < 0.01 | ND | < 0.01 | |

[a] Total residue = XDE-175-J + XDE-175-L.

Table 33-2 Residues of spinetoram and metabolites from supervised trials on almonds in the USA (for estimation of STMR)

| Location, year (Variety) | Form | Application | | | Total/ year g ai/ha | PHI, days | Residue, mg/kg | | | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | ND-J | NF-J | Total [a] | |
| GAP, USA, Tree Nuts | WG | | 26–123 | 4 | 490 | 14 | | | | | | |
| Terra Bella, CA, 2007 070069CA1 (Carmel) | WG | 13 | 117–144 | 4 | 532 | 7 | < 0.01 | ND | ND | ND | < 0.02 | 070069 |
| | | | | | | | < 0.01 | ND | ND | ND | < 0.02 | |
| Wasco, CA, 2007 070069CA2 (Price) | WG | 13 | 117–130 | 4 | 501 | 7 | < 0.01 | ND | ND | ND | < 0.02 | 070069 |
| | | | | | | | < 0.01 | ND | ND | ND | < 0.02 | |
| Earlimart, CA, 2007 070069CA3 (Fritz) | WG | 13 | 117–133 | 4 | 491 | 7 | ND | ND | ND | ND | < 0.02 | 070069 |
| | | | | | | | ND | ND | ND | ND | < 0.02 | |
| Sultana, CA, 2007 070069CA4 (Carmel) | WG | 13 | 123–124 | 4 | 494 | 7 | < 0.01 | ND | ND | ND | < 0.02 | 070069 |
| | | | | | | | < 0.01 | ND | ND | ND | < 0.02 | |
| Sanger, CA, 2007 070069CA5 (Neplus) | WG | 13 | 110–124 | 4 | 480 | 7 | ND | ND | ND | ND | < 0.02 | 070069 |
| | | | | | | | ND | ND | ND | ND | < 0.02 | |
| Sanger, CA, 2007 070069CA6 (Nonpareil) | WG | 13 | 123–123 | 4 | 495 | 0 | 0.011 | ND | ND | ND | 0.021 | 070069 |
| | | | | | | | 0.011 | < 0.01 | ND | ND | 0.021 | |
| | | | | | | 1 | < 0.01 | ND | ND | ND | < 0.02 | |
| | | | | | | | 0.011 | ND | ND | ND | 0.021 | |
| | | | | | | 3 | < 0.01 | ND | ND | ND | < 0.02 | |
| | | | | | | | 0.011 | ND | ND | ND | 0.021 | |

| Location, year (Variety) | Form | Application | | | Total/ year g ai/ha | PHI, days | Residue, mg/kg | | | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | ND-J | NF-J | Total [a] | |
| | | | | | | 7 | < 0.01 | ND | ND | ND | < 0.02 | 070069 |
| | | | | | | | < 0.01 | ND | ND | ND | < 0.02 | |
| | | | | | | 14 | < 0.01 | ND | ND | ND | < 0.02 | 070069 |
| | | | | | | | < 0.01 | ND | ND | ND | < 0.02 | |

[a] Total residue = XDE-175-J + XDE-175-L + ND-J + NF-J.

Table 34-1 Residues of spinetoram from supervised trials on pecan in the USA (for estimation of maximum residue level)

| Location, year (Variety) | Form | Application | | | Total/ season, g ai/ha | PHI, days | Residue, mg/kg | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | Total [a] | |
| GAP, USA, Tree Nuts | WG | | 26–123 | 4 | 490 | 14 | | | | |
| Scott, AR, 2007 070069AR (Stuart) | WG | 13 | 123–126 | 4 | 495 | 7 | ND | ND | < 0.01 | 070069 |
| | | | | | | | ND | ND | < 0.01 | |
| Albany, GA, 2007 070069GA2 (Sumner) | WG | 13 | 122–126 | 4 | 495 | 7 | ND | ND | < 0.01 | 070069 |
| | | | | | | | ND | ND | < 0.01 | |
| Madill, OK, 2007 070069OK1 (Graking) | WG | 13 | 123 | 4 | 492 | 7 | 0.011 | ND | 0.011 | 070069 |
| | | | | | | | 0.010 | ND | 0.010 | |
| Mannsville, OK, 2007 070069OK2 (Beckett) | WG | 13 | 122–123 | 4 | 491 | 7 | 0.012 | ND | 0.012 | 070069 |
| | | | | | | | 0.010 | ND | 0.010 | |
| Claytonville, TX, 2007 070069TX (Beckett) | WG | 13 | 123–124 | 4 | 493 | 7 | 0.011 | ND | 0.011 | 070069 |
| | | | | | | | 0.011 | ND | 0.011 | |
| Chula, GA, 2007 070069GA1 (Sumner) | WG | 13 | 123–125 | 4 | 496 | 0 | < 0.01 | ND | < 0.01 | 070069 |
| | | | | | | | 0.011 | ND | 0.011 | |
| | | | | | | 1 | 0.010 | ND | 0.010 | |
| | | | | | | | 0.011 | ND | 0.011 | |
| | | | | | | 3 | < 0.01 | ND | < 0.01 | |
| | | | | | | | < 0.01 | ND | < 0.01 | |
| | | | | | | 7 | ND | ND | < 0.01 | |
| | | | | | | | ND | ND | < 0.01 | |
| | | | | | | 14 | ND | ND | < 0.01 | |
| | | | | | | | ND | ND | < 0.01 | |

[a] Total residue = XDE-175-J + XDE-175-L.

Table 34-2 Residues of spinetoram and metabolites from supervised trials on pecan in the USA (for estimation of STMR)

| Location, year (Variety) | Form | Application | | | Total/ season, g ai/ha | PHI, days | Residue, mg/kg | | | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | ND-J | NF-J | Total [a] | |
| GAP, USA, Tree Nuts | WG | | 26–123 | 4 | 490 | 14 | | | | | | |
| Scott, AR, 2007 070069AR (Stuart) | WG | 13 | 123–126 | 4 | 495 | 7 | ND | ND | ND | ND | < 0.02 | 070069 |
| | | | | | | | ND | ND | ND | ND | < 0.02 | |
| Albany, GA, 2007 070069GA2 (Sumner) | WG | 13 | 122–126 | 4 | 495 | 7 | ND | ND | ND | ND | < 0.02 | 070069 |
| | | | | | | | ND | ND | ND | ND | < 0.02 | |
| Madill, OK, 2007 070069OK1 (Graking) | WG | 13 | 123 | 4 | 492 | 7 | 0.011 | ND | ND | ND | 0.021 | 070069 |
| | | | | | | | 0.010 | ND | ND | ND | 0.020 | |
| Mannsville, OK, 2007 070069OK2 (Beckett) | WG | 13 | 122-123 | 4 | 491 | 7 | 0.012 | ND | ND | ND | 0.022 | 070069 |
| | | | | | | | 0.010 | ND | ND | ND | 0.020 | |
| Claytonville, TX, 2007 070069TX (Beckett) | WG | 13 | 123–124 | 4 | 493 | 7 | 0.011 | ND | ND | ND | 0.021 | 070069 |
| | | | | | | | 0.011 | ND | ND | ND | 0.021 | |
| Chula, GA, 2007 070069GA1 (Sumner) | WG | 13 | 123–125 | 4 | 496 | 0 | < 0.01 | ND | ND | ND | < 0.02 | 070069 |
| | | | | | | | 0.011 | ND | ND | ND | 0.021 | |
| | | | | | | 1 | 0.010 | ND | ND | ND | 0.02 | |
| | | | | | | | 0.011 | ND | ND | ND | 0.021 | |
| | | | | | | 3 | < 0.01 | ND | ND | ND | < 0.02 | |
| | | | | | | | < 0.01 | ND | ND | ND | < 0.02 | |
| | | | | | | 7 | ND | ND | ND | ND | < 0.02 | |
| | | | | | | | ND | ND | ND | ND | < 0.02 | |
| | | | | | | 14 | ND | ND | ND | ND | < 0.02 | |
| | | | | | | | ND | ND | ND | ND | < 0.02 | |

[a] Total residue = XDE-175-J + XDE-175-L + ND-J + NF-J.

*Animal Feedstuffs*

Table 35-1 Residues of spinetoram in sugar beet leaves or tops from supervised trials on sugar beet in the USA (for estimation of maximum residue level)

| Location, year (Variety) | Form | Application | | | Total/ season, g ai/ha | PHI, days | Residue, mg/kg | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | Total [a] | |
| GAP, US, Root vegetables | SC | | 53-70 | 4 | 281 | 7 | | | | |
| SUGAR BEET TOPS/LEAVES | | | | | | | | | | |
| Olivia, MN, 2004 (Beta) | SC | 24–25 | 70–71 | 4 | 282 | 3 | 0.089 | 0.025 | 0.114 | 040063 |
| | | | | | | | 0.055 | 0.016 | 0.071 | |
| Edgeley, ND, 2004 (Beta) | SC | 24–25 | 70–71 | 4 | 281 | 3 | 0.153 | 0.047 | 0.200 | 040063 |
| | | | | | | | 0.097 | 0.029 | 0.126 | |
| Levelland, TX, 2004 (Wrangler) | SC | 19 | 70–72 | 4 | 284 | 3 | 0.073 | 0.013 | 0.086 | 040063 |
| | | | | | | | 0.062 | 0.013 | 0.075 | |
| Porterville, CA, 2004 (Beta 4030R) | SC | 19 | 70–71 | 4 | 283 | 3 | 0.063 | 0.013 | 0.076 | 040063 |
| | | | | | | | 0.081 | 0.018 | 0.099 | |
| Ephrata, WA, 2004 (Blazer) | SC | 21 | 70–71 | 4 | 283 | 0 | 0.589 | 0.190 | 0.779 | 040063 |
| | | | | | | | 0.542 | 0.172 | 0.714 | |
| | | | | | | 1 | 0.129 | 0.038 | 0.167 | |
| | | | | | | | 0.175 | 0.054 | 0.229 | |
| | | | | | | 3 | 0.111 | 0.033 | 0.144 | |
| | | | | | | | 0.122 | 0.035 | 0.157 | |
| | | | | | | 7 | 0.024 | < 0.01 | 0.024 | |
| | | | | | | | 0.019 | < 0.01 | 0.019 | |
| | | | | | | 14 | < 0.01 | ND | < 0.01 | |
| | | | | | | | 0.011 | ND | 0.011 | |
| Michigan, 2007 (Not specified) | SC | 21–22 | 69–72 | 4 | 283 | 3 | 0.085 | 0.016 | 0.101 | ARAP 07D-001 |
| | | | | | | | 0.168 | 0.031 | 0.199 | |

[a] Total residue = XDE-175-J + XDE-175-L.

Table 35-2 Residues of spinetoram and metabolites in sugar beet leaves or tops from supervised trials on sugar beet in the USA (for estimation of STMR)

| Location, year (Variety) | Form | Application | | | Total/ season, g ai/ha | PHI, days | Residue, mg/kg | | | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | ND-J | NF-J | Total[a] | |
| GAP, US, Root vegetables | SC | | 53-70 | 4 | 281 | 7 | | | | | | |
| SUGAR BEET TOPS/LEAVES | | | | | | | | | | | | |
| Olivia, MN, | SC | 24– | 70–71 | 4 | 282 | 3 | 0.089 | 0.025 | 0.052 | 0.069 | 0.235 | 040063 |

| Location, year (Variety) | Form | Application | | | Total/ season, g ai/ha | PHI, days | Residue, mg/kg | | | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | ND-J | NF-J | Total[a] | |
| 2004 (Beta) | | 25 | | | | | 0.055 | 0.016 | 0.046 | 0.046 | 0.163 | |
| Edgeley, ND, 2004 (Beta) | SC | 24–25 | 70–71 | 4 | 281 | 3 | 0.153 | 0.047 | 0.095 | 0.101 | 0.396 | 040063 |
| | | | | | | | 0.097 | 0.029 | 0.072 | 0.067 | 0.265 | |
| Levelland, TX, 2004 (Wrangler) | SC | 19 | 70–72 | 4 | 284 | 3 | 0.073 | 0.013 | 0.101 | 0.167 | 0.354 | 040063 |
| | | | | | | | 0.062 | 0.013 | 0.090 | 0.141 | 0.306 | |
| Porterville, CA, 2004 (Beta 4030R) | SC | 19 | 70–71 | 4 | 283 | 3 | 0.063 | 0.013 | 0.175 | 0.231 | 0.482 | 040063 |
| | | | | | | | 0.081 | 0.018 | 0.165 | 0.204 | 0.468 | |
| Ephrata, WA, 2004 (Blazer) | SC | 21 | 70–71 | 4 | 283 | 0 | 0.589 | 0.190 | 0.249 | 0.144 | 1.172 | 040063 |
| | | | | | | | 0.542 | 0.172 | 0.214 | 0.111 | 1.039 | |
| | | | | | | 1 | 0.129 | 0.038 | 0.161 | 0.271 | 0.599 | |
| | | | | | | | 0.175 | 0.054 | 0.165 | 0.316 | 0.71 | |
| | | | | | | 3 | 0.111 | 0.033 | 0.125 | 0.343 | 0.612 | |
| | | | | | | | 0.122 | 0.035 | 0.125 | 0.347 | 0.629 | |
| | | | | | | 7 | 0.024 | < 0.01 | 0.038 | 0.171 | 0.233 | |
| | | | | | | | 0.019 | < 0.01 | 0.032 | 0.133 | 0.184 | |
| | | | | | | 14 | < 0.01 | ND | 0.016 | 0.048 | 0.074 | |
| | | | | | | | 0.011 | ND | 0.019 | 0.049 | 0.079 | |
| Michigan, 2007 (Not specified) | SC | 21–22 | 69–72 | 4 | 283 | 3 | 0.085 | 0.016 | 0.174 | 0.121 | 0.396 | ARAP 07D-001 |
| | | | | | | | 0.168 | 0.031 | 0.261 | 0.214 | 0.674 | |

[a] Total residue = XDE-175-J + XDE-175-L + ND-J + NF-J.

Table 36-1 Residues of spinetoram and metabolites in almond hulls from supervised trials on almond in the USA

| Location, year (Variety) | Form | Application | | | Total/ year g ai/ha | PHI, days | Residue, mg/kg | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | Total [a] | |
| GAP, USA, Tree Nuts | WG | | 26–123 | 4 | 490 | 14 | | | | |
| Terra Bella, CA, 2007 070069CA1 (Carmel) | WG | 13 | 117–144 | 4 | 532 | 7 | 0.695 | 0.135 | 0.830 | 070069 |
| | | | | | | | 0.500 | 0.101 | 0.601 | |
| Wasco, CA, 2007 070069CA2 (Price) | WG | 13 | 117–130 | 4 | 501 | 7 | 0.378 | 0.083 | 0.461 | 070069 |
| | | | | | | | 0.457 | 0.109 | 0.566 | |
| Earlimart, CA, 2007 070069CA3 (Fritz) | WG | 13 | 117–133 | 4 | 491 | 7 | 0.705 | 0.146 | 0.851 | 070069 |
| | | | | | | | 0.358 | 0.076 | 0.434 | |

| Location, year (Variety) | Form | Application | | | Total/ year g ai/ha | PHI, days | Residue, mg/kg | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | Total [a] | |
| Sultana, CA, 2007 070069CA4 (Carmel) | WG | 13 | 123–124 | 4 | 494 | 7 | 1.901 | 0.419 | 2.320 | 070069 |
| | | | | | | | 1.394 | 0.306 | 1.700 | |
| Sanger, CA, 2007 070069CA5 (Neplus) | WG | 13 | 110–124 | 4 | 480 | 7 | 0.737 | 0.137 | 0.874 | 070069 |
| | | | | | | | 0.702 | 0.129 | 0.831 | |
| Sanger, CA, 2007 070069CA6 (Nonpareil) | WG | 13 | 123–123 | 4 | 495 | 0 | 1.326 | 0.246 | 1.572 | 070069 |
| | | | | | | | 1.366 | 0.281 | 1.647 | |
| | | | | | | 1 | 1.182 | 0.216 | 1.398 | 070069 |
| | | | | | | | 1.270 | 0.234 | 1.504 | |
| | | | | | | 3 | 0.875 | 0.173 | 1.048 | 070069 |
| | | | | | | | 0.883 | 0.171 | 1.054 | |
| | | | | | | 7 | 0.858 | 0.190 | 1.048 | 070069 |
| | | | | | | | 0.883 | 0.202 | 1.085 | |
| | | | | | | 14 | 0.525 | 0.107 | 0.632 | 070069 |
| | | | | | | | 0.616 | 0.134 | 0.750 | |

[a] Total residue = XDE-175-J + XDE-175-L.

Table 36-2 Residues of spinetoram and metabolites in almond hulls from supervised trials on almond in the USA (for estimation of STMR)

| Location, year (Variety) | Form | Application | | | Total/ year g ai/ha | PHI, days | Residue, mg/kg | | | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | ND-J | NF-J | Total [a] | |
| GAP, USA, Tree Nuts | WG | | 26–123 | 4 | 490 | 14 | | | | | | |
| Terra Bella, CA, 2007 070069CA1 (Carmel) | WG | 13 | 117–144 | 4 | 532 | 7 | 0.695 | 0.135 | 0.128 | 0.308 | 1.27 | 070069 |
| | | | | | | | 0.500 | 0.101 | 0.086 | 0.191 | 0.88 | |
| Wasco, CA, 2007 070069CA2 (Price) | WG | 13 | 117–130 | 4 | 501 | 7 | 0.378 | 0.083 | 0.040 | 0.051 | 0.55 | 070069 |
| | | | | | | | 0.457 | 0.109 | 0.046 | 0.050 | 0.66 | |
| Earlimart, CA, 2007 070069CA3 (Fritz) | WG | 13 | 117–133 | 4 | 491 | 7 | 0.705 | 0.146 | 0.086 | 0.102 | 1.04 | 070069 |
| | | | | | | | 0.358 | 0.076 | 0.039 | 0.045 | 0.52 | |
| Sultana, CA, 2007 070069CA4 (Carmel) | WG | 13 | 123–124 | 4 | 494 | 7 | 1.901 | 0.419 | 0.152 | 0.177 | 2.65 | 070069 |
| | | | | | | | 1.394 | 0.306 | 0.113 | 0.124 | 1.94 | |
| Sanger, CA, 2007 070069CA5 (Neplus) | WG | 13 | 110–124 | 4 | 480 | 7 | 0.737 | 0.137 | 0.094 | 0.105 | 1.07 | 070069 |
| | | | | | | | 0.702 | 0.129 | 0.084 | 0.108 | 1.02 | |

| Location, year (Variety) | Form | Application | | | Total/ year g ai/ha | PHI, days | Residue, mg/kg | | | | | Report No. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | g ai/hL | g ai/ha | No | | | XDE-175-J | XDE-175-L | ND-J | NF-J | Total [a] | |
| Sanger, CA, 2007 070069CA6 (Nonpareil) | WG | 13 | 123–123 | 4 | 495 | 0 | 1.326 | 0.246 | 0.098 | 0.095 | 1.77 | 070069 |
| | | | | | | | 1.366 | 0.281 | 0.112 | 0.111 | 1.87 | |
| | | | | | | 1 | 1.182 | 0.216 | 0.098 | 0.097 | 1.59 | 070069 |
| | | | | | | | 1.270 | 0.234 | 0.107 | 0.101 | 1.71 | |
| | | | | | | 3 | 0.875 | 0.173 | 0.092 | 0.135 | 1.28 | 070069 |
| | | | | | | | 0.883 | 0.171 | 0.093 | 0.125 | 1.27 | |
| | | | | | | 7 | 0.858 | 0.190 | 0.084 | 0.091 | 1.22 | 070069 |
| | | | | | | | 0.883 | 0.202 | 0.080 | 0.079 | 1.25 | |
| | | | | | | 14 | 0.525 | 0.107 | 0.055 | 0.088 | 0.78 | 070069 |
| | | | | | | | 0.616 | 0.134 | 0.068 | 0.097 | 0.91 | |

[a] Total residue = XDE-175-J + XDE-175-L + ND-J + NF-J.

## FATE OF RESIDUES IN STORAGE AND PROCESSING

### *In storage*

No information was provided to the Meeting as spinetoram is not intended for use in stored products.

### *In processing*

The Meeting received information on processing of oranges to juice and oil, and apples to juice and puree (sauce).

*Orange*

An orange processing study was conducted in the USA in 2007 where orange samples were processed to dried pulp, juice, and oil with corresponding processing factors calculated (McKellar, 2008; Report ARAP 07D-001).

Orange trees were treated three times with a SC formulation containing 120 g/L spinetoram, at the exaggerated rate of 350 g ai/ha, equivalent to 5× the GAP rate. Applications were made at intervals of 4 days, using low spray volume application. Samples (approximately 200 kg) of mature orange fruits from the control and treated plots were harvested at the PHI of 1 day and shipped at ambient temperature, to the processing facility. The following processed commodities were prepared, following procedures that simulated commercial practices: orange peel, pulp, dried pulp, juice, and oil.

Peel: After inspection of the samples, an aliquot of the oranges were removed and peeled by hand to generate orange peel, from which a representative sample fraction was removed, packaged, labelled and placed in frozen storage.

Oil: The remaining oranges were washed for 5 minutes. Approximately 3 kg of oranges per batch were abraded for 1 minute. The scarified fruit was weighed and retained for juice processing. The collected oil-water emulsion was screened using a ca. 180 µm screen to separate any fragments from the oil-water emulsion. The separated fragments were set aside for later addition to the shredded peel. The first run oil-water emulsion was processed through the cream separator and IEC centrifuge to separate the oil. The free oil was removed and measured with a 50 mL volumetric pipette and frozen. The residual emulsion was then frozen overnight, thawed, centrifuged and removed with a 50 mL volumetric pipette and added to the oil collected the previous day. The entire collected combined sample of the oil recovered from both processing days was weighed and then packaged, labelled and placed in frozen storage.

Juice and peel/pulp: An aliquot of the scarified oranges were weighed and transferred to the juice extractor to recover the juice from the peel. The juice and peel/pulp recovered were weighed and a representative orange pulp sample fraction was removed, packaged, labelled and placed in frozen storage.

The collected juice was transferred to the pulper finisher and screened using ca. 1.19 mm screen to remove vesicular membranes, seeds, segment membranes and peel fragments from the juice. The collected rag and seeds were set aside for later addition to the shredded peel. A representative sample of the fresh filtered juice was removed, packaged, labelled and placed in frozen storage for the required sample fraction. The remaining juice was discarded.

Dried pulp: The peel from the juice extractor was shredded and the shredded peel was combined with the scarified flavedo from the scarification process and the rag and seeds from the juice finisher extraction process to generate wet peel. Lime was added to the wet peel and mixed for 17 minutes. The limed peel was pressed in a fruit press and the expressed liquid weighed, checked for pH and Brix and discarded. The wet peel/pulp was placed on the air drier and dried to below 10% moisture. The dried pulp was milled using a hammermill. A representative sample of the dried pomace was removed, packaged, labelled and placed in frozen storage. The remaining dried pomace was discarded.

All samples were stored up to 87 days before analysis, which is within the storage time of the stability study conducted separately.

Residues of spinetoram and its metabolites were determined in whole orange samples collected from plots treated at 5× the GAP, and each processed fraction prepared, using the method GRM 05.04, with an LOQ and LOD of 0.01 mg/kg and 0.003 mg/kg, respectively.

Residues in orange and processed fractions are summarized in Table 37. Residues are reported as the sum of residues of XDE-175-J, XDE-175-L, N-demethyl-175-J and N-formyl-175-J.

Table 37 Residues of spinetoram and its metabolites in orange and orange processed products

| Processed Product | Spinetoram residue | | Spinetoram + two metabolites | | |
|---|---|---|---|---|---|
| | Residues mg/kg | Processing factor | Residues mg/kg | Processing factor | Reference |
| Orange (RAC) | 0.201 | | 0.280 | | McKellar, 2008 Report ARAP 07D-001 |
| Peel | 0.279 | 1.4 | 0.384 | 1.4 | |
| Pulp after juicing | 0.061 | 0.30 | 0.091 | 0.33 | |
| Dried pulp | 0.478 | 2.4 | 0.478 | 2.3 | |
| Juice | < 0.01 | < 0.05 | < 0.02 | < 0.07 | |
| Oil | 28.8 | 143 | 32.23 | 115 | |

*Apple*

Two processing trials on apples were carried out in France in 2005 (Dolder and Schelle, 2006; Report No. 050028). Apple trees were treated with four applications of the WG formulation containing 250 g/kg spinetoram at the rate of 100 g ai/ha. Applications were made at approximately 7-day intervals with a calibrated sprayer simulating a commercial application. Water volumes used were approximately 800–1200 L/ha for apples. Samples were collected 7 days after the last application.

About 60 kg of mature apples were sent to the processing facility for processing into dry pomace, juice, sauce and canned apples. Processing procedures simulated commercial practices. The fruits were washed manually with tap water. The following processed samples were prepared:

Apple juice: The washed apples were crushed in an apple mill and thereafter pressed in a fruit pack press to get wet pomace and raw juice. A part of the wet pomace was dried overnight in an oven and the sample of dry pomace was weighed to determine the dry weight.

The raw juice was filtered and enzymes were added to the filtered juice. The time and temperature during enzyme treatment was recorded. The cleared juice was filtered once or twice and, after filtration, the clear juice was filled in glass bottles which were closed airtight and pasteurized at 81–89 °C and cooled down. The cooled clear juice samples were stored deep frozen until analysis.

Apple puree: Washed fruits were cut in small pieces before putting them into a stainless steel pot. After adding water to the apples (about 150–200 mL per kg of apples), the mixture was heated until the fruits were soft. The fruits were then passed through a food mill-sieve combination in order to remove the peel and cores. A part of the raw stewed sauce was filled in preserving glass bottles. The glass bottles were closed airtight and pasteurized at 80–89 °C for 30 minutes. After cooling down, the apple puree samples were stored deep frozen until analysis.

Canned apples: Washed fruits were peeled manually with a knife and the peel and cores weighed and removed. The peeled fruits were cut into halves or quarters and filled in suitable glass containers. A heated sugar-water solution was put into the container filled with fruits. The glass containers were then closed airtight and pasteurized for 30–35 minutes at 80–85 °C. After heating, the glass containers were opened and sample of canned fruits taken. After cooling down, the samples were stored deep frozen until analysis.

Residues of spinetoram and metabolites were determined using method GRM 05.04 with an LOQ of 0.01 mg/kg and an LOD of 0.003 mg/kg. The results are summarized in Table 37. Residues reported are the sum of residues of XDE-175-J, XDE-175-L, *N*-demethyl-175-J, and *N*-formyl-175-J.

Table 38 Residues of spinetoram and metabolites in apples and processed fractions

| Processed product | Spinetoram residue | | Spinetoram + two metabolites | | |
|---|---|---|---|---|---|
| | Residues mg/kg | Processing factor | Residues mg/kg | Processing factor | Reference |
| Apple (RAC) | 0.020 | | 0.041 | | Dolder and Schelle, 2008 Report 050028 |
| Juice | < 0.01 | < 0.50 | < 0.02 | < 0.48 | |
| Dry pomace | 0.212 | 10.6 | 0.331 | 7.9 | |
| Puree (sauce) | 0.012 | 0.60 | 0.022 | 0.53 | |
| Canned apples | < 0.01 | < 0.50 | < 0.02 | < 0.48 | |
| Apple (RAC) | 0.041 | | 0.051 | | Dolder and Schelle, 2008 Report 050028 |
| Juice | < 0.01 | < 0.24 | < 0.02 | < 0.39 | |
| Dry pomace | 0.173 | 5.6 | 0.210 | 4.1 | |
| Puree (sauce) | 0.015 | 0.29 | 0.022 | 0.42 | |
| Canned apples | < 0.01 | < 0.24 | < 0.02 | < 0.39 | |
| | | Mean | | Mean | |
| Juice | | < 0.37 | | < 0.44 | |
| Dry pomace | | 8.1 | | 6.0 | |
| Puree (sauce) | | 0.45 | | 0.47 | |
| Canned apples | | < 0.50 | | < 0.44 | |

## RESIDUES I ANIMAL COMMODITIES

### *Farm animal feeding studies*

*Lactating cows*

Six groups of lactating Holstein dairy cows (3–5 years old; body weight 474-656 kg at receipt) were dosed daily for 29 consecutive days via gelatine capsules containing spinetoram (Dolder and Schelle, 2007; Report 060052) (Table 39). Each group consisted of at least 3 cows. Four of the groups were dosed with test substance A, containing 26% XDE-175-J, 6% XDE-175-L, 30% *N*-formyl-175-J and

28% *N*-demethyl-175-J. One group was given test substance B containing only XDE-175-J and XDE-175-L (86% total). The remaining group was the control group which was dosed with empty gelatine capsules.

Table 39 Animal Dosing Groups

| Dose Level | Number of Cows | Dose, ppm in diet | | |
|---|---|---|---|---|
| | | Test Substance A | | Test Substance B |
| | | Total [a] | Spinetoram | Spinetoram |
| Control | 4 | - | - | - |
| 0.3× | 3 | 1.2 | 0.4 | - |
| 1× | 12 | 3.7 | 1.3 | - |
| 3× | 3 | 11.5 | 3.8 | - |
| 10× | 3 | 38.6 | 12.9 | - |
| 10× | 3 | - | | 37.6 |

[a] Total dose= sum of XDE-175-J, XDE-175-L, N-demethyl-175-J, and N-formyl-175-J.

Milk was collected twice daily (AM and PM) via surge milking machines and placed into labelled collection containers. The milk from AM and PM milking of each treated animal was pooled in proportion to the amount of milk collected at each of the two daily milkings. Milk samples from the control animals were also pooled in this manner. Only milk from the same cow for a given study day's AM and PM milking was combined to form a pooled sample. The calculated amounts of PM and AM milk were combined to form the pooled 250 g sample. The average daily milk production in dose groups ranged between 26 and 29 kg during the dosing period.

Milk samples from Days 14 and 28 were separated into cream and skim milk from the three cows each in the control and 1× dose groups that were not designated for use in the depletion phase and the three cows in each of the 10×-A and 10×-B dose groups.

Three cows from each group were sacrificed at the end of a 29-day dosing period in order to determine residue levels in tissues (muscle, fat, liver and kidney). One extra cow in the control group along with nine extra cows in the 1X dose group were used to evaluate residue depletion in milk and tissues during a withdrawal period following the completion of the dosing period. Of the nine cows in the 1× dose group, three cows were assigned to each of three sacrifice dates following completion of the dosing period. The three sacrifice dates in the depletion phase were 14, 28 and 56 days after completion of dosing. The one extra cow in the control group used in the withdrawal phase of the study was sacrificed at 56 days after completion of the dosing period. Samples of liver, kidney, muscle and fat (approximately 1 kg each, if available, of mesenterial, perirenal and subcutaneous) were collected from all animals, cubed, and frozen until analysis. Samples were analysed within 20 days of collection; therefore, no storage stability studies were conducted.

Milk and tissue samples were analysed for residues of XDE-175-J, XDE-175-L, *N*-formyl-175-J and *N*-demethyl-175-J by method GRM 6.08 with LOQ of 0.01 mg/kg for each of the analytes.

Residues found in milk samples collected during the dosing phase of the study are summarized in Table 40**.** No residue was detected in any of the control samples throughout the dosing phase. Residues in the 0.3× dose group were generally between the LOD (0.003 mg/kg) and LOQ (0.01 mg/kg) throughout the dosing period. The average spinetoram residues in milk for study days 10–28 for the 0.3×, 1×, 3×, 10×-A and 10×-B dose groups were < 0.01, 0.018, 0.072, 0.253, and 0.711 mg/kg, respectively. Residues found in milk from cows given the parent compounds only were about three times higher than those in milk from cows given a mixture of the parent, *N*-demethyl-175-J and *N*-formyl-175-J, reflecting the ratio of the parent in the test substances.

Table 40 Mean spinetoram residues in milk during dosing phase

| Dose Group | Residues of XDE-175-J and XDE-175-L in milk on specific dosing day (mg/kg) | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | -2 | 3 | 7 | 10 | 14 | 16 | 18 | 20 | 22 | 24 | 26 | 28 |
| Control [a] | < 0.01 | < 0.01 | < 0.01 | < 0.01 | < 0.01 | < 0.01 | < 0.01 | < 0.01 | < 0.01 | < 0.01 | < 0.01 | < 0.01 |
| 0.3× | < 0.01 | < 0.01 | < 0.01 | < 0.01 | < 0.01 | < 0.01 | < 0.01 | < 0.01 | < 0.01 | < 0.01 | < 0.01 | < 0.01 |
| 1× | < 0.01 | 0.013 | 0.022 | 0.021 | 0.019 | 0.017 | 0.019 | 0.017 | 0.018 | 0.018 | 0.019 | 0.017 |
| 3× | < 0.01 | 0.049 | 0.065 | 0.076 | 0.077 | 0.071 | 0.070 | 0.067 | 0.076 | 0.087 | 0.076 | 0.080 |
| 10×-A | < 0.01 | 0.21 | 0.20 | 0.23 | 0.23 | 0.17 | 0.25 | 0.25 | 0.37 | 0.28 | 0.30 | 0.29 |
| 10×-B | < 0.01 | 0.059 | 0.69 | 0.70 | 0.70 | 0.69 | 0.72 | 0.71 | 0.73 | 1.01 | 0.94 | 0.87 |

| Dose Group | Residues of XDE-J, XDE-L, *N*-demethyl-175-J and *N*-formy-175-J in milk on specific dosing day (mg/kg) | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | -2 | 3 | 7 | 10 | 14 | 16 | 18 | 20 | 22 | 24 | 26 | 28 |
| Control [a] | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 |
| 0.3× | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 |
| 1× | < 0.02 | 0.023 | 0.032 | 0.031 | 0.027 | 0.027 | 0.029 | 0.027 | 0.028 | 0.028 | 0.029 | 0.027 |
| 3× | < 0.02 | 0.059 | 0.075 | 0.086 | 0.087 | 0.081 | 0.080 | 0.077 | 0.086 | 0.097 | 0.086 | 0.090 |
| 10×-A | < 0.02 | 0.22 | 0.22 | 0.26 | 0.26 | 0.19 | 0.28 | 0.26 | 0.40 | 0.30 | 0.32 | 0.32 |
| 10×-B | < 0.02 | 0.60 | 0.70 | 0.71 | 0.71 | 0.70 | 0.73 | 0.72 | 0.74 | 1.02 | 0.95 | 0.88 |

[a] Includes cows designated for the depletion phase.

Samples from milk collected on days 14 and 28 were separated into skim milk and cream. Table 41 shows a summary of the residues. No residues were detected in skim milk from the 1× dose group. In the 10×-A and 10×-B dose groups average total residues in skim milk ranged from just below the LOQ to 0.075mg/kg. In all of the dose groups on both study days residues in cream were much higher than the residues in skim milk. Mean total residues from the 1× dose group in cream were 0.187 and 0.231 mg/kg. Mean total residues in the 10×-A and 10×-B groups ranged 0.64–1.95 and 3.11–5.84 mg/kg. These results indicate that residues of spinetoram in whole milk preferentially partition into cream. The median ratio of spinetoram residues in cream to those in whole milk is 6.6.

Table 41 Mean spinetoram residues in skim milk and cream

| Dose Group | Residues of XDE-175-J and XDE-175-L on specific dosing day (mg/kg) | | | | | |
|---|---|---|---|---|---|---|
| | Whole Milk | | Cream | | Skim Milk | |
| | Day 14 | Day 28 | Day 14 | Day 28 | Day 14 | Day 28 |
| 1X | 0.027 | 0.023 | 0.177 | 0.221 | < 0.01 | < 0.01 |
| 10X-A | 0.12 | 0.29 | 0.616 | 1.84 | < 0.01 | 0.028 |
| 10X-B | 0.70 | 0.87 | 3.09 | 5.81 | 0.060 | 0.065 |

| Dose Group | Residues of XDE-J, XDE-L, *N*-demethyl-175-J and *N*-formy-175-J on specific dosing day (mg/kg) | | | | | |
|---|---|---|---|---|---|---|
| | Whole Milk | | Cream | | Skim Milk | |
| | Day 14 | Day 28 | Day 14 | Day 28 | Day 14 | Day 28 |
| 1X | 0.037 | 0.033 | 0.187 | 0.231 | < 0.02 | < 0.02 |
| 10X-A | 0.13 | 0.32 | 0.642 | 1.95 | < 0.02 | 0.038 |
| 10X-B | 0.71 | 0.88 | 3.11 | 5.84 | 0.070 | 0.075 |

Residues found in kidney, liver, muscle, and fat samples collected from animals sacrificed upon completion of the dosing phase are presented in Table 42. With one exception, no residues were detected in the control samples. One kidney control sample contained residues below the LOQ possibly due to contamination. All tissues from treated cows contained residues and they increased from the lowest to highest dose groups. Residue concentrations were lowest in the muscle followed by kidney, liver, and fat. Residues in fat were significantly higher than residues in the other tissues. Across all the dose levels the mesenterial and perirenal fat samples had similar residue concentrations which were roughly twice the concentrations found in the subcutaneous fat. As expected, the composite fat samples, which consisted of equal parts of the three fats collected, yielded residues very close to the average residue concentration found in the three fats. These results indicate that residues of spinetoram tend to accumulate in fatty tissue.

Table 42 Spinetoram residues in kidney, liver, muscle and fat following a dosing period of 29 days

| Dose Group | Residues of XDE-J and XDE-L on specific dosing day (mg/kg) | | | | | | |
|---|---|---|---|---|---|---|---|
| | Kidney | Liver | Muscle | Subcutaneous Fat | Mesenterial Fat | Perirenal Fat | Composite Fat[1] |
| Control | < 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 |
| 0.3× | < 0.01 | < 0.01 | < 0.01 | 0.077 | 0.093 | 0.10 | 0.082 |
| | < 0.01 | < 0.01 | < 0.01 | 0.063 | 0.10 | 0.11 | 0.11 |
| | < 0.01 | < 0.01 | < 0.01 | 0.021 | 0.10 | 0.091 | 0.075 |
| Maximum | < 0.01 | < 0.01 | < 0.01 | 0.077 | 0.10 | 0.11 | 0.11 |
| Mean | < 0.01 | < 0.01 | < 0.01 | 0.054 | 0.10 | 0.10 | 0.090 |
| 1× | 0.016 | 0.057 | < 0.01 | 0.087 | 0.44 | 0.50 | 0.38 |
| | 0.040 | 0.052 | 0.043 | 0.52 | 0.59 | 0.69 | 0.55 |
| | 0.011 | 0.040 | < 0.01 | 0.14 | 0.29 | 0.34 | 0.26 |
| Maximum | 0.040 | 0.057 | 0.043 | 0.52 | 0.59 | 0.69 | 0.55 |
| Mean | 0.022 | 0.050 | 0.021 | 0.25 | 0.44 | 0.51 | 0.39 |
| 3× | 0.074 | 0.11 | 0.086 | 0.74 | 1.22 | 1.41 | 1.25 |
| | 0.068 | 0.11 | 0.019 | 0.57 | 0.83 | 0.64 | 0.39 |
| | 0.048 | 0.11 | 0.010 | 0.035 | 0.25 | 0.20 | 0.13 |
| Maximum | 0.074 | 0.11 | 0.086 | 0.74 | 1.22 | 1.41 | 1.25 |
| Mean | 0.063 | 0.11 | 0.038 | 0.45 | 0.76 | 0.75 | 0.59 |
| 10×-A | 0.29 | 0.47 | 0.082 | 0.15 | 0.29 | 0.84 | 0.35 |
| | 0.25 | 0.41 | 0.13 | 1.14 | 3.54 | 3.07 | 2.68 |
| | 0.30 | 0.21 | 0.24 | 2.96 | 3.69 | 3.64 | 3.26 |
| Maximum | 0.30 | 0.47 | 0.24 | 2.96 | 3.69 | 3.64 | 3.26 |
| Mean | 0.28 | 0.36 | 0.15 | 1.42 | 2.51 | 2.52 | 2.10 |
| 10×B | 1.74 | 1.29 | 0.53 | 8.69 | 11.23 | 16.52 | 15.29 |
| | 0.81 | 0.62 | 0.49 | 4.69 | 8.37 | 11.04 | 9.72 |
| | 1.01 | 2.39 | 0.41 | 2.32 | 9.22 | 14.77 | 8.85 |
| Maximum | 1.74 | 2.39 | 0.53 | 8.69 | 11.23 | 16.52 | 15.29 |
| Mean | 1.19 | 1.43 | 0.48 | 5.24 | 9.61 | 14.14 | 11.28 |

| Dose Group | Residues of XDE-J, XDE-L, *N*-demethyl-175-J and *N*-formy-175-J on specific dosing day (mg/kg) | | | | | | |
|---|---|---|---|---|---|---|---|
| | Kidney | Liver | Muscle | Subcutaneous Fat | Mesenterial Fat | Perirenal Fat | Composite Fat [a] |
| Control | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 |
| | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 |
| | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 |
| 0.3× | < 0.02 | < 0.02 | < 0.02 | 0.087 | 0.10 | 0.11 | 0.092 |
| | < 0.02 | 0.010 | < 0.02 | 0.073 | 0.11 | 0.12 | 0.12 |
| | < 0.02 | < 0.02 | < 0.02 | 0.031 | 0.11 | 0.10 | 0.085 |
| Maximum | < 0.02 | < 0.02 | < 0.02 | 0.087 | 0.11 | 0.12 | 0.12 |
| Mean | < 0.02 | < 0.02 | < 0.02 | 0.064 | 0.10 | 0.11 | 0.10 |
| 1× | 0.043 | 0.10 | < 0.02 | 0.097 | 0.46 | 0.53 | 0.40 |
| | 0.075 | 0.095 | 0.053 | 0.54 | 0.61 | 0.72 | 0.57 |
| | 0.036 | 0.092 | < 0.02 | 0.15 | 0.30 | 0.36 | 0.27 |
| Maximum | 0.075 | 0.10 | 0.053 | 0.54 | 0.61 | 0.72 | 0.57 |
| Mean | 0.052 | 0.097 | 0.031 | 0.26 | 0.46 | 0.54 | 0.41 |
| 3× | 0.13 | 0.22 | 0.11 | 0.81 | 1.29 | 1.49 | 1.32 |
| | 0.12 | 0.19 | 0.029 | 0.60 | 0.86 | 0.66 | 0.40 |
| | 0.091 | 0.21 | 0.033 | 0.045 | 0.27 | 0.23 | 0.15 |
| Maximum | 0.13 | 0.22 | 0.11 | 0.81 | 1.29 | 1.49 | 1.33 |
| Mean | 0.12 | 0.20 | 0.057 | 0.48 | 0.81 | 0.79 | 0.63 |
| 10×-A | 0.57 | 0.93 | 0.17 | 0.18 | 0.34 | 0.95 | 0.42 |
| | 0.44 | 0.73 | 0.18 | 1.22 | 3.72 | 3.23 | 2.80 |
| | 0.40 | 0.36 | 0.29 | 3.12 | 3.89 | 3.84 | 3.44 |
| Maximum | 0.57 | 0.93 | 0.29 | 3.12 | 3.89 | 3.84 | 3.44 |
| Mean | 0.47 | 0.68 | 0.21 | 1.51 | 2.65 | 2.67 | 2.22 |
| 10×-B | 1.80 | 1.37 | 0.54 | 8.75 | 11.29 | 16.61 | 15.36 |
| | 0.84 | 0.66 | 0.50 | 4.71 | 8.41 | 11.08 | 9.76 |
| | 1.08 | 2.57 | 0.44 | 2.36 | 9.30 | 14.84 | 8.92 |
| Maximum | 1.80 | 2.57 | 0.54 | 8.75 | 11.29 | 16.61 | 15.36 |
| Mean | 1.24 | 1.54 | 0.50 | 5.28 | 9.67 | 14.18 | 11.34 |

[a] Contained equal parts of subcutaneous, mesenterial, and perirenal fats.

Residues found in milk samples collected from cows during the depletion phase are shown in Table 43. With one exception, residues were not detectable in milk by the fourth day after the last dose was administered. Concentrations just above the LOD were detected in one cow through the ninth day after the final dose. No further residue was detected beyond that point. No residue was ever detected in milk from the cow in the control group.

Table 43 Residues of spinetoram in milk during the depletion phase

| Dose Group | Residues of XDE-J, XDE-L, *N*-demethyl-175-J and *N*-formy-175-J on days after final dose (mg/kg) | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 0 | 2 | 4 | 6 | 9 | 13 | 20 | 27 | 34 | 41 | 48 | 55 |
| | (29) | (31) | (33) | (35) | (38) | (42) | (49) | (56) | (63) | (70) | (77) | (84) |
| Control | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 |
| 1X | 0.036 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | - | - | - | - | - | - |
| | 0.029 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | - | - | - | - | - | - |
| | 0.020 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | - | - | - | - | - | - |

| Dose Group | Residues of XDE-J, XDE-L, *N*-demethyl-175-J and *N*-formy-175-J on days after final dose (mg/kg) | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 0 | 2 | 4 | 6 | 9 | 13 | 20 | 27 | 34 | 41 | 48 | 55 |
| | (29) | (31) | (33) | (35) | (38) | (42) | (49) | (56) | (63) | (70) | (77) | (84) |
| | 0.025 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | - | - | - | - |
| | 0.024 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | - | - | - | - |
| | 0.027 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | - | - | - | - |
| | 0.024 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 |
| | 0.029 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 |
| | 0.026 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 |
| Mean | 0.027 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 |

Days in parentheses are days after the beginning of dosing period.

Residues found in kidney, liver, muscle, and fat samples collected during the depletion phase are presented in Table 44. Residue concentrations were lowest in the muscle followed by kidney, liver, and fat. On the day the last dose was given, the mesenterial and perirenal fat samples had similar residue concentrations which were roughly twice the concentrations found in the subcutaneous fat. However, in the fat samples taken 14 days after the last dose was given, the residue in the subcutaneous fat was higher than in the other two fat samples. As expected, the composite fat samples, which consisted of equal parts of the three fats collected, yielded residues very close to the average residue concentration found in the three fats. Residue decline continued through 28-days after the last dose. No residue was detected in kidney, liver or muscle from any cow by 28 days after the final dose or in fat 56 days following the final dose.

Table 44 Residues of spinetoram in tissues during the depletion phase

| Days After Final Dose | Study Day | Residues of XDE-J, XDE-L, *N*-demethyl-175-J and *N*-formy-175-J on days after final dose (mg/kg) | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | Kidney | Liver | Muscle | Sub. Fat | Mes. Fat | Per. Fat | Comp. Fat |
| 0 | 29 | 0.043 | 0.103 | 0.020 | 0.097 | 0.46 | 0.53 | 0.40 |
| 0 | 29 | 0.075 | 0.095 | 0.053 | 0.55 | 0.61 | 0.72 | 0.57 |
| 0 | 29 | 0.036 | 0.092 | 0.023 | 0.15 | 0.30 | 0.36 | 0.27 |
| | Mean | 0.052 | 0.097 | 0.032 | 0.26 | 0.46 | 0.54 | 0.41 |
| 14 | 43 | < 0.02 | < 0.02 | < 0.02 | 0.26 | 0.23 | 0.14 | 0.22 |
| 14 | 43 | < 0.02 | < 0.02 | < 0.02 | 0.051 | < 0.02 | 0.021 | < 0.02 |
| 14 | 43 | < 0.02 | < 0.02 | < 0.02 | 0.10 | 0.055 | 0.026 | 0.063 |
| | Mean | < 0.02 | < 0.02 | < 0.02 | 0.14 | 0.10 | 0.062 | 0.10 |
| 28 | 57 | < 0.02 | < 0.02 | < 0.02 | 0.022 | 0.022 | < 0.02 | < 0.02 |
| 28 | 57 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 |
| 28 | 57 | < 0.02 | < 0.02 | < 0.02 | 0.044 | 0.024 | 0.023 | 0.037 |
| | Mean | < 0.02 | < 0.02 | < 0.02 | 0.029 | 0.022 | < 0.02 | 0.025S |
| 56 | 85 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 |
| 56 | 85 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 |
| 56 | 85 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 |
| | Mean | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 |
| Control | | | | | | | | |
| 56 | 85 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 |

Cattle, except those in the control group, were dosed with a total dose of 3.7 ppm in the diet.

Note: Residues are reported as sum of XDE-175-J, XDE-175-L, *N*-demethyl-175-J and *N*-formyl-175-J concentrations. All concentrations are reported in mg/kg.

*Laying hens*

The Meeting received no information on poultry feeding studies.

## ADDITIONAL INFORMATION

The currently existing national residue definitions for plant and animal commodities are:

Australia: XDE-175-J and XDE-175-L;

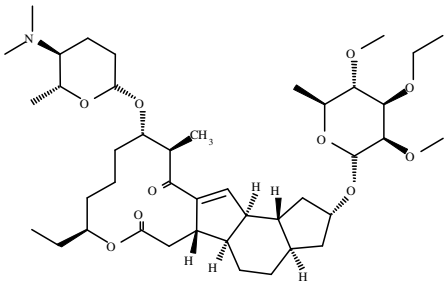
Canada: XDE-175-J, XDE-175-L, N-demethyl-175-J and N-formyl-175-J (for risk assessment in poultry commodities, 3'-O-deethyl-175-J and L, and O-demethyl-175-L are added) ;

New Zealand: XDE-175-J and XDE-175-L; and

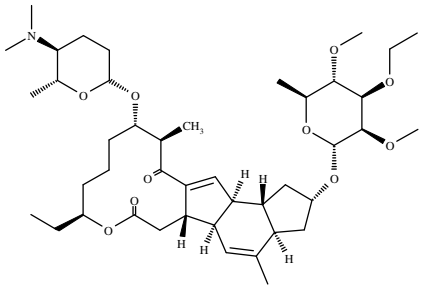
USA: XDE-175-J, XDE-175-L, N-demethyl-175-J and N-formyl-175-J ;

## APPRAISAL

Spinetoram, a multi-component tetracyclic macrolide in the class of spinosyn insecticides, consists of two components shown below, present approximately in a three to one ratio. It was identified as a priority new compound at the 39th Session of the CCPR in 2007 (ALINORM 07/30/24—Rev.1) for evaluation by the 2008 JMPR. The Meeting received information on physical and chemical properties, animal and plant metabolism, environmental fate, analytical methods, storage stability, use patterns, supervised trials, processing and farm animal feeding.

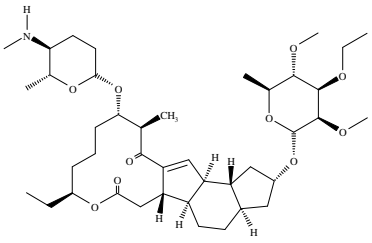


XDE-175-J (Major component)

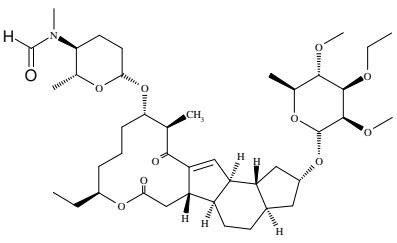


XDE-175-L (Minor component)

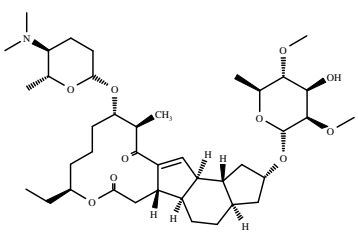
In this appraisal, the following abbreviated names were used for metabolites:



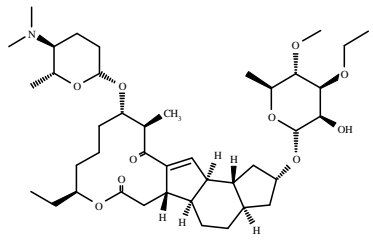
N-demethyl-175-J



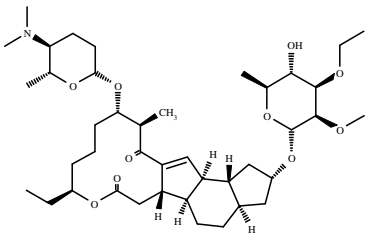
N-formyl-175-J



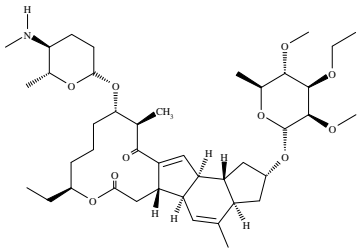
3'-O-deethyl-175-J



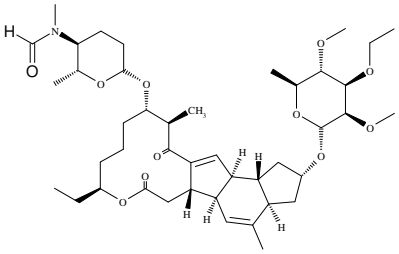
2'-O-demethyl-175-J



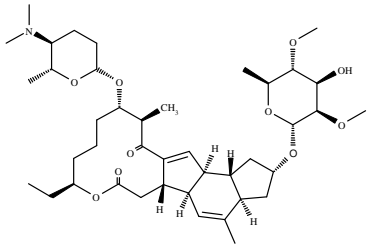
4'-O-demethyl-175-J



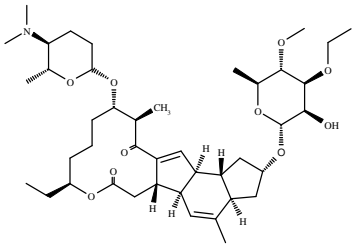
N-demethyl-175-L



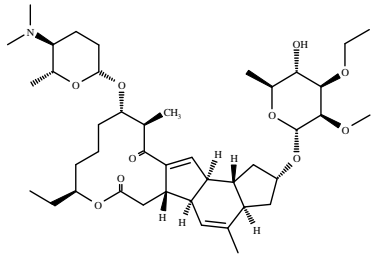
N-formyl -175-L



3'-O-deethyl-175-L



2'-O-demethyl-175-L



4'-O-demethyl-175-L

***Animal metabolism***

The Meeting received information on the fate of orally-dosed spinetoram in lactating goats and laying hens.

When either $^{14}$C-XDE-175-J or $^{14}$C-XDE-175-L, uniformly labelled with $^{14}$C in the macrolide ring, was administered orally at a dose equivalent to a dietary concentration of 10–11 ppm to a lactating goat once a day for five consecutive days, 51% or 78% of the administered dose of $^{14}$C-XDE-175-J or $^{14}$C-XDE-175-L, respectively, were recovered in faeces. Radioactivity recovered in urine was insignificant; less than 0.2% of the administered dose.

Radioactivity started to appear in milk within 24 hours after the first application but cumulative milk sample contained only about 0.3% of the total administered dose of $^{14}$C-XDE-175-J or 0.2% of that of $^{14}$C-XDE-175-L. The maximum total radioactive residues were 0.047 mg/kg in parent equivalents for $^{14}$C-XDE-175-L and 0.039 mg/kg $^{14}$C-XDE-175-L.

Total radioactive residues (TRR) in tissues after sacrifice (21 h after the last dose) showed the tendency to be higher in fatty tissues, with 0.235 mg/kg and 0.119 mg/kg in parent equivalents in fat (after the administration of $^{14}$C -XDE-175-J and $^{14}$C-XDE-175-L, respectively), and 0.116 mg/kg and 0.099 mg/kg in liver. TRR in kidney, muscle and milk were much lower.

The primary residue was XDE-175-J or XDE-175-L (42–84% of TRR) in all tissues and milk, except liver, indicating that minimal metabolism had occurred. In liver, XDE-175-J or XDE-175-L was the primary residue but at lower levels (30 or 26% of TRR) with N-demethyl-175-J or -L at very low levels (< 2% of the TRR), and an unidentified metabolite. No residue components other than the unchanged parent compounds were identified in milk, kidney or fat. Radioactive residues in muscle

also consisted primarily of XDE-175-J or XDE-175-L and much lesser amounts of what seemed to be the same unidentified metabolite as found in liver. There were many minor metabolites detected but all were less than 10% of the TRR. Unextracted radioactivity was less than 15% of the TRR in all samples except liver in which it was around 25%.

When either $^{14}$C-XDE-175-J or $^{14}$C-XDE-175-L, (uniformly labelled with $^{14}$C in the macrolide ring), was administered orally at a dose equivalent to a dietary concentration of 10 ppm to a group of laying hens once a day for seven consecutive days, 93% or 91% of the administered dose of $^{14}$C-XDE-175-J or $^{14}$C-XDE-175-L, respectively, were recovered in excreta.

Eggs and tissues contained a low proportion of the administered dose (< 3%) for both $^{14}$C-XDE-175–J and $^{14}$C-XDE-175-L. TRR in eggs increased over the experimental period and reached a maximum of 0.20 and 0.49 mg/kg for $^{14}$C-XDE-175-J and $^{14}$C-XDE-175-L, respectively, on day 7. TRR in the tissues were highest in abdominal fat (1.04 mg/kg for XDE-175-J, and 2.46 mg/kg for XDE-175-L), followed by skin with subcutaneous fat, liver, eggs and muscle. There is a tendency for radioactivity to be found at higher levels in tissues with higher fat content.

Unchanged XDE-175-J or XDE-175-L remained as the primary residue in the egg (58–49%) and tissues (45–70% of TRR) other than liver (13–12%). 3'-*O*-deethyl-175-J was detected in abdominal fat (1.8% of TRR) and in liver (18%) and *O*-demethyl-175-J was present in all tissues (3.2–6.5% of TRR) while 3'-*O*-deethyl-175-L and *O*-demethyl-175-L were found in all tissues and eggs (5.2–13% and 13–20% of TRR respectively). Unextracted radioactivity was less than 7% of the TRR in all samples except muscle and eggs in which it was 12–20%.

Limited metabolism of spinetoram was observed in ruminants and hens as the unchanged parent compound was the primary residue component in milk and all ruminant tissues as well as eggs and all avian tissues except liver for both XDE-175-J and XDE-175-L. Metabolism of spinetoram appears to be primarily through demethylation of the N-dimethyl moiety on the forosamine sugar to give the *N*-demethyl metabolite (goat) and dealkylation of the rhamnose sugar to give the *O*-deethyl and/or *O*-demethyl (two possible isomers) metabolites (hen).

***Plant metabolism***

The Meeting received information on the fate of spinetoram after foliar applications of $^{14}$C-XDE-175-J or $^{14}$C-XDE-175-L, uniformly labelled with $^{14}$C in the macrolide ring, on apple, lettuce and turnip representing the fruits, leafy crops and root crops respectively.

In all three crops tested, applied parent compounds decreased over test period. Among the two spinetoram components, XDE-175-L tended to be metabolized faster than XDE-175-J.

*Apple*

When a branch of apple tree with immature fruits was treated with single foliar application of either $^{14}$C-XDE-175-J or $^{14}$C-XDE-175-L at the rate of 1.8 kg ai/ha (4.8×) or 1.1 kg ai/ha (8.9×), respectively, apple fruits harvested seven days (PHI in US GAP) after the application contained 1.2 mg/kg or 0.36 mg/kg of radioactive residues. Washing of fruits (0–30 DAT) with acetonitrile and then dichloromethane removed 63–97% of TRR.

In the case of XDE-175-J application, in apples taken seven DAT, the parent compound was the major residue at 43% of the TRR with N-demethyl-175-J at 9.5% and N-formyl-175-J at 5.1% of the TRR. After the treatment with XDE-175-L, the parent compound was extensively degraded and metabolized and only 1.3% of the TRR remained as the parent compound with 1.0% of the TRR as N-demethyl-175-L and another 1.0% as N-formyl-175-L.

In the washed fruits obtained from the $^{14}$C-XDE-175-J treated branch, peels contained 2–11% of TRR while pulp contained less than 1% (≤ 0.007 mg/kg). In washed fruits from $^{14}$C-XDE-175-L treated branch, peel contained 6–33% of TRR while pulp contained less than 4%.

Unextractable radioactive residues were < 10% of TRR in or on all samples after extraction with a mixture of acetonitrile and water (80:20, v/v). Several minor metabolites were also detected in

the treated apples and leaves, each at ≤ 7.5% of TRR. A multi-component mixture of extensively degraded compounds represented up to 39–77% of TRR but each component was less than 1% of TRR.

Comparison of radioactivity in treated fruits and shielded fruits indicates that translocation was negligible.

*Lettuce*

Red leaf lettuce was treated with either single or multiple foliar spray applications of $^{14}$C-XDE-175-J or $^{14}$C-XDE-175-L. For $^{14}$C-XDE-175-J, single applications were made at rates equivalent to 0.90 kg ai/ha while the same amount of the test compound was sprayed in three separate applications with the equal rate at weekly intervals. For $^{14}$C-XDE-175-L, plants were treated in a similar fashion but at a rate equivalent to 0.30 kg ai/ha. For both compounds, the applied amounts approximately correspond to four times the maximum seasonal rate on the label and reflect the ratio between XDE-175-J and XDE-175-L in spinetoram formulations.

Washing leaves (0-7 DAT) with dichloromethane and then acetonitrile removed 76–96% of TRR.

The lettuce samples taken one day (PHI in US GAP) after the single application of XDE-175-J or XDE-175-L contained 34 mg/kg in parent equivalents or 7.6 mg/kg of radioactive residues, respectively. For treatment with XDE-175-J, the parent was 31%, N-demethyl-175-J was 20% and N-formyl-175-J was 11% of the TRR. For treatment with XDE-175-L, the parent was 12%, N-demethyl-175-L was 7.2% and N-formyl-175-L was 4.0% of the TRR. With the multiple applications, both TRR and percentage of these three compounds tended to be lower than with single applications of the same total rate for both parent compounds. Only 0.2–5.2% of TRR remained unextractable in all treated lettuce samples after extraction with acetonitrile/water (75:25, v/v).

Several minor metabolites were observed in the 14C-XDE-175-J and 14C-XDE-175-L treated lettuce at ≤ 6% of TRR. A multi-component mixture of extensively degraded compounds represented up to 13–78% of TRR, each component at less than 3% of TRR.

*Turnip*

Turnips were treated with either a single or multiple foliar applications of $^{14}$C-XDE-175-J or $^{14}$C-XDE-175-L in the same manner as in the lettuce study. In turnip roots harvested three days after the application, TRR was quite low at 0.12 mg/kg for XDE-175-J treatment and 0.031 mg/kg for XDE-175-L treatment.

In $^{14}$C-XDE-175-J treated turnip tops, 59–91% of TRR were surface residues found in the dichloromethane and acetonitrile washings. In $^{14}$C-XDE-175-L treated turnip tops, surface residues were 39–80% of TRR.

Major components identified at three DAT were the parent compounds (XDE-175-J and -L), *N*-dimethyl-175-J and *N*-formyl-175-J in roots and tops but all less than 25% of the TRR.

Several minor metabolites assumed to be structurally similar to the parent compound were also observed in treated turnip roots and tops, each less than 4% of TRR. A multi-component mixture of extensively degraded compounds represented 10–74% of TRR; each compound at less than 1% TRR.

Metabolism of spinetoram was observed to be similar in the three crops studied—apple, lettuce and turnip—indicating that a common metabolism is expected for not only fruits, leafy vegetables and root vegetables but also other plants. It appears that three metabolic pathways are responsible for the breakdown of spinetoram in plants. The first one involves changes to the N-demethyl moiety on the forosamine sugar to give the N-demethyl and N-formyl metabolites. N-formyl metabolites were found only in plants. The second involves cleavage of the macrolide ring system at one or more positions, ultimately resulting in a complex residue mixture consisting of numerous components. The third, (applicable only to XDE-175-J), involves changes to the rhamnose sugar

producing the 3-*O*-deethyl and C9-pseudoaglycone-175-J metabolites. All the metabolites occurring as a result of changes in forosamine and rhamnose are further degraded via the second pathway.

***Environmental fate in soil***

The Meeting reviewed information on aerobic soil metabolism, aqueous photolysis and hydrolysis, and rotational crop study, as spinetoram was intended for protection of root vegetables.

*Aerobic soil metabolism*

Aerobic soil metabolism studies were conducted using $^{14}$C-XDE-175-J or $^{14}$C-XDE-175-L, uniformly labelled with $^{14}$C in the macrolide ring applied to various soils and incubated under aerobic conditions at 25, 20 or 10 °C. Under aerobic conditions, spinetoram applied to soil was degraded relatively rapidly. In all soils tested, XDE-175-L was degraded faster than XDE-175-J. After one year of incubation at 25 °C, 1.2–2.8% and 0.3–2.9% of applied XDE-175-J and XDE-175-L respectively, remained as the parent in US soils tested. In European soils (except the loamy sand), after 127 days of incubation at 20 °C, 2.0–4.9% and 1.4–5.0% of applied XDE-175-J and XDE-175-L respectively remained as the parent. Carbon dioxide was evolved slowly from all soils and accounted for 5.0–35% and 9.5–32% of the applied XDE-175-J and XDE-175-L respectively after one year at 25 °C, and 0.8–1.1% and 1.2–3.2% of the applied XDE-175-J and XDE-175-L respectively after 127 days at 20 °C.

Major degradation products, N-demethyl-175-J and N-demethyl-175-L were formed and then degraded during the study periods. As minor products (at or less than 10% of the applied dose), N-demethyl-N-nitroso-175-J, N-demethyl-N-nitroso-175-L, N-succinyl-175-J and N-succinyl-175-L were also formed and degraded. Many other degradates were formed but at very low concentrations.

While extractable radioactivity decreased, non-extractable radioactivity steadily increased to reach 22–29% and 32–37% of the applied XDE-175-J and XDE-175-L respectively after one year at 25 °C; and 5–15% and 11–24% of the applied XDE-175-J and XDE-175-L respectively after 127 days at 20 and 10 °C.

*Aqueous photolysis*

Under xenon light (simulating 40°N latitude summer sunlight) in aqueous buffer solution at pH 7 at 25 °C, XDE-175-J and XDE-175-L degraded rapidly with $DT_{50}$ of 0.5 days and 0.3 days respectively. Numerous (more than 70) minor degradates were observed after irradiation of XDE-175-J and -L. N-demethyl-175-L was observed as a major photodegradation product of XDE-175-L.

At test termination, greater than 90% of the applied amount remained as parent in the dark controls indicating that negligible transformation of the parent compounds occurred in the dark. No degradates were observed in the dark controls.

*Aqueous hydrolysis*

In sterile aqueous buffer solutions at pH 5 and 7, no degradation was observed for both XDE-J and XDE-175-L for 30 days at 20 °C. At pH 9, a degradate of XDE-175-J was observed but the concentration of XDE-175-J did not decrease below 89% and therefore, XDE-175-J can be regarded to be relatively stable, also at pH 9. After 30 days at pH 9, XDE-175-L decreased from 92% to 82% with N-demethyl-175-L as the major degrade at 12% at the end of the testing period. No minor degradates were detected. $DT_{50}$ of XDE-175-L at pH 9 was calculated to be 154 days.

***Residues in succeeding crops***

In an outdoor confined rotation study, radish, lettuce and wheat were planted at 30, 120 and 365 days after the application of $^{14}$C-XDE-175-J or $^{14}$C-XDE-175-L at rate of 405 or 135 g ai/ha respectively to soil corresponding to the maximum seasonal rate and reflecting the ratio of these two active ingredients in spinetoram formulations.

TRRs were very low for all samples at all plant back intervals with the maximum at 0.085 mg/kg in parent equivalents. Unextractable residues in crops were less than 0.019 mg/kg.

Extraction of residues indicated that no greater than 0.065, 0.004 and 0.007 mg/kg were found in the neutral organic phases, acidic organic phases, and in the extracted aqueous phase, respectively. In any immature or mature sample, no single component exceeded 0.025 mg/kg or 0.007 mg/kg, respectively. At 120 DAT and 365 DAT, no radioactive residues were associated with any of XDE-175-L, N-demethyl-175-L or N-formyl-175-L. Of the XDE-175-J treated 120 DAT and 365 DAT crop samples, radish (immature tops and mature tops), lettuce (immature), and wheat forage, hay, and straw contained TRR greater than 0.010 mg/kg, but the concentrations were too low for identification. The lower residues in 30 DAT samples were characterized, but could not be identified.

The levels of radioactivity taken up from soil treated with [$^{14}$C]spinetoram into the three succeeding crops (radish, lettuce, and wheat) planted 30, 120, or 365 days after treatment, were below 0.085 mg/kg spinetoram equivalents. Since such low radioactive residues were found in analysed fractions of these rotational crop samples, spinetoram is unlikely to be taken up readily by succeeding crops.

### *Methods of analysis*

Analytical methods for determination of residues of spinetoram and its metabolites were developed for a wide range of matrices of plant and animal origin. In general, these methods employ extraction of spinetoram and its metabolites with a mixture of acetonitrile and water (80:20, v/v), addition of a stable isotope internal standard solution containing XDE-175 and metabolites, and then, without any clean-up or with solid phase clean-up using a C18 cartridge, analysis with HPLC with positive-ion electrospray tandem mass spectrometry (LC-MS/MS). Three methods, two for plant matrices and one for animal matrices, are capable of determining XDE-175-J, XDE-175-L, N-demethyl-175-J and -L, and N-formyl-175-J and -L. One method for animal matrices, however, determines XDE-175-J, XDE-175-L, N-demethyl-175-J and -L, and 3'-O-deethyl-175-J and -L.

The methods for plant matrices were validated for each analyte at 0.01–1.0 mg/kg, and in the case of lettuce at 0.01–10 mg/kg. Mean recovery was in a range of 82–111%. The validated limit of quantification was 0.01 mg/kg for all matrices.

The methods for animal matrices were validated for each analyte at 0.01–15 mg/kg in bovine muscle and kidney; 0.01–0.10 mg/kg in poultry muscle; 0.01–50 mg/kg in liver, milk and cream, and eggs; and 0.01–150 mg/kg in fat. Mean recovery ranged between 83 and 119%. The validated limit of quantification was 0.01 mg/kg for all matrices.

The existing multi-residue enforcement methods, FDA PAM I screen methods, were found to be unsuitable for the determination of spinetoram and its metabolites in plant and animal matrices. The DFG S19 multi-residue method was validated successfully only for the determination of spinetoram and its N-demethyl and N-formyl metabolites in apples, grapes and oranges.

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

Stability of spinetoram and its N-demethyl and N-formyl metabolites (each at a fortification level of 0.10 ppm) in homogenized orange, lettuce, sugar beet, soya bean and wheat grain stored in deep freezer at –20 °C was investigated over 12 months. No significant decrease of spinetoram was observed in all samples, except lettuce, during the test period. In lettuce, remaining XDE-175-L and N-dimethyl-175-L were 60 and 65% respectively (unadjusted for procedural recovery) at 372 days after initiation of the study.

The Meeting concluded that at –20 °C, spinetoram and its N-demethyl and N-formyl metabolites were stable for 12 months in orange, sugar beet, soya bean and wheat. In lettuce, XDE-175-J, the major component, and its N-demethyl and N-formyl metabolites were also stable for 12 months but XDE-175-L and N-dimethyl-175-L were stable only up to eight months.

As samples of animal tissues, milk and eggs from the metabolism and feeding studies were analysed within 20 days of sample collection in supervised trials, no information was provided to the Meeting on storage stability of spinetoram in animal commodities.

***Residue definition***

Spinetoram consists of two closely related active ingredients, XDE-175-J and XDE-175-L, present approximately in a three to one ratio.

In apple, lettuce and turnip receiving 4× to 9× the rate of either of the two active ingredients, major metabolites were the parent compounds (XDE-175-J and XDE-175-L), N-demethyl-175-J and N-formyl-175-J. In most cases, PHI XDE-175-J was the primary component of residues. N-demethyl-175-L and N-formyl-175-L were also detected but no more than 7.2% and 4.0% respectively of TRR on one DAT or thereafter.

In goats and hens, metabolism of spinetoram was limited. The parent compounds remained as major components in milk and all ruminant tissues as well as eggs, and all avian tissues except liver, in which 3'-*O*-deethyl metabolites were detected at similar levels as the parent, but less than 20%.

Sufficiently validated LC-MS/MS methods were available for determining the parent compounds and their N-demethyl and N-formyl metabolites in a wide range of plant commodities and animal tissues, milk and eggs.

Based on the above findings, the Meeting considered that the two parent compounds, XDE-175-J and XDE-175-L, were suitable residues for enforcement. However, as N-demethyl-175-J is a major metabolite in both plants and animals and covered by the ADI, and N-formyl-175-J, a major metabolite in plants, is also found in crops after application of spinetoram, the Meeting decided to include these two metabolites as well as the two spinetoram components in the residue definition for estimation of dietary intake.

XDE-175-J and XDE-175-L have logPow of 4.09 and 4.49 respectively at pH 7 at 20 °C, implying that spinetoram may be fat-soluble. In animal metabolism studies, residue concentrations were found to be higher in tissues with higher fat content. In addition, an animal feeding study with lactating cows indicates that spinetoram residue concentrations in milk fat were 4.4–9.5 times higher than those in whole milk and those in composite fat were 14–24 times higher than in muscle. The Meeting agreed that spinetoram residue is fat-soluble.

The Meeting recommended the following residue definition for plant and animal commodities:

- Definition of the residue (for compliance with the MRL):*Spinetoram.*
- Definition of the residue (for estimation of dietary intake):*Spinetoram and N-demethyl and N-formyl metabolites of the major spinetoram component.*
- The residue is fat-soluble.
- Note: Spinetoram consists of two related components.

***Results of supervised residue trials on crops***

The Meeting received supervised trial data for spinetoram on orange, pome fruits, stone fruits, leaf lettuce, tomato, sugar beet and tree nuts.

For all analytes and matrices, the LOQ was 0.01 mg/kg. The LOD was reported to be 0.003 mg/kg for trials conducted in the USA and 0.005 mg/kg for trials conducted in Australia.

*Citrus fruits*

Twelve supervised trials were conducted on oranges in the USA.

Six trials conducted using low spray volume applications (approximately 700 L/ha) were in accordance with US GAP for citrus fruits (maximum rate of 103 g ai/ha, three applications, maximum

seasonal rate of 210 g ai/ha, PHI one day). Spinetoram residues from these trials in rank order were: < 0.01 (2), 0.012, 0.022, 0.028 and 0.03 mg/kg.

Corresponding total residues of spinetoram and the two metabolites in rank order were: < 0.02, 0.022, 0.03, 0.052, 0.052 and 0.066 mg/kg.

Six other trials conducted using high spray volume applications (approximately 3300 L/ha) were in accordance with US GAP. Residues from these trials in rank order were: < 0.01, 0.012, 0.015, 0.018, 0.021 and 0.02 mg/kg.

Corresponding total residues of spinetoram and the two metabolites in rank order were: < 0.02, 0.039, 0.041, 0.046, 0.047 and 0.069 mg/kg.

The Meeting considered that these two sets of trials conducted in the same locations could not be regarded as independent from each other and decided to use one data set for the estimation of maximum residue level. Taking into consideration the results of the two data sets being mutually supportive, the Meeting estimated a maximum residue level based on spinetoram residues and an STMR based on the total residues of spinetoram and the two metabolites for oranges at 0.07 and 0.0435 mg/kg.

*Pome fruits*

Numerous supervised trials were conducted on apple in Australia (20), Canada (8), New Zealand (20) and the USA (12).

Six trials conducted in the USA using low spray volume applications (approximately700 L/ha) were in accordance with US GAP for pome fruits (maximum rate of 123 g ai/ha, five applications, maximum seasonal rate of 500 g ai/ha, PHI seven days). Spinetoram residues from these trials in rank order were: < 0.01 (3), 0.01, 0.013 and 0.028 mg/kg.

Corresponding total residues of spinetoram and the two metabolites in rank order were < 0.02 (3), 0.023, 0.036 and 0.038 mg/kg.

Six other trials conducted in the USA using high spray volume applications (approximately 3300 L/ha) were in accordance with US GAP. Spinetoram residues from these trials in rank order were < 0.01 (4), 0.012 and 0.02 mg/kg.

Corresponding total residues of spinetoram and the two metabolites in rank order were < 0.02 (2), 0.022, 0.022, 0.026 and 0.037 mg/kg.

Supervised trials were conducted in four locations in Canada and in the USA in accordance with Canadian GAP for pome fruits (maximum rate of 103 g ai/ha, three applications, maximum seasonal rate of 315 g ai/ha, PHI seven days). Since two plots were in each location, only higher residues were selected for each location. Spinetoram residues from these trials in rank order were < 0.01, 0.015, 0.017 and 0.028 mg/kg. These trials were also in compliance with US GAP. Corresponding total residues of spinetoram and the two metabolites in rank order were < 0.02, 0.025, 0.038 and 0.038 mg/kg.

Two trials conducted in Australia and five trials in New Zealand were according to GAP in New Zealand for pome fruits (maximum 2.5 g ai/hL, minimum 50 g ai/ha, four applications and PHI seven days). Spinetoram residues from these trials in rank order were < 0.01 (7) mg/kg. Corresponding total residues of spinetoram and the two metabolites in rank order were < 0.02 (7) mg/kg.

Although application rates were different (PHI in all related GAPs is seven days), results of trials matching three different GAPs were mutually supportive. In ten trials conducted in the USA and Canada following US GAP, which would lead to the highest residues, spinetoram residues were in rank order: < 0.01 (4), 0.01 0.013 0.015 0.017 and 0.028 ( 2) mg/kg. Corresponding total residues of spinetoram and the two metabolites in rank order were: <0.02 (3), 0.023, 0.025, 0.036, 0.038 (3) mg/kg.

Eight trials were conducted on pear in Australia and eight other in New Zealand. No trials in Australia matched GAP of New Zealand for pome fruits. Spinetoram residues from two trials conducted in New Zealand in accordance with GAP of New Zealand in rank order were < 0.01 and 0.02 mg/kg. Corresponding total residues of spinetoram and the two metabolites in rank order were < 0.02 and 0.03 mg/kg.

Since the results from trials on apple and pear were similar, the Meeting estimated a maximum residue level based on spinetoram residues and an STMR based on the total residues of spinetoram and the two metabolites, for pome fruits on a basis of apple trials, at 0.05 and 0.025 mg/kg respectively.

*Stone fruits*

A large number of supervised trials were conducted on cherry, peach and apricot in Australia and New Zealand. A few trials on nectarines were also conducted in Australia.

However, since proposed GAP in Australia for stone fruits has not been approved, no maximum residue level could be estimated.

*Tomato*

Six supervised trials conducted in the USA were according to US GAP for fruiting vegetables (maximum rate of 88 g ai/ha, six applications, maximum seasonal rate of 298 g ai/ha and PHI one day). Residues from these trials in rank order were < 0.01 (2), 0.01, 0.0156, 0.024 and 0.025 mg/kg.

Corresponding total residues of spinetoram and the two metabolites in rank order were < 0.02 (3), 0.02, 0.025, 0.034 and 0.035 mg/kg.

The Meeting estimated a maximum residue level based on spinetoram residues and an STMR based on the total residue of spinetoram and the two metabolites in tomato at 0.06 and 0.02 mg/kg respectively.

*Lettuce*

Six supervised trials were conducted on leaf lettuce in the USA in accordance with US GAP for leafy vegetables (maximum rate of 88 g ai/ha, six applications, maximum seasonal rate of 298 g ai/ha and PHI one day). Residues in rank order were 0.15, 0.31, 0.32, 0.34, 0.55 and 7.80 mg/kg.

Corresponding total residues of spinetoram and the two metabolites in rank order were 0.28, 0.56, 0.64, 1.16, 1.35 and 9.55 mg/kg.

The residue values of 7.80 and 9.55 mg/kg were very high compared to the rest of results. The study report indicates that the trial was conducted in the same manner as the other trials and there was no indication for this trial being invalid. Using all the residue values, the Meeting estimated a maximum residue level based on spinetoram residues and an STMR for spinetoram in lettuce at 10 and 0.895 mg/kg.

As foliar applications on leaf lettuce were expected to result in higher residues than those on head lettuce, the Meeting agreed that these maximum residue level and STMR are applicable also to head lettuce.

*Sugar beet*

Six supervised trials were conducted in the USA. In one trial according to US GAP (maximum rate of 70 g ai/ha, four applications, maximum seasonal rate of 281 g ai/ha and PHI seven days) for tuberous vegetables, e.g., potato and sugar beet, the residue was < 0.01 mg/kg. In the other five trials, root samples were taken three days after the last application, earlier than the required PHI of seven days in GAP. Residues in these five trials were < 0.01 mg/kg (5).

Corresponding total residues of spinetoram and the two metabolites in rank order were < 0.02 (6) mg/kg.

The Meeting estimated a maximum residue level based on spinetoram residues and an STMR for spinetoram in sugar beet at 0.01 (*) and 0.02 mg/kg respectively.

*Tree nuts*

Six supervised trials were conducted on almonds in the USA. One trial was in accordance with US GAP for tree nuts (maximum rate of 123 g ai/ha, four applications, maximum seasonal rate of 490 g ai/ha and PHI 14 days), and residues were < 0.01 mg/kg. In the other five trials, nut samples were taken seven days after the last application rather than the required PHI of 14 days in GAP. Residues in these five trials were < 0.01 mg/kg (5).

Corresponding total residues of spinetoram and the two metabolites in rank order were < 0.02 (6) mg/kg.

Six supervised trials were also conducted on pecan in the USA. In one trial according to US GAP for tree nuts, residues were < 0.01 mg/kg. In the other five trials, nut samples were taken seven days after the last application rather than the required PHI of 14 days in GAP. Residues in these five trials were < 0.01 mg/kg (2) and 0.01 (3) mg/kg. A decline study indicates that it is likely that residues would be less than 0.01 mg/kg if samples were taken on the required PHI of 14 days and in general residues were not expected to occur in edible portions of tree nuts due to negligible translocation of spinetoram.

From the results of trials on almonds and pecan, the Meeting estimated a maximum residue level based on spinetoram residues and an STMR based on the total residue of spinetoram and the two metabolites in tree nuts at 0.01 and 0.02 mg/kg.

*Sugar beet leaves or tops*

Among six supervised trials conducted in the USA, only one was in compliance with US GAP and residues were 0.024 mg/kg. In other trials, samples were collected only three days after the last application instead of the required PHI of seven days and they contained finite level of residues. However, these trials were in compliance with US GAP for leaf of root and tuberous vegetables for forage (maximum rate of 80 g ai/ha, four applications, maximum seasonal rate of 281 g ai/ha and PHI of three days). Residues from these trials were 0.086, 0.099, 0.11, 0.16 and 0.20 (2) mg/kg.

The Meeting estimated an STMR and a highest residue in sugar beet leaves or tops based on spinetoram residues for calculation of animal burden at 0.135 and 0.20 mg/kg.

*Almond hulls*

Among six supervised trials conducted in the USA, only one trial was in accordance with US GAP and residues are 0.75 mg/kg. In other trials, samples were collected seven days after the last application instead of the required PHI of 14 days and they contained finite level of residues. The Meeting concluded that it was not possible to estimate a maximum residue level for spinetoram in almond hulls from the results of these trials.

***Fate of residues during processing***

The Meeting received information on processing of oranges to juice and oil, and apples to juice and puree (sauce).

Processing factors were calculated for oranges (juice, peel and pulp after juicing, dried pulp and oil) and for apples (juice, dry pomace and puree (sauce)).

| Processed Orange Product | Processing factor | | STMR/STMR-P (mg/kg) |
|---|---|---|---|
| | Spinetoram residues | Spinetoram+2 metabolites | |
| Orange | - | - | 0.045 |
| Juice | < 0.05 | < 0.07 | 0.003 |
| Dried pulp | 2.4 | 2.3 | 0.105 |
| Apple | - | - | 0.025 |
| Juice | < 0.37 | < 0.44 | 0.011 |
| Dry pomace | 8.1 | 6.0 | 0.15 |

| Processed Orange Product | Processing factor | | STMR/STMR-P (mg/kg) |
|---|---|---|---|
| | Spinetoram residues | Spinetoram+2 metabolites | |
| Puree (sauce) | 0.45 | 0.47 | 0.012 |

For the purpose of calculating animal dietary burden for estimating maximum residue levels for commodities of animal origin, STMR-P for citrus pulp, dry and apple pomace, dry were calculated based on spinetoram residues to be 0.048 and 0.081 mg/kg.

***Farm animal dietary burden***

Dry apple pomace, dry citrus pulp and sugar leaves or tops may be fed to dairy cattle and beef cattle but not as a major ingredient. The dietary burdens were calculated from the highest residue and STMR of sugar beet leaves or tops, and the STMRs of apple pomace, dry, using the OECD feedstuffs tables (Annex 6 of the 2006 Report of the JMPR).

Summary of livestock dietary burdens (ppm of dry matter diet)

| | US-Canada | | EU | | Australia | |
|---|---|---|---|---|---|---|
| | max | mean | max | mean | max | mean |
| Beef cattle | 0.018 | 0.018 | 0.192[a] | 0.135[b] | 0.018 | 0.018 |
| Dairy cattle | 0.0089 | 0.0089 | 0.270[c] | 0.185[d] | 0.0089 | 0.0089 |

[a] Suitable for estimating maximum residue levels for meat and edible offal.

[b] Suitable for estimating STMRs for meat and edible offal.

[c] Suitable for estimating a maximum residue level for milk and fat.

[d] Suitable for estimating an STMR for milk and fat.

*Residues in milk and cattle tissues*

Lactating dairy cows were dosed daily for 29 consecutive days via gelatin capsules containing a mixture of spinetoram and N-demethyl and N-formyl metabolites of XDE-175-J (1.2–38.6 ppm in diet) or spinetoram only (37.6 ppm).

Residues in the milk in 1.2 ppm (equivalent to 0.4 mg XDE-175-J and -L) dose group were generally between the LOD (0.003 mg/kg) and LOQ (0.01 mg/kg) throughout the dosing period.

No or low concentration residues were detected in skim milk; even in 11.5 and 38.6 ppm (total) doses groups, mean total residues of the four compounds in skim milk ranged from just below the LOQ to 0.075 mg/kg. In all of the dose groups on day 14 and 28, total residues in cream were much higher than the residues in skim milk at 0.187 and 0.237 mg/kg in the 1.2 ppm doses groups. The mean total residues in cream from the 11.5 and 38.6 ppm doses groups ranged from 0.64 to 5.84 mg/kg. The average ratio of residues in cream to those in whole milk is 6:6.

All tissues from treated cows contained residues and they increased from the lowest to highest dose groups. Residue concentrations were lowest in the muscle followed by kidney, liver, and fat. Residues in fat were significantly higher than residues in the other tissues. These results indicate that residues of spinetoram tend to accumulate in fatty tissue.

With one exception, residues were not detectable in milk by the fourth day after the last dose was administered. Concentrations just above the LOD were detected in one cow through day nine after the final dose. No further residue was detected beyond that point.

Residues in tissues continuously declined through 28-day depletion period after the last dose. No residue was detected in kidney, liver or muscle from any cow by 28 days after the final dose or in fat 56 days following the final dose.

The dietary burdens for beef and dairy cattle are both lower than the lowest feeding level (1.2 ppm, equivalent to 0.4 ppm of spinetoram only) in the feeding study. Therefore the MRL and STMR were estimated using the residue concentrations in milk and tissues at the lowest feeding level and the dietary burden. The calculated residues in cattle tissues and milk are summarized below.

| Dietary burden mg/kg<br>Feeding level [mg/kg] | Spinetoram residues, mg/kg | | | | |
|---|---|---|---|---|---|
| | Milk | Muscle | Liver | Kidney | Fat |
| MRL | highest | highest | highest | highest | highest |
| 0.270/0.192 | < 0.00675 | < 0.00480 | < 0.00480 | < 0.00480 | 0.0743 |
| [0.4] | [< 0.01] | [< 0.01] | [< 0.01] | [< 0.01] | [0.11] |
| | Spinetoram, *N*-demethyl-175-J and *N*-formyl-175-J, mg/kg | | | | |
| STMR | mean | mean | mean | mean | mean |
| 0.185/0.135 | < 0.00925 | < 0.00625 | < 0.00625 | < 0.00625 | 0.0463 |
| [0.4] | [< 0.02] | [< 0.02] | [< 0.02] | [< 0.02] | [0.10] |

The Meeting estimated a maximum residue level for spinetoram in edible offal (mammalian) and whole milk at 0.01(*) mg/kg and in mammalian fats at 0.2 mg/kg. STMRs were estimated to be 0.00925 mg/kg for whole milk, 0.00625 mg/kg for meat (muscle) and edible offal (mammalian), and 0.046 mg/kg for mammalian fats.

The Meeting estimated a maximum residue level for spinetoram and an STMR for spinetoram and the two metabolites in milk fat, using the median ratio between residues in cream and whole milk of 6:6 and assuming that cream contains 50% fat, at 0.1 mg/kg and 0.12 mg/kg respectively.

*Poultry*

No data were provided on poultry feeding study. Since there was no treated commodities that can be fed to hens, the Meeting considered that it was unnecessary to estimate maximum residue levels for poultry tissues or eggs.

## RECOMMENDATIONS

On the basis of the data from supervised trials, the Meeting concluded that the residue levels listed below are suitable for establishing maximum residue limits and for IEDI assessment. Spinetoram is considered to be fat soluble.

Definition of the residue (for compliance with the MRL): *Spinetoram.*

Definition of the residue (for estimation of dietary intake*): Spinetoram and N-demethyl and N-formyl metabolites of the major spinetoram component.*

The residue is fat-soluble.

Note: Spinetoram consists of two related components.

| CCN | Commodity | Recommended MRL, mg/kg | | STMR or STMR-P (mg/kg) |
|---|---|---|---|---|
| | | New | Previous | |
| MO 0105 | Edible offal, mammalian | 0.01* | - | 0.00625 |
| VL 0482 | Lettuce, Head | 10 | - | 0.895 |
| VL 0483 | Lettuce, Leaf | 10 | | 0.895 |
| MM 0095 | Meat (from mammals other than marine mammals) | 0.2 (fat) | - | 0.00625 (muscle)<br>0.042 (fat) |
| FM 0183 | Milk fats | 0.1 | - | 0.12 |
| ML 0106 | Milks | 0.01* | - | 0.00925 |

| CCN | Commodity | Recommended MRL, mg/kg | | STMR or STMR-P (mg/kg) |
|---|---|---|---|---|
| | | New | Previous | |
| FC 0004 | Oranges, Sweet, Sour | 0.07 | - | 0.0435 |
| JF 0004 | Orange juice | | | 0.003 |
| FP 0009 | Pome fruits | 0.05 | | 0.025 |
| JF 0226 | Apple juice | | | 0.011 |
| | Apple puree or sauce | | | 0.012 |
| VR 0596 | Sugar beet | 0.01* | - | 0.02 |
| VO 0448 | Tomato | 0.06 | - | 0.02 |
| TN 0085 | Tree nuts | 0.01 | - | 0.02 |
| AB 0001 | Citrus pulp, dry | | | 0.048 [a] |
| AB 0226 | Apple pomace, dry | | | 0.081 [a] |
| AV 0596 | Sugar beet leaves or tops | | | 0.135 [a] |

[a] For dietary burden calculation. based on spinetoram residues only.

## DIETARY RISK ASSESSMENT

### *Long-term intake*

The International Estimated Dietary Intakes (IEDIs) of spinetoram were calculated for the 13 GEMS/Food cluster diets using STMRs estimated by the current Meeting (see Annex 3 of the 2008 Report of the JMPR). The ADI is 0–0.05 mg/kg bw and the calculated IEDIs were 0–1% of the maximum ADI. The Meeting concluded that the long-term intake of residues of spinetoram resulting from the uses considered by the current JMPR is unlikely to present a public health concern.

### *Short-term intake*

The 2008 JMPR decided that an ARfD is unnecessary. The Meeting therefore concluded that the short-term intake of residues of spinetoram is unlikely to present a public health concern.

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