DINOTEFURAN (255)

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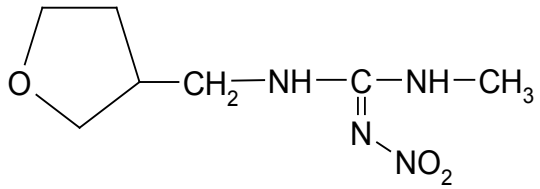
EXPLANATION

Dinotefuran is an insecticide used for the control a range of sucking insects, such as whiteflies, plant bugs, leafhoppers and mealybugs, in vegetables, fruit, paddy rice and turf. The formulated products can be applied to foliage, soil, nursery boxes and to paddy water by spray, drench, broadcast and 'pricking-in-hole' treatment. The Meeting received information on identity, animal and plant metabolism, environmental fate in soil, rotational crops, analytical methods, storage stability, use patterns, supervised trials, farm animal feeding studies and fate of residues in processing.

IDENTITY

Common name	Dinotefuran
Chemical name	
IUPAC:	(*RS*)-1-methyl-2-nitro-3-(tetrahydro-3-furylmethyl) guanidine
CAS:	N-methyl-N′-nitro-N″-[(tetrahydro-3-furanyl) methyl] guanidine
CAS Registry No:	165252-70-0
CIPAC No:	749
Synonyms:	MTI-446
Structural formula:	



Molecular formula:	$C_7H_{14}N_4O_3$
Molecular weight:	202.21

PHYSICAL AND CHEMICAL PROPERTIES

Pure active ingredient

Property	Results	Reference
Appearance	White solid (crystalline) (99.6% purity)	Shimono, 1999
Melting point	94.5–101.5 °C (99.5% purity)	Sydney, 1998
	107.5 °C (99.9% purity)	Malinski, 2000
Relative density	1.40 g/cm^3 at 20 °C (99.9% purity)	Malinski, 2000
Vapour pressure	5.0×10^{-5} Pa at 25 °C (99.5% purity) $< 1.7 \times 10^{-6}$ Pa at 30 °C $< 1.8 \times 10^{-6}$ Pa at 40 °C (99.9% purity) $< 2.1 \times 10^{-6}$ Pa at 50 °C	Sydney, 1998 Malinski, 2000
Volatility (Henry's law constant)	The Henry's Law Constant for dinotefuran at 20 °C was not calculated because of the lack of actual vapour pressure results.	Labano, 2011 (Expert statement)

Property	Results	Reference
Solubility in water	39.0 ± 2.1 g/L in purified water at 10 °C 54.3 ± 1.3 g/L in purified water at 20 °C 89.7 ± 2.5 g/L in purified water at 30 °C 52.3 ± 1.0 g/L in pH5.0 buffer solution at 20 °C 54.5 ± 0.8 g/L in pH7.0 buffer solution at 20 °C 51.2 ± 1.8 g/L in pH9.0 buffer solution at 20 °C (99.5% purity)	Sydney, 1998
	39.83 g/L at 20 °C (99.9% purity)	Malinski, 2000
Partition coefficient n-octanol/water	Log Pow = -0.915 at pH5.0 at 25 °C Log Pow = -0.644 at pH7.0 at 25 °C Log Pow = -0.760 at pH9.0 at 25 °C (99.5% purity)	Sydney, 1998
	Log Pow = -0.549 at 25 °C (99.9% purity)	Malinski, 2000
Hydrolysis	Dinotefuran was hydrolytically stable at pH4, 7 and 9 at 50 °C. Less than 10% hydrolysis was observed after 5 days. At pH11 and 13 at 50 °C, significant hydrolysis was observed, with respective half-lives of 45 hours and 4.2 hours obtained. (99.5% purity)	Sydney, 1998 95/MTO098/1216
Photolysis	DT_{50} = 0.9 days in buffer solution (pH7) at 23 °C DT_{50} = 2.4 days at 50 °N, 2.3 days at 40 °N and 2.3 days at 30 °N to natural sunlight	Van der Gaauw, 2002
Dissociation constant	pKa = 12.6 (99.9% purity)	Malinski, 2000
Solubility in organic solvents (99.9% purity)	Hexane: 9.0 g/L at 20 ± 0.5 °C Heptane: 10.5 g/L at 20 ± 0.5 °C Xylene: 71.8 mg/L at 20 ± 0.5 °C Toluene: 148 mg/L at 20 ± 0.5 °C Dichloromethane: 60.9 g/L at 20 ± 0.5 °C Acetone: 57.8 g/L at 20 ± 0.5 °C Methanol: 57.2 g/L at 20 ± 0.5 °C Ethanol: 19.4 g/L at 20 ± 0.5 °C Ethyl acetone: 5.17 g/L at 20 ± 0.5 °C	Malinski, 2000

Formulations: Water soluble granule (SG) 200 g ai/kg or 700 g ai/kg

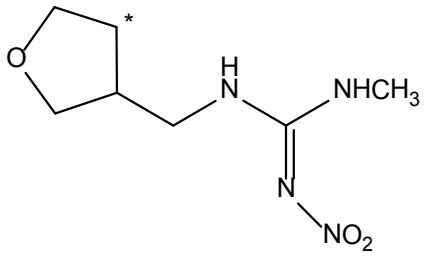
Water dispersible granule (WG) 200 g/kg

Wettable powder (WP) 100 g/kg

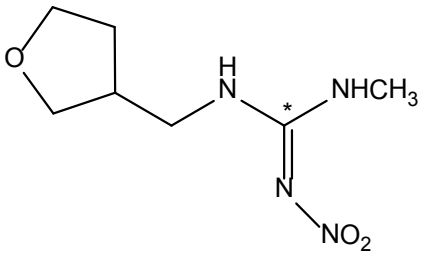
Water soluble liquid (SL) 100 g ai/L

METABOLISM AND ENVIRONMENTAL FATE

The metabolism of dinotefuran has been investigated in animals and plants. The fate and behaviour of dinotefuran in animals, plants and the environment was investigated using the [^{14}C] labelled test materials shown in Figures 1.



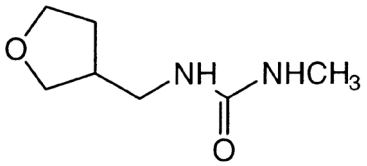
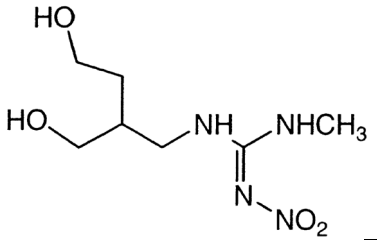
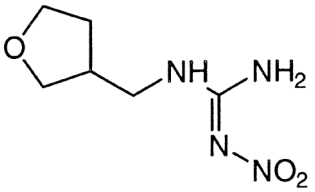
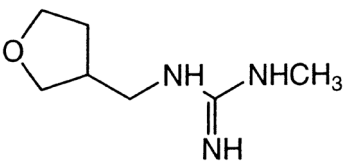
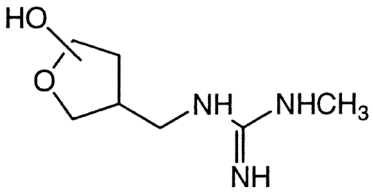
[^{14}C-furanyl]dinotefuran

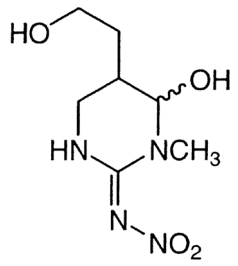
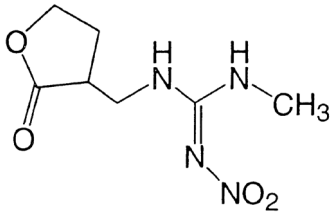
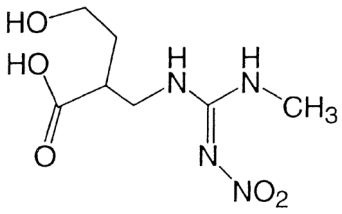
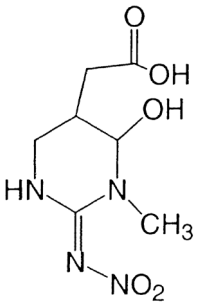
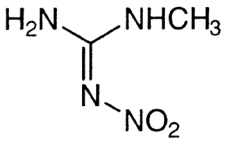
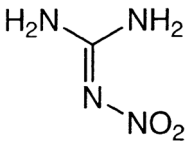
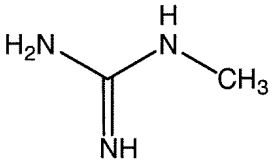


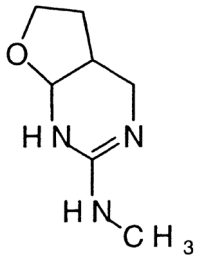
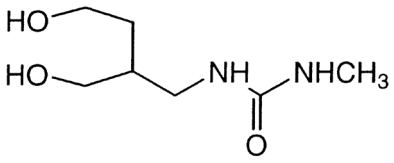
[^{14}C-guanidine]dinotefuran

Figure 1 [^{14}C]-Labelled test materials used in animals, plants metabolism studies, and the environmental fate studies

The chemical structures of the major degradation compounds from the metabolism of dinotefuran are provided below.

Compound name		Structure	Found in metabolism studies
UF	1-methyl-3-(tetrahydro-3-furylmethyl)urea		Rat, Livestock, Plants
446-DO	1-[4-hydroxy-2-(hydroxymethyl) butyl] -3-methyl-2-nitroguanidine		Rat, Livestock, Plants
FNG	2-nitro-1-(tetrahydro-3-furylmethyl)guanidine		Rat, Livestock, Plants
DN	1-methyl-3-(tetrahydro-3-furylmethyl)guanidium dihydrogen		Rat, Livestock, Plants
DN-OH	1-(2-hydroxytetrahydro-3-furylmethyl)-3-methylguanidine (DN-2-OH) or 1-(3-hydroxytetrahydro-3-furylmethyl)-3-methylguanidine (DN-3-OH)		Rat, Plants

Compound name		Structure	Found in metabolism studies
PHP	6-hydroxy-5-(2-hydroxyethyl)-1-methyl-1,3-diazinane-2-ylidene-*N*-nitroamine		Rat, Livestock, Plants
446-CO	1-methyl-2-nitro-3-(2-oxotetrahydro-3-furylmethyl)guanidine		Rat, Livestock
446-OH+COOH	2-(2-hydroxyethyl)-3-(3-methyl-2-nitroguanidino) propionic acid		Rat, Livestock
PHP-COOH	2-(6-hydroxy-1-methyl-2-(nitroimino)-1,3-diazinane-5-yl)acetic acid		Livestock
MNG	1-methyl-2-nitroguanidine		Rat, Plants
NG	Nitroguanidine		Plants
MG	1-methylguanidine		Rat, Plants

Compound name		Structure	Found in metabolism studies
BCDN	3-(methylamino)-9-oxa-2-aza-4-azoniabicyclo[4.3.0]non-3-ene hydrogen		Rat, Plants
UF-DO	1-[4-hydroxy-2-(hydroxymethyl) butyl]-3-methylurea		Plants

Animal metabolism

The Meeting received studies on the metabolism of dinotefuran in rats, lactating goat and laying hens. The study on rats was evaluated by the WHO Core Assessment Group of the 2012 JMPR. A summary of the rat metabolism is given in this section.

Rats

In rats given [^{14}C]furanyl or [^{14}C]-guanidine-labelled dinotefuran orally by gavage, absorption was rapid and accounted for at least 88% of the total administered radioactivity after a single low dose (50 mg/kg bw) or high dose (1000 mg/kg bw). The maximum plasma concentrations of radioactivity were reached after approximately 0.5 and 2 hours after administration of the low and high doses, respectively, whereas the half-lives in plasma ranged from 4 to 15 hours for the low and high dose, respectively. Radioactivity was widely distributed throughout the body. Elimination of the radioactivity was mainly via urine (≥ 88% of the administered dose), whereas elimination via faeces accounted for 1–3% after oral administration and 1% after intravenous administration. Residues in tissues 168 hours after a single oral or intravenous dose as well as after repeated oral dosing accounted for less than 0.5% of the administered radioactivity, and the concentrations in most tissues were below the limit of detection (0.001 mg/kg).

Metabolism of dinotefuran in rats was limited, with more than 90% of the dose being eliminated as unchanged parent molecule, which was also the major component in plasma, milk, bile and most tissues collected 4–8 hours after administration. About 20 metabolites were identified; the metabolic routes included hydroxylation on the tetrahydrofuran ring, followed by further oxidation, reduction and acetylation. Other routes of metabolism involved desmethylation, nitro-reduction and hydrolysis.

Lactating goat

The metabolism of dinotefuran by the lactating dairy goats was studied by Hatzenbeler and Lentz (2002: 013346-1). A goat was orally dosed with gelatine capsules containing a 1:1 mixture of [^{14}C]furanyl and [^{14}C]guanidine dinotefuran, once daily for 5 consecutive days. Each daily dose was equivalent to approximately 10 ppm based on the feed consumption of 1672 g/day. The animals were dosed immediately after the morning milking and daily sample collections.

Milk, urine, faeces and cage washes were collected during the dosing period. Milk samples were collected twice daily. Animals were sacrificed within 5–8 hours of the last dose. Composite muscle (loin, front and rear leg), composite fat (omental and perirenal), liver, kidney, heart, blood, GI tract contents and GI tract tissue samples were collected.

Approximately 100% of the administered [^{14}C]furanyl and [^{14}C]guanidine-dinotefuran was recovered from the goats. Radioactive residues in the faeces, cage wash and urine accounted for 81.9% of the total administered dose. Total ^{14}C residues in the milk accounted for 0.3% of the administered dose. Tissues contained approximately 1.1% of the administered dose. The remaining 16.6% of the administered radioactivity resided in the gastrointestinal tract and its contents. The total radioactive residue (TRR), expressed as mg dinotefuran equivalent/kg, in milk, muscle, fat, liver, kidney, heart and blood is summarized in Table 1. The TRR levels in milk reached a steady state of approximately 0.045 mg eq/kg by Day 2 of dosing. The residue levels detected in milk, liver, kidney, fat, muscle, blood and heart were low (0.012–0.272 mg eq/kg) and accounted for 0.01–0.73% of the administered dose. The majority of the dose was excreted in urine and faeces.

Table 1 Total radioactive residues (TRR) in goat milk and tissues

Sample	TRR (mg dinotefuran equivalent/kg)	% of administered dose
Milk [a]	0.044–0.097	0.03–0.07
Muscle	0.044	0.73
Fat	0.012	0.01
Liver	0.138	0.15
Kidney	0.272	0.05
Heart	0.045	0.01
Blood	0.049	0.17

[a] For Day 1–4 p.m. and a.m. milk were combined and residue levels of the combined sample were 0.044–0.047 mg eq/kg. Day 5 (a partial day) sample consisting of p.m. milk only and showed higher residue levels (0.097 mg eq/kg)

Solvent (ethanol and water) extracted 97.2% of TRR (0.043 mg ^{14}C eq/kg) in milk. Dinotefuran was the major component at 40.1% TRR (0.018 mg/kg). Minor metabolites were PHP, 446-DO, UF and FNG, all < 10% TRR and < 0.005 mg/kg. Bound residues were detected at only 0.001 mg eq/kg in the post extraction solids (PES).

Approximately 93.8% of the TRR in muscle (0.041 mg eq/kg) was extracted with solvent (acetonitrile and water). The remaining 6.2% TRR (0.003 mg eq/kg) was present in the PES as bound residues. The major residues present in the muscle sample were parent dinotefuran (0.018 mg/kg, 41.3% TRR) and UF (0.006 mg eq/kg, 14.6% TRR). Minor metabolites PHP, FNG and DN each accounted for less than 7% TRR and ≤ 0.003 mg eq/kg.

Solvent (acetonitrile and water) extracted 91.4% (0.011 mg eq/kg) of the TRR in fat. Parent dinotefuran (0.002 mg/kg) was the major residue detected in the extract accounting for 20% of the TRR. Minor metabolites UF, FNG and DN each accounted for less than 9% TRR and ≤ 0.001 mg eq/kg. Bound residues accounted for approximately 8.6% of the TRR (0.001 mg eq/kg).

Approximately 76.3% (0.105 mg eq/kg) of the TRR in liver was extracted with solvent (acetonitrile and water). Dinotefuran was present as the major residue (0.017 mg/kg, 12.1% TRR) in the liver extracts. Minor metabolites FNG, UF and DN were all detected in the liver at < 10% TRR and < 0.01 mg eq/kg. Bound residues were only 0.033 mg eq/kg (23.7% TRR) in the PES.

The liver PES (23.7% TRR, 0.033 mg eq/kg) was subject to mild acid extraction. Mild acid released an additional 20.2% of the TRR (0.028 mg eq/kg) from the PES. The HPLC profile of the acid extracts was shown to be similar to the neutral liver extracts above. The final bound residue in the liver following neutral and acid extraction was 3.4% TRR (0.005 mg eq/kg).

Approximately 93.3% (0.254 mg eq/kg) of the TRR in kidney was extracted with solvent (acetonitrile and water). FNG (0.055 mg eq/kg, 20.1% TRR) was the major metabolite detected in the kidney extract. Parent dinotefuran was present as the second major residue and accounted for 12.7% TRR (0.035 mg/kg). Minor metabolites PHP, UF and DN were all detected in the kidney at < 10% TRR and 0.009–0.017 mg eq/kg. Bound residues were detected at only 0.018 mg eq/kg (6.7% TRR) in the PES.

The major residue present in urine was unchanged parent dinotefuran accounted for 49.4% TRR (2.346 mg/kg). Minor metabolites PHP, UF, FNG and DN were each less than 7% TRR and ≤ 0.3 mg eq/kg in the urine.

Greater than 70% (2.190 mg eq/kg) of the TRR was extracted from a representative faeces sample. DN was the major metabolite accounted for 48.7% of the TRR (1.479 mg eq/kg) in the faeces extracted fraction. Unchanged parent accounted for 12.4% TRR and 0.377 mg/kg. Minor metabolites PHP, UF and FNG each accounted for less than 1% TRR and ≤ 0.03 mg eq/kg in the faeces extractables. Bound residues accounted for 27.9% TRR (0.848 mg eq/kg).

The major extracted metabolites of dinotefuran present in goat tissues were identified. These metabolites resulted from several metabolic reactions, including loss of the nitro group to form DN, loss of the methyl group to form FNG, hydrolysis of the nitroimino moiety to form UF, hydrolysis of tetrahydrofuran ring to form 446-DO, and intramolecular ring formation to form PHP. Minor metabolites included NG and/or MNG in trace levels and conjugates of NG, MNG, PHP and 446-DO.

Table 2 Dinotefuran and related residues identified in milk and tissues

Compound	Milk, Day 3	Muscle	Fat	Liver	Kidney
	% of TRR (mg dinotefuran equivalent/kg)				
Extract	97.2 (0.043)	93.8 (0.041)	91.4 (0.011)	76.3 (0.105)	93.3 (0.254)
Dinotefuran	40.1 (0.018)	41.3 (0.018)	20.0 (0.002)	12.1 (0.017)	12.7 (0.035)
PHP	8.1 (0.004)	4.6 (0.002)	nd	nd	3.4 (0.009)
446-DO	9.1 (0.004)	nd	nd	nd	nd
UF	8.7 (0.004)	14.6 (0.006)	7.4 (0.001)	6.8 (0.009)	5.0 (0.014)
FNG	6.9 (0.003)	6.5 (0.003)	3.1 (BDL)	5.1 (0.007)	20.1 (0.055)
DN	nd	3.7 (0.002)	8.5 (0.001)	2.2 (0.003)	6.2 (0.017)
Others [a]	nd	12.5 (0.005)	28.1 (0.004)	15.5 (0.021)	21.4 (0.057)
Polars [a, b]	24.3 (0.010)	10.6 (0.005)	25.0 (0.003)	34.6 (0.048)	24.5 (0.067)
Unextracted (PES)	2.8 (0.001)	6.2 (0.003)	8.6 (0.001)	23.7 (0.033)	6.7 (0.018)
TRR	0.044	0.044	0.012	0.138	0.272

[a] All single components detected in the extractable fraction were less than 10% of the TRR

[b] Consists of multiple components as determined in the kidney.

nd = Not detected

Based on the identified metabolites, a metabolic pathway of dinotefuran in goat is proposed in Figure 2.

Laying hens

The elimination, distribution and metabolic fate of dinotefuran by egg laying hens were studied by Hatzenbeler and Lentz (2002: 013347-1). Ten Leghorn hens were orally dosed with a 1:1 mixture of [^{14}C]furanyl and [^{14}C]guanidine dinotefuran, once daily for 5 consecutive days. Each daily dose was equivalent to approximately 10 ppm relative to food intake.

Excreta and eggs were collected during the dosing period. A cage rinse was collected at termination. The hens were sacrificed within 4–5 hours after the last dose. Liver, composite fat (abdominal tissue and skin), composite muscle (thigh and breast), blood, undeveloped eggs and gastrointestinal tract samples were collected.

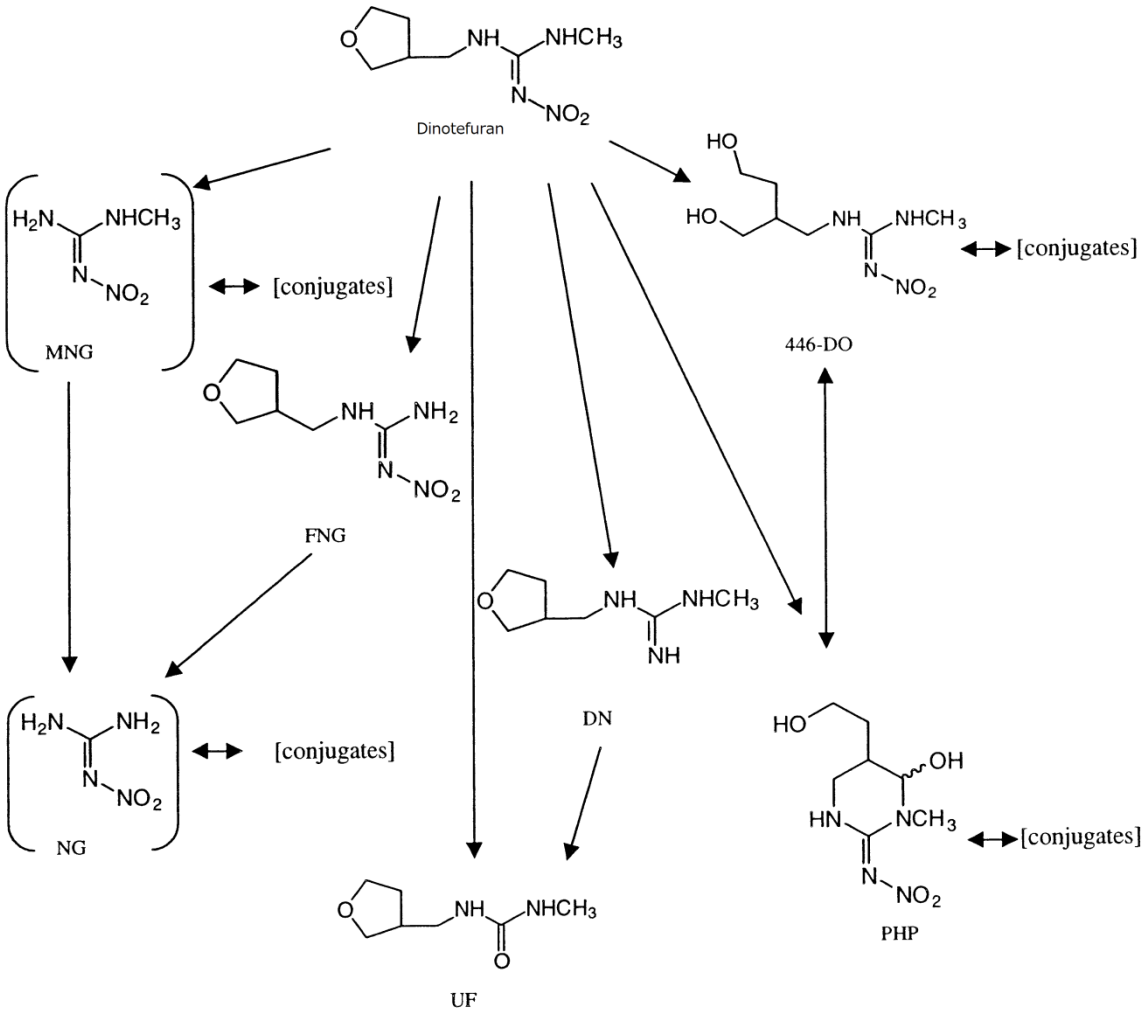


Figure 2 Proposed Metabolic Pathway of Dinotefuran in Lactating Goat

Approximately 91% of the administered [^{14}C]furanyl and [^{14}C]guanidine dinotefuran was recovered from the hens. Radioactive residues in the excreta and cage wash accounted for 88.9% of the total administered dose. Total ^{14}C residues in the eggs accounted for 0.07% (0.02% in the yolk and 0.05% in the white) of the administered dose. Tissues contained approximately 0.23% of the administered dose. The gastrointestinal tract and its contents accounted for 1.31% of the administered radioactivity residues. The total radioactive residue (TRR), expressed as mg dinotefuran equivalent/kg, in egg yolk, egg white, liver, fat, muscle and blood is summarized in Table 3.

Egg white (Day 4), egg yolk (Day 4), liver, fat, muscle and excreta (Day 4) samples were extracted with various solvents. The metabolites isolated from excreta were parent dinotefuran and FNG.

Table 3 Total radioactive residues (TRR) in egg and tissues of hens

Sample	TRR (mg dinotefuran equivalent/kg)	% of administered dose
Egg white	0.007–0.023	0.05
Egg yolk	BDL–0.024	0.02
Muscle	0.049	0.12
Fat	0.010	0.01
Liver	0.134	0.09
Blood	0.084	0.01

BDL = Below Detection Limit

Solvent (acetonitrile and water) extracted 94.9% TRR (0.0216 mg eq/kg) in egg white. Dinotefuran was the major component at 57.0% TRR (0.0130 mg/kg). The minor metabolite FNG was detected at 13.1% TRR (0.0030 mg eq/kg). All other components were detected at < 0.003 mg eq/kg in the egg white. Bound residues were detected at only 0.0012 mg eq/kg in the post-extraction solids (PES).

Solvent (acetonitrile and water) extracted 82.4% TRR (0.0133 mg eq/kg) in egg yolk. Dinotefuran was the major ^{14}C residue at 44.2% TRR (0.0071 mg/kg). Minor metabolite FNG was detected at 8.0% TRR (0.0013 mg eq/kg). All other components were detected at ≤ 0.002 mg eq/kg in the egg yolk. Bound residues were detected at only 0.0028 mg eq/kg in the PES.

Solvent (acetonitrile and water) extracted 80.6% TRR (0.0433 mg eq/kg) in muscle. Dinotefuran was detected at 9.1% TRR (0.0049 mg/kg). Minor metabolites FNG, UF and DN were detected at 2.2% TRR (0.0012 mg eq/kg), 7.4% TRR (0.0040 mg eq/kg) and 6.7% TRR (0.0036 mg eq/kg), respectively. All other components were detected at < 0.008 mg eq/kg in the muscle. Bound residues were detected at only 0.0104 mg eq/kg in the PES.

Solvent (acetonitrile and water) extracted 93.7% TRR (0.0100 mg eq/kg) in fat. Dinotefuran was detected at 10.8% TRR (0.0012 mg/kg). Minor metabolites FNG and UF were detected at 5.6% TRR (0.0006 mg eq/kg) and 6.0% TRR (0.0006 mg eq/kg), respectively. All other components were detected at < 0.003 mg eq/kg in the fat. Bound residues were detected at only 0.0007 mg eq/kg in the PES.

Solvent (acetonitrile and water) extracted 64.4% TRR (0.0786 mg eq/kg) in liver. Dinotefuran was detected at 9.3% TRR (0.0113 mg/kg). Minor metabolites DN, FNG and UF were detected at 3.3% TRR (0.0040 mg eq/kg), 6.5% TRR (0.0080 mg eq/kg) and 4.6% TRR (0.0056 mg eq/kg), respectively. In the HPLC, a polar region of interest was detected at 33.1% TRR (0.0404 mg eq/kg). Characterization of the polar region showed that it consisted of multiple components each present at less than 0.005 mg eq/kg except one region which was present at 0.0173 mg eq/kg. LC/MS analysis of this region showed that it consisted of multiple components with molecular weight of 234 (possibly 446-OH + COOH) and 265. All other components were detected at < 0.01 mg eq/kg in the liver. Bound residues were detected at 0.0435 mg eq/kg in the PES.

Bound residues present in the liver sample were subjected to a mild acid extraction (50:50, ACN: 1 N HCl, v:v). Approximately 7.7% TRR was released from the bound residue of the liver PES using this mild acid extraction. The HPLC profile was shown to be similar to the neutral liver extracts above. Since approximately 28.0% TRR (0.0344 mg eq/kg) was still bound after mild acid extraction of the liver PES, the solids were subjected to acid hydrolysis with 1 N HCl. Approximately 16.4% of the TRR (0.0202 mg eq/kg) was released by acid hydrolysis. The HPLC profile of the acid hydrolysis extract showed no individual component was present at levels greater than 0.005 mg eq/kg.

Excreta contained dinotefuran (24.4% TRR), FNG (16.1% TRR), metabolites with molecular weights of 232 (possibly PHP-COOH, 9.3% TRR), 234 (possibly 446-OH + COOH, 28.0% TRR) and 216 (possibly 446-CO, 9.4% TRR).

The major extracted metabolites of dinotefuran present in hen tissues were identified. These metabolites resulted from several metabolic reactions, including loss of the nitro group to form DN, loss of the methyl group to form FNG, hydrolysis of the nitroimino moiety to form UF, hydrolysis of the tetrahydrofuran to form 446-DO and intramolecular ring formation to form PHP. These metabolites underwent further metabolism forming NG and/or MNG at trace levels and possibly 446-OH+COOH, 446-CO and PHP-COOH.

Table 4 Dinotefuran and related residues identified in egg and tissues

Compound	Egg whites, Day 4	Egg yolk, Day 4	Muscle	Fat	Liver
	% of TRR (mg dinotefuran equivalent/kg)				
Extract	94.9 (0.0216)	82.4 (0.0133)	80.6 (0.0433)	93.7 (0.0100)	64.4 (0.0786)
Dinotefuran	57.0 (0.0130)	44.2 (0.0071)	9.1 (0.0049)	10.8 (0.0012)	9.3 (0.0113)

Compound	Egg whites, Day 4	Egg yolk, Day 4	Muscle	Fat	Liver
	% of TRR (mg dinotefuran equivalent/kg)				
Unknown 1 (MW = 232)	6.3 (0.0014)	8.7 (0.0014)	5.8 (0.0031)	11.8 (0.0013)	nd
Unknown 2 (MW = 234 [a])	3.8 (0.0009)	nd	3.9 (0.0021)	6.2 (0.0007)	2.2 (0.0026)
Unknown 3 (MW = 216 [b])	3.5 (0.0008)	9.0 (0.0014)	1.9 (0.0010)	7.7 (0.0008)	5.4 (0.0066)
UF	nd	nd	7.4 (0.0040)	6.0 (0.0006)	4.6 (0.0056)
FNG	13.1 (0.0030)	8.0 (0.0013)	2.2 (0.0012)	5.6 (0.0006)	6.5 (0.0080)
DN	nd	nd	6.7 (0.0036)	nd	3.3 (0.0040)
Others	nd	nd	43.7 (0.0234)	3.6 (0.0004)	nd
Polars [c]	24.3 (0.0025)	12.5 (0.0020)	nd	23.8 (0.0026)	33.1 (0.0404)
Unextracted (PES)	5.1 (0.0012)	17.6 (0.0028)	19.4 (0.0104)	6.3 (0.0007)	35.6 (0.0435)
TRR	0.0227	0.0161	0.0537	0.0107	0.1221

[a] This region may also contain PHP based on HPLC retention time.

[b] This region may also contain 446-DO based on HPLC retention time.

[c] Consists of multiple components as determined in the liver.

nd = Not detected

Based on the identified metabolites, a metabolic pathway of dinotefuran in hen is proposed in Figure 3.

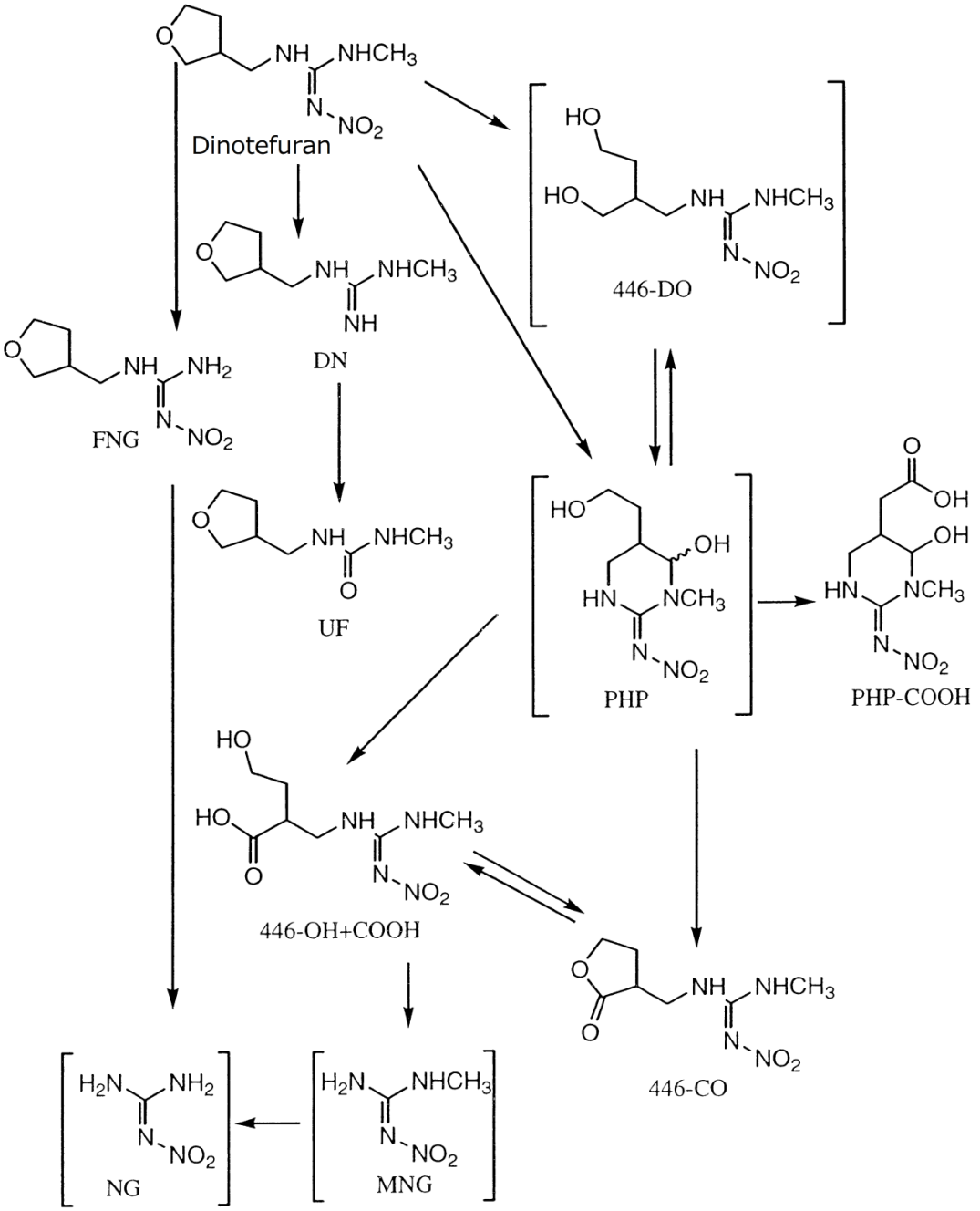


Figure 3 Proposed Metabolic Pathway of Dinotefuran in Laying Hens

Summary of animal metabolism

The metabolism of ^{14}C labelled dinotefuran has been studied in lactating goat and laying hens. In both studies, dinotefuran was metabolized to several metabolites. Dinotefuran was the most important component in milk and muscle for lactating goat, and in eggs for laying hens.

Plant metabolism

Plant metabolism studies were performed on apples, lettuce, rice and oilseed rape with dinotefuran ^{14}C-labelled in two positions to track metabolites from different parts of the molecule following cleavage. Metabolites were identified using multiple chromatographic systems and authentic standards.

Apples

The metabolism of dinotefuran in apples (variety Granny Smith) was studied under field conditions. The spray application was conducted using an equimolar mixture of [^{14}C]furanyl]-dinotefuran and [^{14}C]guanidine]-dinotefuran (Figure 1). The test substance was applied as a foliar spray using a 20% SG formulation at a nominal rate of 200 g ai/ha (1× rate) on one tree and at an exaggerated rate treatment at 2000 g ai/ha (10× rate) on the second tree to generate metabolites for identification (Panthani, 2002: 012786-1). The trees were treated 21 days before mature fruit harvest. Mature fruits were collected at harvest and analysed for total radioactive residues (TRR) and the nature of major residues. Leaf samples were collected immediately after test substance application and at fruit harvest. Leaves were analysed for TRR. Mature apple fruits from each group were washed with water to remove surface residues. The surface-washed fruit were cut into small pieces, and the fruit homogenized. The fruit homogenates were centrifuged to give an aqueous fraction (juice) and a solid fraction (pomace). Aliquots from the juice fraction were weighed and analysed by LSC to determine the total radioactivity. The total radioactivity in the pomace fraction was determined by combustion analysis or by extraction method (as the sum of extracted and unextracted ^{14}C). The residue levels in the whole fruit are derived from the sum of the contributions from surface wash, solid fractions (pomace), and aqueous fractions (juice).

The overall residue levels (TRR) in the mature apples were 0.153 mg dinotefuran equivalent/kg for fruits from the apple tree treated at the nominal application rate (1×) and 1.924 mg eq/kg in apples from exaggerated rate (10×) treatment. The pomace fraction was extracted with 70% aqueous methanol and the extractable residues were characterized by HPLC-RAD. The surface wash (water rinse), extract from the intact fruit and the juice fraction were also characterized by HPLC-RAD. The ^{14}C-residue profiles of the surface wash, pomace extract and juice of mature apples treated at 1× and 10× rate were qualitatively similar. Fractions of purified juice from mature apples were used for identifying and characterizing the nature of ^{14}C residues.

Table 5 Recovery of total radioactive residues in apple fruit

Application rate	Fraction	[^{14}C]dinotefuran	
		TRR (%)	mg/kg
200 g ai/ha (1× rate)	Surface wash	69.1	0.106
	Aqueous fraction (juice)	21.3	0.033
	Solids (pomace)	9.5	0.015
	Total (whole fruit)	100.0	0.153
2000 g ai/ha (10× rate)	Surface wash	62.1	1.194
	Aqueous fraction (juice)	27.5	0.530
	Solids (pomace)	10.4	0.200
	Total (whole fruit)	100.0	1.924

Dinotefuran and metabolites, PHP, 446-DO, UF, BCDN, DN, FNG, dehydrated PHP, UF-DO, NG and MNG were detected in the apple fractions. A number of minor ^{14}C peaks were also present in chromatograms and these minor components eluted as partially separated peaks in the polar region.

Dinotefuran was one of the major ^{14}C residues present in the apple fruit accounting for approximately 29–33% TRR in the mature apples (0.044 mg/kg, 28.8% TRR–1×; 0.633 mg/kg, 32.9% TRR–10×).

UF accounted for 0.031 mg/kg, 20.0% TRR–1× rate and 0.403 mg/kg, 20.9% TRR–10× rate. DN was present at a concentration ranging from 0.016 mg/kg, 10.4% TRR–1× rate and 0.134 mg/kg, 6.9% TRR–10× rate. PHP was also present at > 10% TRR (0.021 mg/kg, 13.5% TRR–1×; 0.254 mg/kg, 13.2% TRR–10×). NG, MNG, 446-DO, BCDN, UF-DO and FNG were identified in the apple fruit as minor metabolites (< 5% TRR). A polar fraction was detected in chromatograms at a level > 10% (15.2% TRR–1× and 12.2% TRR–10×). NG and MNG each accounted for approximately 2% of the polar fraction. The concentration of several (5–6) unknown polar components was also determined by TLC. None of these components was more than 3.7% of the TRR.

Table 6 Distribution of total radioactive residues in mature apple fruits

	200 g ai/ha (1×)		2000 g ai/ha (10×)	
	TRR (%)	mg/kg	TRR (%)	mg/kg
Extract	96.6	0.148	97.6	1.878
Dinotefuran	28.8	0.044	32.9	0.633
NG [a]	1.7	0.003	1.6	0.032
MNG [a]	1.9	0.003	1.7	0.033
PHP [b]	13.5	0.021	13.2	0.254
446-DO	1.5	0.002	2.7	0.052
UF	20.0	0.031	20.9	0.403
BCDN [c]	3.2	0.005	2.6	0.050
DN	10.4	0.016	6.9	0.134
UF-DO	2.5	0.004	3.6	0.070
Unknown (13.8 min Peak)	0.2	< 0.001	1.0	0.019
FNG	1.2	0.002	1.5	0.029
Polar unknowns [d]	11.7	0.018	8.9	0.171
Unextracted	3.4	0.005	2.4	0.046
Total	100.0	0.153	100.0	1.924

[a] NG and MNG eluted with a number of minor polar components in the early region of the chromatogram. The individual components of the region were quantitated by TLC after acid hydrolysis.

[b] PHP was converted to dehydrated PHP during region isolation using HPLC mobile phase containing acid and concentration by rotary evaporation. The concentration of PHP is the sum of PHP and dehydrated PHP.

[c] BCDN was identified by LC/MS comparison with BCDN succinate standard and TLC.

[d] contained 5–6 individual components

Dinotefuran was extensively metabolized in apples. These metabolites resulted from several metabolic reactions, including N-demethylation to form FNG, loss of the nitro group to form DN, hydrolysis of the nitroimino moiety to form UF, tetrahydrofuran portion of the molecule to form MNG. N-demethylation of MNG resulted in the formation of NG.

Based on the results of the study, it can be concluded that dinotefuran, when sprayed onto apples, stayed on the surface of the fruit. Only a small percentage of the applied dinotefuran penetrated slowly into the fruit and was extensively metabolized to polar products.

Lettuce

The metabolism of [^{14}C] dinotefuran in lettuce was studied under greenhouse conditions The test substance was applied as a 1:1 mixture of [^{14}C]furanyl]–and [^{14}C]guanidine]-dinotefuran as a foliar spray using a 20% SG formulation at a nominal rate of 150 g ai/ha. An exaggerated rate treatment at 1500 g ai/ha was carried out to generate metabolites for identification (Panthani, 2002: 012785-1). Lettuce plants were treated 14 days before mature harvest (approximately 8 weeks from seeding).

Residue levels in the mature lettuce were 1.791 and 10.616 mg/kg for the 150 and 1500 g ai/ha applications, respectively. The solvent extracted radioactivity from the mature lettuce accounted for 97.6 and 98.0% TRR for the 150 and 1500 g ai/ha applications, respectively.

The metabolite profiles of lettuce extracts from 150 and 1500 g ai/ha were qualitatively similar. Dinotefuran and metabolites, PHP, 446-DO, UF, DN-OH, BCDN, DN, NG and MNG were detected in the lettuce extracts based on the HPLC retention time comparison with reference standards and/or their relative retention time. A number of minor ^{14}C peaks were also present and these minor components eluted as partially separated peaks in the polar region.

The distribution of ^{14}C residues (sum of free and conjugate) was calculated from the analyses of the extracts and also of acid hydrolysed extracts.

Dinotefuran was the major residue in the mature lettuce. The level of dinotefuran residues in the lettuce ranged from 1.103 mg/kg (61.6% TRR) to 6.864 mg/kg (64.7% TRR) for the 150 and 1500 g ai/ha applications, respectively. PHP, 446-DO, UF, DN-OH, BCDN, DN, NG and MNG were detected as metabolites in the mature lettuce, but none of these metabolites were present at greater

than 5% TRR level. The data below summarizes the distribution of ^{14}C residues in the lettuce extracts. Among these metabolites, relatively higher amounts of PHP, UF and DN were detected as compared to the other minor metabolites, approaching 4–5% of the TRR level. Levels of unextracted residues in the mature lettuce were less than 2.5% of the TRR.

Table 7 Distribution of ^{14}C residue in the mature lettuce after post-emergence application of [^{14}C]Dinotefuran 14 days before harvest

	150 g ai/ha		1500 g ai/ha	
	TRR (%)	mg/kg	TRR (%)	mg/kg
Extract	97.57	1.748	97.95	10.399
Dinotefuran	61.57	1.103	64.66	6.864
NG	1.06	0.019	0.46	0.049
MNG	2.64	0.047	1.45	0.154
PHP	5.11	0.092	5.08	0.539
446-DO	2.97	0.053	3.60	0.382
UF	3.79	0.068	4.08	0.434
DN-OH	1.02	0.018	1.22	0.130
BCDN	2.39	0.043	2.68	0.284
DN	4.98	0.089	3.88	0.412
Polar Unknown [a]	4.90	0.088	2.92	0.310
Other Unknowns [b]	7.11	0.127	7.91	0.840
Unextracted	2.43	0.044	2.05	0.218
Total	100.0	1.791	100.0	10.616

[a] Four minor unidentified polar components each representing less than 2% of the TRR.

[b] Minor unknowns each represent less than 2% of the TRR.

Potatoes

The metabolism of [^{14}C]dinotefuran in potato plants grown outdoors under field conditions was investigated. Dinotefuran, as a 1:1 mixture of the two labels, was applied to potato plants as a foliar spray at the BBCH stage 50–59 (just before flowering). The application rates were 100 g ai/ha, 200 g ai/ha (six plants each), and 1000 g ai/ha (exaggerated dose; one plant) (Mamouni, 2002: RCC 734310). One potato plant at each of the two lower application rates was harvested as “new potatoes” (54 days after treatment). The remaining five plants per dosage as well as the plant of exaggerated dose were harvested at maturity (75 days after treatment).

The results obtained by direct combustion are summarized in Table 8. The radioactive residue is reported in mg dinotefuran equivalents per kg fresh weight.

Table 8 Total radioactive residues in potatoes

Harvest	Dose rate (g ai/ha)	Measured by combustion						
		Foliage mg/kg	Whole potato mg/kg	Peel		Pulp		Total mg/kg
				%TRR	mg/kg	%TRR	mg/kg	
First 54 DAT [a]	100	0.706	0.029	14.7	0.002	85.3	0.013	0.016
	200	0.499	0.036	11.4	0.001	88.6	0.010	0.012
Second 75 DAT	100	1.053	0.007	17.1	0.002	82.9	0.007	0.009
	200	0.664	0.013	18.2	0.003	81.8	0.013	0.016
	1000	3.014	0.078	25.7	0.028	74.3	0.081	0.109

[a] DAT: days after treatment

The nature of the radioactive residue in the potato tubers was investigated. Tubers (whole potato) were homogenised and extracted with acetonitrile/water and water. These extracts were combined, concentrated, purified and analysed by HPLC and TLC. Additionally, the total radioactive residue in peel and pulp (peeled potato tubers) was determined. The total radioactive residue in the whole potato was very low for the 100 and 200 g ai/ha treatment rates, even at the first harvest interval. The majority of the radioactive residue was extractable at room temperature with acetonitrile/water followed by water (94.5% to 97.7% of TRR for all samples).

In the first harvest (100 g ai/ha), dinotefuran accounted for 13.1% of TRR corresponding to 0.004 mg/kg. MNG was found at a concentration of 0.002 mg/kg (7.6% TRR). Four other metabolites were detected at concentrations of 0.001 mg/kg and were characterized by co-chromatography with reference metabolites as UF, PHP, 446-DO and FNG. At the 200 g ai/ha application rate, the metabolic pattern was qualitatively similar to that of the 100 g ai/ha dose rate. Dinotefuran accounted for 8.5% of TRR corresponding to 0.003 mg/kg. The concentrations of the other metabolites were all less than 0.01 mg/kg.

The metabolic pattern of the mature potatoes was qualitatively similar to that of the first harvest, but the concentrations of dinotefuran and metabolites were even lower.

Table 9 Distribution of [^{14}C]dinotefuran and its metabolites in whole potato extracts

	First Harvest				Second Harvest					
	100 g ai/ha		200 g ai/ha		100 g ai/ha		200 g ai/ha		1000 g ai/ha	
	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg
Extract	97.5	0.029	97.7	0.036	94.5	0.007	94.9	0.013	96.4	0.078
Dinotefuran	13.1	0.004	8.5	0.003	13.0	0.001	14.5	0.002	10.8	0.009
UF	3.6	0.001	6.3	0.002	3.5	< 0.001	7.0	0.001	6.7	0.005
PHP	4.6	0.001	6.1	0.002	6.9	0.001	6.9	0.001	5.8	0.005
M3 (partly NG)	61.7	0.019	57.1	0.021	69.0	0.005	37.5	0.005	50.7	0.041
FNG	4.4	0.001	5.6	0.002	2.1	< 0.001	4.4	0.001	8.0	0.006
446-DO	2.5	0.001	4.7	0.002	[a]	[a]	3.9	0.001	5.0	0.004
MNG	7.6	0.002	9.6	0.003	[a]	[a]	20.7	0.003	9.4	0.008
Unextracted	2.5	0.001	2.3	< 0.001	5.5	0.001	5.2	0.001	3.6	0.002

[a] Not detected or below the limit of quantification.

The concentrations of parent dinotefuran and metabolite in the whole potato tubers 54 days after treatment with [^{14}C]dinotefuran at rates of 100 and 200 g ai/ha (first harvest, "new potato") were very low ranging between 0.001 and 0.004 mg/kg. At the second harvest interval (maturity, 75 days after treatment), the concentrations of all radioactive fractions were markedly decreased to values between below the detection limit and 0.005 mg/kg. The metabolic pattern remained the same. Despite the low concentrations it was possible to confirm the identity of dinotefuran and six metabolites (UF, PHP, 446-DO, NG, MNG and FNG) by co-chromatography on TLC and HPLC with reference compounds.

Rice

The metabolism of [^{14}C] dinotefuran in rice was studied under greenhouse conditions to determine the amount and nature of total ^{14}C residues in rice grain and straw (McClanahan, 2000: 7466-98-0021-EF-001-001). The study consisted of a preliminary phase and a definitive phase. The preliminary phase was used to determine if metabolic cleavage of the two labels occurred, determine if dinotefuran or metabolites were volatilized, obtain preliminary tissue samples, and develop extraction methods for tissue samples. In the definitive phase, experiments simulating rice-paddy application of dinotefuran were performed to quantify total ^{14}C levels in rice plant tissue and grain and identify major components of the residue and their distribution in the plant. In each phase, rice plants at appropriate stages were treated by soil treatment or by spray treatment at a rate of 0.40 kg ai/ha. For soil treatment, the water from the pot in which the rice is being grown was drained, and a water solution of dinotefuran was applied to the soil surface drop-wise. The rice plants were then flooded to an appropriate depth. For spray treatment, the rice plants were sprayed with an appropriate formulation of dinotefuran.

For the preliminary study, two separate test substances were used ([^{14}C]furanyl-dinotefuran and [^{14}C]guanidine-dinotefuran). Rice seedlings were treated with each test substance 7 days after transplant. Final harvest occurred 14 days after treatment. The results of the preliminary study indicated there was no metabolic separation of the two labelled moieties of dinotefuran. The major residue in each of the samples analysed from the preliminary study was dinotefuran. Volatility and CO_2 production were means of dissipation of the radioactivity in the preliminary study.

For the definitive study, one test substance was used, which consisted of an equimolar mixture of [^{14}C]furanyl-dinotefuran and [^{14}C]guanidine-dinotefuran. Rice plants were grown in pots in a greenhouse. There were two application times (5 and 20 days after bolting, DAB) (bolting: BBCH 55) and two application methods (soil and spray application). Extracts of whole rice grain, brown rice, polished rice, chaff, bran, straw, root and soil were analysed by HPLC to determine the number of metabolites and their relative distribution in the samples. The structures of the metabolites of dinotefuran were determined by several methods. Qualitatively, the metabolic composition of each rice plant matrix was similar, regardless of the application method or application timing.

Residue levels in whole grain from the soil applications were low (0.345 and 0.396 mg/kg for the 5 and 20 DAB soil applications, respectively). Residues were greatly reduced when the chaff was removed from the whole grain to produce brown rice. The residues were further reduced upon polishing of the brown rice to produce polished rice. The residue levels in brown rice were 0.055 and 0.052 mg/kg for the 5 and 20 DAB applications, while the residue levels in polished rice were 0.033 and 0.039 mg/kg for the 5 and 20 DAB applications.

For soil treatment, extracted radioactivity from the whole rice grain accounted for 78.9 and 81.4% TRR for the 5 and 20 DAB applications, respectively. Extracted radioactivity from the brown rice accounted for 65.4 and 61.0% TRR for the 5 and 20 DAB applications. Dinotefuran was the major residue in whole rice grain and brown rice. The level of dinotefuran residues in the whole grain ranged from 0.228 mg/kg (66.0% TRR) to 0.240 mg/kg (60.5% TRR) for the 5 and 20 DAB applications, respectively. Dinotefuran residues were greatly reduced when the chaff was removed from the whole grain to produce brown rice, accounting for only 0.015 mg/kg (26.3% TRR) and 0.014 mg/kg (26.2% TRR) for the 5 and 20 DAB applications, respectively. The residues of dinotefuran were further reduced upon polishing of the brown rice to produce polished rice, decreasing to 0.010 mg/kg (29.9% TRR) and 0.008 mg/kg (21.2% TRR) for the 5 and 20 DAB applications, respectively. PHP, 446-DO, UF, DN-OH, BCDN, DN and Region A were detected as metabolites in whole grain or brown rice. Among these metabolites, relatively higher amounts of UF were detected as compared to the other minor metabolites, accounting for 0.019 mg/kg (5.6% TRR) and 0.024 mg/kg (6.1% TRR) in whole grain from the 5 and 20 DAB applications, and 0.005 mg/kg (8.6% TRR) and 0.003 mg/kg (6.4% TRR) in brown rice from the 5 and 20 DAB applications.

Total residues in rice straw were 1.822 and 1.347 mg/kg for the 5 and 20 DAB soil applications, respectively. Extracted radioactivity accounted for 81.5 and 81.8% TRR for the 5 and 20 DAB applications, respectively. Dinotefuran was the major residue in straw, accounting for 0.965 mg/kg (53.0% TRR) and 0.695 mg/kg (51.6% TRR) for the 5 and 20 DAB applications, respectively. PHP, 446-DO, UF, DN-OH, BCDN, DN and a group of compounds eluting as Region A were detected as metabolites in straw. Among these metabolites, relatively higher amounts of UF and DN were observed as compared to the other minor metabolites. UF accounted for 0.215 mg/kg (11.8% TRR) and 0.181 mg/kg (13.4% TRR) in straw from the 5 and 20 DAB applications, while DN accounted for 0.091 mg/kg (5.0% TRR) and 0.089 mg/kg (6.6% TRR) for the 5 and 20 DAB applications. Aliquots of straw were surface-washed with water. For the soil application, surface-washing removed only 4.9 and 6.1% TRR. Dinotefuran was the major residue in the straw surface wash, accounting for 0.038 and 0.045 mg/kg for the 5 and 20 DAB applications. Metabolites eluting in Region A, PHP, 446-DO, UF and DN were also present in the straw surface washes. Dinotefuran was the major residue in the surface-washed straw. Region A, PHP, 446-DO, UF and DN were also present.

Residues in rice root from the soil application were low, accounting for 0.107 and 0.126 mg/kg for the 5 and 20 DAB applications. Extracted radioactivity accounted for 68.9 and 65.0% TRR for the 5 and 20 DAB applications, respectively. Dinotefuran and DN were the major residues in root. Dinotefuran accounted for 0.021 and 0.039 mg/kg for the 5 and 20 DAB applications, while DN accounted for 0.045 and 0.024 mg/kg for the 5 and 20 DAB applications.

Residues in soil from soil application were similar to those in rice root, accounting for 0.138 and 0.213 mg/kg for the 5 and 20 DAB applications. Extracted radioactivity accounted for 62.0 and 47.2% TRR for the 5 and 20 DAB applications, respectively. Dinotefuran and DN were the major

residues in soil. Dinotefuran accounted for 0.057 and 0.065 mg/kg for the 5 and 20 DAB applications, while DN accounted for 0.025 and 0.034 mg/kg for the 5 and 20 DAB applications. The half-life of dinotefuran in soil was calculated from the data from the 5 DAB application (including the data for the 25 DAB sampling from the 5 DAB application) to be 37 days.

Table 10 Summary of residue levels in rice plant tissues (final harvest)

Sample	Residue Level (mg/kg) [a]				
	Control	Soil application at 5 DAB	Soil application at 20 DAB	Foliar spray at 5 DAB	Foliar spray at 20 DAB
Whole Grain	0.011	0.345	0.396	5.845	5.096
Brown Rice	0.008	0.055	0.052	0.611	0.338
Chaff	0.027	1.126	1.061	33.831	19.012
Polished Rice	0.008	0.033	0.039	0.335	0.151
Bran	0.014	0.196	0.120	4.037	1.730
Straw	0.022	1.822	1.347	7.570	8.146
Washed straw [b]	NA [c]	1.362	1.218	7.790	11.172
Roots	0.005	0.107	0.126	0.015	0.022
Soil	0.008	0.138	0.213	0.010	0.014

[a] Residue levels were determined as the sum of residues in extracts, PES and surface wash (washed straw only), except for control samples and spray application roots and soil, where residue levels were determined by combustion.

[b] A separate aliquot of straw was analysed by surface-washing and then extraction.

[c] NA = Not analysed. No surface wash was done on control straw.

Total residues in the whole rice grain for the spray applications accounted for 5.845 mg/kg (5 DAB application) and 5.096 mg/kg (20 DAB application). Residues were greatly reduced during processing to produce the brown rice and polished rice fractions, similar to what was observed for the soil application. The residue levels in brown rice were 0.611 and 0.338 mg/kg for the 5 and 20 DAB applications, while the residue levels in polished rice were 0.335 and 0.151 mg/kg for the 5 and 20 DAB applications.

Extracted radioactivity from the whole rice grain accounted for 70.2 and 81.4% TRR for the 5 and 20 DAB applications, respectively. Extracted radioactivity from the brown rice accounted for 89.7 and 92.7% TRR for the 5 and 20 DAB applications. Dinotefuran was the major residue in whole rice grain and brown rice. The level of dinotefuran in the whole grain ranged from 2.098 mg/kg (35.9% TRR) to 2.687 mg/kg (52.7% TRR) for the 5 and 20 DAB applications, respectively. Dinotefuran residues were greatly reduced when the chaff was removed from the whole grain to produce brown rice, accounting for only 0.204 mg/kg (33.4% TRR) and 0.181 mg/kg (53.6% TRR) for the 5 and 20 DAB applications, respectively. The residues of dinotefuran were further reduced upon polishing of the brown rice to produce polished rice, decreasing to 0.140 mg/kg (41.7% TRR) and 0.073 mg/kg (48.4% TRR) for the 5 and 20 DAB applications, respectively. PHP, 446-DO, UF, DN-OH, BCDN, DN and a group of compounds eluting as Region A were detected as metabolites in whole grain or brown rice. Among these metabolites, relatively higher amounts of UF were observed as compared to the other minor metabolites, accounting for 1.006 mg/kg (17.2% TRR) and 0.697 mg/kg (13.7% TRR) in whole grain from the 5 and 20 DAB applications, and 0.105 mg/kg (17.2% TRR) and 0.048 mg/kg (14.1% TRR) in brown rice from the 5 and 20 DAB applications.

Total residues in rice straw were 7.570 and 8.146 mg/kg for the 5 and 20 DAB spray applications, respectively. Extracted radioactivity accounted for 93.88 and 95.48% TRR for the 5 and 20 DAB applications, respectively. Dinotefuran was the major residue in straw, accounting for 4.037 mg/kg (53.3% TRR) and 5.617 mg/kg (69.0% TRR) for the 5 and 20 DAB applications, respectively. PHP, 446-DO, UF, DN-OH, BCDN, DN and a group of compounds eluting as Region A were detected as metabolites in straw. Among these metabolites, relatively higher amounts of UF and DN were observed as compared to the other minor metabolites. UF accounted for 1.204 mg/kg (15.9% TRR) and 0.718 mg/kg (8.8% TRR) in straw from the 5 and 20 DAB applications, while DN accounted for 0.645 mg/kg (8.5% TRR) and 0.467 mg/kg (5.7% TRR) for the 5 and 20 DAB applications. Aliquots of straw were surface-washed with water. For the spray application, surface-washing removed only 15.5 and 13.3% TRR. Dinotefuran was the major residue in the straw surface

wash, accounting for 0.792 and 0.994 mg/kg for the 5 and 20 DAB applications. Metabolites eluting in Region A, PHP, 446-DO and UF were also present in the surface wash. Dinotefuran was the major residue in the surface-washed straw. Region A, PHP, 446-DO, UF and DN were also present.

Residues in root and soil from the spray application were very low (0.015–0.022 mg/kg for roots, 0.010–0.014 mg/kg for soil).

Table 11 Distribution of metabolites in rice grain following soil application of [^{14}C]dinotefuran 5 and 20 days after bolting

	%TRR and mg/kg in Plant Matrix [a]									
	Rice Grain Matrices, 5 DAB									
Fraction	Whole Grain		Brown Rice		Chaff		Polished Rice		Bran	
	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg
Extract	78.91	0.272	65.41	0.036	76.73	0.864	54.34	0.018	78.13	0.153
Dinotefuran	65.97	0.228	26.30	0.015	50.87	0.573	29.94	0.010	27.74	0.054
Region A [b]	0.63	0.002	15.83	0.009	5.68	0.064	5.92	0.002	19.06	0.037
PHP	ND [c]	ND	3.07	0.002	1.53	0.017	2.34	0.001	4.77	0.009
446-DO	1.03	0.004	2.09	0.001	ND	ND	3.15	0.001	ND	ND
UF	5.60	0.019	8.57	0.005	12.05	0.136	7.55	0.003	7.27	0.014
DN-OH	0.55	0.002	ND	ND	ND	ND	ND	ND	ND	ND
BCDN	0.79	0.003	ND	ND	ND	ND	ND	ND	ND	ND
DN	2.68	0.009	2.75	0.002	4.37	0.049	0.82	< 0.0005	4.53	0.009
Others [d]	1.66	0.006	6.80	0.004	2.23	0.025	4.62	0.002	14.77	0.029
Unextracted (PES)	21.09	0.073	34.59	0.019	23.27	0.262	45.66	0.015	21.87	0.043
Total	100.00	0.345	100.00	0.055	100.00	1.126	100.00	0.033	100.00	0.196
	Rice Grain Matrices, 20 DAB									
Fraction	Whole Grain		Brown Rice		Chaff		Polished Rice		Bran	
	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg
Extract	81.43	0.322	60.96	0.032	78.58	0.834	38.26	0.015	72.80	0.088
Dinotefuran	60.50	0.240	26.15	0.014	53.04	0.563	21.19	0.008	26.94	0.032
Region A [b]	1.71	0.007	14.75	0.008	2.67	0.028	7.46	0.003	18.71	0.023
PHP	2.12	0.008	3.35	0.002	2.04	0.022	1.87	0.001	3.93	0.005
446-DO	2.85	0.011	2.26	0.001	ND	ND	1.19	0.001	ND	ND
UF	6.11	0.024	6.40	0.003	12.02	0.128	4.74	0.002	6.84	0.008
DN-OH	1.06	0.004	ND	ND	ND	ND	ND	ND	ND	ND
BCDN	2.61	0.010	ND	ND	ND	ND	ND	ND	ND	ND
DN	4.48	0.018	2.32	0.001	3.93	0.042	0.54	< 0.0005	3.79	0.005
Others [d]	ND	ND	5.73	0.003	4.87	0.052	1.26	0.001	12.60	0.015
Unextracted (PES)	18.57	0.074	39.04	0.020	21.42	0.227	61.74	0.024	27.20	0.033
Total	100.00	0.396	100.00	0.052	100.00	1.061	100.00	0.039	100.00	0.120

[a] Calculations were based on mg/kg level determined as sum of residue levels from extracts and PES.

[b] Region A consisted of multiple radioactive components eluting from 2 to 8 min. This region may contain MNG and conjugates of UF, PHP and 446-DO by analogy to chaff.

[c] ND = Not detected (below limit of detection). Non-detect values are calculated as zero.

[d] Consists of low-level radioactivity; no discernible peaks.

Table 12 Distribution of metabolites in root and straw following soil application of [^{14}C] dinotefuran 5 and 20 days after bolting

	%TRR and mg/kg in Matrix [a]									
	Other Matrices, 5 DAB				Straw for Surface Washing [b]					
Fraction	Root		Straw		Washed straw		Straw surface wash		Total	
	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg
Extract	68.90	0.074	81.50	1.485	73.20	0.997	4.94	0.067	78.15	1.064
Dinotefuran	20.12	0.021	52.98	0.965	45.68	0.622	2.81	0.038	48.49	0.660
Region A [c]	5.72	0.006	5.22	0.095	4.98	0.068	0.23	0.003	5.21	0.071
PHP	ND [d]	ND	0.82	0.015	0.59	0.008	0.06	0.001	0.64	0.009
446-DO	ND	ND	2.69	0.049	2.34	0.032	0.09	0.001	2.44	0.033
UF	ND	ND	11.82	0.215	11.57	0.157	1.08	0.015	12.64	0.172
Region E	ND	ND	0.82	0.015	ND	ND	ND	ND	ND	ND

	%TRR and mg/kg in Matrix [a]									
	Other Matrices, 5 DAB				Straw for Surface Washing [b]					
Fraction	Root		Straw		Washed straw		Straw surface wash		Total	
	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg
DN-OH	ND	ND	0.41	0.007	0.37	0.005	ND	ND	0.37	0.005
BCDN	ND	ND	0.41	0.007	0.44	0.006	ND	ND	0.44	0.006
DN	41.82	0.045	4.97	0.091	5.27	0.072	0.60	0.008	5.87	0.080
Others [e]	1.24	0.001	1.39	0.025	1.98	0.027	0.06	0.001	2.04	0.028
Unextracted (PES)	31.10	0.033	18.50	0.337					21.85	0.298
Total	100.00	0.107	100.00	1.822					100.00	1.362
	Other Matrices, 20 DAB				Straw for Surface Washing [b]					
Fraction	Root		Straw		Washed straw		Straw surface wash		Total	
	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg
Extractable	64.95	0.082	81.75	1.101	83.29	1.014	6.13	0.075	89.43	1.089
Dinotefuran	30.85	0.039	51.58	0.695	58.80	0.716	3.71	0.045	62.52	0.761
Region A [c]	12.21	0.015	4.58	0.062	4.16	0.051	0.36	0.004	4.52	0.055
PHP	ND	ND	0.65	0.009	0.58	0.007	0.06	0.001	0.64	0.008
446-DO	ND	ND	2.04	0.028	2.67	0.032	0.13	0.002	2.79	0.034
UF	ND	ND	13.41	0.181	9.66	0.118	1.23	0.015	10.89	0.133
DN-OH	ND	ND	0.57	0.008	ND	ND	ND	ND	ND	ND
BCDN	1.69	0.002	0.65	0.009	ND	ND	ND	ND	ND	ND
DN	19.48	0.024	6.62	0.089	4.83	0.059	0.55	0.007	5.38	0.065
Others [e]	0.71	0.001	1.63	0.022	2.58	0.031	0.10	0.001	2.69	0.033
Unextracted (PES)	35.05	0.044	18.25	0.246					10.57	0.129
Total	100.00	0.126	100.00	1.347					100.00	1.218

[a] Calculations were based on mg/kg level determined as sum of residue levels from extracts and PES.

[b] This measurement was designed to address the degree to which residues were removable by surface-washing, but it was not a measurement of the total straw residues.

[c] Region A consisted of multiple radioactive components eluting from 2to 8 min. This region may contain MNG and conjugates of UF, PHP and 446-DO by analogy to chaff.

[d] ND = Not detected (below limit of detection). Non-detect values are calculated as zero.

[e] Consists of low-level radioactivity; no discernible peaks.

Table 13 Distribution of metabolites in rice grain following spray application of [^{14}C] dinotefuran 5 and 20 days after bolting

	%TRR and mg/kg in Plant Matrix [a]									
	Rice Grain Matrices, 5 DAB									
Fraction	Whole Grain		Brown Rice		Chaff		Polished Rice		Bran	
	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg
Extract	70.24	4.105	89.73	0.548	78.08	26.415	86.78	0.290	83.08	3.354
Dinotefuran	35.89	2.098	33.40	0.204	41.00	13.872	41.74	0.140	27.66	1.117
Region A [b]	5.62	0.328	17.01	0.104	4.16	1.406	8.85	0.030	24.84	1.003
PHP	1.69	0.099	7.05	0.043	2.28	0.770	4.34	0.015	5.65	0.228
446-DO	1.55	0.090	3.48	0.021	2.45	0.829	5.03	0.017	1.00	0.040
UF	17.21	1.006	17.18	0.105	16.17	5.470	22.82	0.076	10.38	0.419
Region E	ND [c]	ND	1.16	0.007	1.11	0.376	ND	ND	ND	ND
DN-OH	0.70	0.041	ND	ND	0.90	0.305	ND	ND	1.99	0.080
BCDN	0.63	0.037	0.83	0.005	0.71	0.241	ND	ND	1.00	0.040
DN	6.46	0.378	6.15	0.038	6.30	2.132	1.65	0.006	9.39	0.379
Others [d]	0.49	0.029	3.48	0.021	3.00	1.014	2.34	0.008	1.16	0.047
Unextracted (PES)	29.76	1.740	10.27	0.063	21.92	7.416	13.22	0.044	16.92	0.683
Total	100.00	5.845	100.00	0.611	100.00	33.831	100.00	0.335	100.00	4.037
	Rice Grain Matrices, 20 DAB									
Fraction	Whole Grain		Brown Rice		Chaff		Polished Rice		Bran	
	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg
Extract	81.36	4.146	92.74	0.313	88.20	16.769	75.32	0.114	86.20	1.491
Dinotefuran	52.72	2.687	53.55	0.181	59.04	11.225	48.43	0.073	51.54	0.892
Region A [b]	3.58	0.182	8.93	0.030	3.37	0.641	6.63	0.010	14.57	0.252
PHP	1.79	0.091	4.08	0.014	1.79	0.340	1.51	0.002	3.28	0.057
446-DO	2.03	0.104	3.31	0.011	2.21	0.419	2.26	0.003	1.47	0.025

	%TRR and mg/kg in Plant Matrix [a]									
	Rice Grain Matrices, 5 DAB									
Fraction	Whole Grain		Brown Rice		Chaff		Polished Rice		Bran	
	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg
UF	13.67	0.697	14.09	0.048	13.35	2.538	14.24	0.021	9.31	0.161
Region E	0.57	0.029	0.13	< 0.0005	0.81	0.154	ND	ND	ND	ND
DN-OH	0.49	0.025	ND	ND	0.56	0.107	ND	ND	0.78	0.013
BCDN	0.57	0.029	ND	ND	0.55	0.104	ND	ND	ND	ND
DN	5.21	0.265	3.40	0.011	5.28	1.004	0.90	0.001	4.91	0.085
Others [d]	0.73	0.037	5.26	0.018	1.25	0.238	1.36	0.002	0.34	0.006
Unextracted (PES)	18.64	0.950	7.26	0.025	11.80	2.243	24.68	0.037	13.80	0.239
Total	100.00	5.096	100.00	0.338	100.00	19.012	100.00	0.151	100.00	1.730

[a] Calculations were based on mg/kg level determined as sum of residue levels from extracts and PES.

[b] Region A consisted of multiple radioactive components eluting from 2 to 8 min. This region may contain MNG and conjugates of UF, PHP and 446-DO by analogy to chaff.

[c] ND = Not detected (below limit of detection). Non-detect values are calculated as zero.

[d] Consists of low-level radioactivity; no discernible peaks.

Table 14 Distribution of metabolites in straw following spray application of [^{14}C] dinotefuran 5 and 20 days after bolting

	%TRR and mg/kg in Matrix [a]							
	5 DAB		Straw for Surface Washing [b]					
Fraction	Straw		Washed straw		Straw surface wash		Total	
	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg
Extract	93.88	7.106	73.61	5.735	15.52	1.209	89.13	6.944
Dinotefuran	53.33	4.037	46.52	3.624	10.17	0.792	56.69	4.416
Region A [c]	2.14	0.162	2.06	0.161	1.20	0.093	3.26	0.254
PHP	2.35	0.178	1.40	0.109	0.56	0.044	1.96	0.152
446-DO	3.31	0.250	2.50	0.195	0.47	0.036	2.97	0.231
UF	15.91	1.204	11.12	0.866	2.37	0.185	13.49	1.051
Region E	0.91	0.069	0.74	0.057	ND [d]	ND	0.74	0.057
DN-OH	1.25	0.094	ND	ND	ND	ND	ND	ND
BCDN	1.71	0.130	0.96	0.075	ND	ND	0.96	0.075
DN	8.52	0.645	5.96	0.465	0.76	0.059	6.72	0.524
Others [e]	4.45	0.336	2.36	0.184	ND	ND	2.36	0.184
Unextracted (PES)	6.12	0.464					10.87	0.847
Total	100.00	7.570					100.00	7.790

	%TRR and mg/kg in Matrix [a]							
	20 DAB		Straw for Surface Washing [b]					
Fraction	Straw		Washed straw		Straw surface wash		Total	
	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg
Extract	95.48	7.777					90.54	10.115
Dinotefuran	68.96	5.617	54.58	6.098	8.90	0.994	63.48	7.092
Region A [c]	1.73	0.141	1.31	0.147	0.68	0.076	1.99	0.223
PHP	4.02	0.327	3.24	0.362	0.55	0.061	3.79	0.432
446-DO	3.73	0.304	2.78	0.310	0.31	0.034	3.09	0.345
UF	8.81	0.718	9.19	1.026	2.21	0.247	11.40	1.274
Region E	0.92	0.075	1.24	0.138	ND	ND	1.24	0.138
DN-OH	ND	ND	ND	ND	ND	ND	ND	ND
BCDN	1.58	0.129	0.85	0.095	ND	ND	0.85	0.095
DN	5.73	0.467	4.01	0.448	0.69	0.078	4.71	0.526
Others [e]	ND	ND	ND	ND	ND	ND	ND	ND
Unextracted (PES)	4.52	0.368					9.46	1.057
Total	100.00	8.146					100.00	11.172

[a] Calculations were based on mg/kg level determined as sum of residue levels from extracts and PES.

[b] This measurement was designed to address the degree to which residues were removable by surface-washing, but it was not a measurement of the total straw residues.

[c] Region A consisted of multiple radioactive components eluting from 2 to 8 min. This region may contain MNG and conjugates of UF, PHP and 446-DO by analogy to chaff.

[d] ND = Not detected (below limit of detection). Non-detect values are calculated as zero.

[e] Consists of low-level radioactivity; no discernible peaks.

Region A was shown to contain MNG and conjugates of UF, PHP and 446-DO by a number of techniques, including HPLC, acid hydrolysis and derivatisation. Levels of unextracted residues from the 5 DAB spray treatment brown rice, chaff and straw were 10.3% TRR (0.063 mg/kg), 21.9% TRR (7.416 mg/kg) and 6.1% TRR (0.464 mg/kg), respectively. Experiments were conducted which showed that the majority of the radioactivity in the PES was the result of reincorporation of radiolabel into natural products (carbohydrate, protein and lignin). A portion of the applied [^{14}C] dinotefuran was completely degraded to $^{14}CO_2$ and reincorporated into these natural products. These experiments included both strong acid hydrolysis (done with brown rice, chaff and straw) and enzyme hydrolysis (done with brown rice only).

Strong acid hydrolysis conditions were chosen for each matrix to hydrolyse the major components in the PES from each matrix. Therefore, 0.95 N HCl under refluxing conditions was used to hydrolyse starch in brown rice, and 72% H_2SO_4 at room temperature followed by 4% H_2SO_4 under refluxing conditions was used to hydrolyse cellulose in chaff and straw. For each matrix, the glucose that was formed from the hydrolysis was converted into a phenylglucosazone derivative that was recrystallized to constant specific activity. For brown rice, the distribution of labelled glucose was calculated at 1.9% TRR (0.011 mg/kg). For chaff and straw, the distribution of labelled glucose was calculated at 1.2% TRR (0.408 mg/kg) and 1.8% (0.133 mg/kg), respectively. The data from the PES characterization with brown rice confirmed that a portion of dinotefuran was completely degraded and reincorporated into glucose subunits in starch in brown rice. The data from the PES characterization with chaff and straw confirmed that a portion of dinotefuran was completely degraded and reincorporated glucose subunits in cellulose in these matrices.

Surfactant and enzyme treatments were used for brown rice PES samples only. For treatment with surfactants, SDS was used to solubilize protein in brown rice samples. Protease enzyme was used to hydrolyse protein in brown rice samples. For brown rice PES from the 5 DAB spray treatment and 20 DAB soil treatment, the protein fraction accounted for 1.5% TRR (0.009 mg/kg) and 2.5% TRR (0.001 mg/kg). The data from the PES characterization with brown rice confirmed that a portion of dinotefuran was completely degraded and reincorporated into amino acids in protein and into lignin in samples from both spray and soil treatments.

Rape seed

The metabolism of dinotefuran in rape seed plants was investigated under natural conditions following foliar application (Völkel, 2002: RCC 767880). The plants were treated at pre-flowering (growth stage 50–59) with formulated [^{14}C]dinotefuran. Doses of 100 g ai/ha (low dose), 200 g ai/ha (high dose) and 1000 g ai/ha (exaggerated dose, plant pot) were applied using a hand held plant sprayer. The highest dose rate corresponded to five times the likely maximum commercial field rate for oilseed rape to provide materials to assist metabolite identification. Planting, cultivation and harvesting of the mature winter rape plants was carried out according to common agricultural practice.

The plants harvested were separated into seeds and foliage. Additionally, the plant roots were worked up. From all plant parts, the radioactive residues increased with the dose rate applied. However, the total radioactive residue in the whole rape plant was very low. Only 4.5%, 5.8% and 3.5% of the amount applied was detected for the 100, 200 and 1000 g ai/ha treatment rates, respectively. These residues corresponded to 0.207 mg parent equivalents per kg plant fresh weight, 0.491 mg/kg and 2.073 mg/kg for the three doses, respectively.

For seeds, the residue levels for the 100, 200 and 1000 g ai/ha doses were determined to be 0.055, 0.127 and 0.696 mg/kg fresh weight, representing 0.14, 0.17 and 0.12% of the amount applied. Accordingly, residue levels for foliage were determined to be 0.259, 0.650 and 2.351 mg/kg fresh weight and for roots 0.097, 0.138 and 1.077 mg/kg fresh weight, respectively. All residue levels reflect the factor used for the application rates, i.e. 1×, 2× and 10× 100 g ai/ha. All plant parts were extracted with organic or aqueous solvent mixtures at ambient temperature. From the foliage most of the radioactive residues (TRR) were extracted representing 94.4–95.9% of the radioactivity in this plant part. Similar values were obtained for roots, amounting to 80.0–88.3% of extracted TRR. The proportion of extracted radioactivity of seeds of the different doses was comparable and amounted to 74.8–81.9% of TRR. In terms of concentration, the extracted residues ranged from 0.042 mg/kg (100 g ai/ha), 0.095 mg/kg (200 g ai/ha) to 0.570 mg/kg (1000 g ai/ha). The remaining unextracted radioactive residues amounted to 18.1–25.2% of TRR.

Table 15 Total radioactive residues in oil seed rape following treatment with [^{14}C]dinotefuran

		Fresh weight, g	Applied, %	Fresh weight parent equivalents, mg/kg [a]
100 g ai/ha	Seeds	247.0	0.14	0.055
	Foliage	1494.5	3.98	0.259
	Roots	359.8	0.36	0.097
	Total	2101.3	4.48	0.207
200 g ai/ha	Seeds	267.7	0.17	0.127
	Foliage	1621.3	5.31	0.650
	Roots	459.4	0.32	0.138
	Total	2348.4	5.80	0.491
1000 g ai/ha	Seeds	45.8	0.12	0.696
	Foliage	363.3	3.25	2.351
	Roots	38.3	0.16	1.077
	Total	447.4	3.53	2.073

[a] mg/kg plant part (seed, foliage or root)

Up to 17 radioactive fractions were detected in the extracts of the seeds, showing similar metabolic patterns for all treatment rates. The parent dinotefuran was found at levels of 0.006 mg/kg (14.8% TRR), 0.016 mg/kg (18.7% TRR) and 0.095 mg/kg (18.0% TRR) in seeds for doses 100 g ai/ha, 200 g ai/ha and 1000 g ai/ha, respectively. Metabolite MNG was found as the most significant fraction, i.e. the fraction exceeding 10% of TRR. It amounted to 0.005 mg/kg, 0.004 mg/kg and 0.071 mg/kg in seeds for the three application rates respectively. None of the other radioactive fractions exceeded 8.2% of TRR. Metabolites UF, PHP, FNG, MG, DN and BCDN were found at low concentrations not exceeding 0.003 mg/kg for the low or high dosed plants with the exception of PHP amounting to 0.006 mg/kg.

In the extracts, very polar unknown fractions (M3.1 to M3.8) were also found. These polar fractions were eluting very close together at short retention times. None of these fractions individually exceeds 0.007 mg/kg in seeds for both low and high dose. The actual concentration will be lower assuming a lower molecular weight of these polar degradation products. Besides the characterised fractions and these very polar metabolites, up to three more unknown radioactive fractions (M5, M6, M10 and M12) were detected in seed extracts, not exceeding 0.003 mg/kg in the seed extracts of the low and the high dose treated plants. Most of the radioactivity in the seeds that remained unextracted by organic solvents could be solubilised by acid and basic hydrolysis, leaving only a maximum of 2.6% TRR (0.001 mg/kg) non-extracted.

Table 16Distribution of dinotefuran and its metabolites in oil seed rape seeds following treatment with [^{14}C]dinotefuran

	100 g ai/ha		200 g ai/ha		1000 g ai/ha	
	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg
Dinotefuran	14.77	0.006	18.68	0.016	18.00	0.095
UF	0.98	< 0.001	2.14	0.001	1.37	0.006
PHP	6.82	0.003	7.03	0.006	4.71	0.025
M3.1	6.05	0.002	5.45	0.005	4.78	0.025
M3.2	5.84	0.002	8.20	0.007	8.73	0.046
M3.3	5.56	0.002	5.86	0.005	6.68	0.035
MNG	12.38	0.005	4.76	0.004	13.37	0.071
M3.5	4.15	0.002	2.76	0.002	ND	ND
M3.6	2.11	0.001	3.45	0.003	ND	ND
M3.7	1.41	0.001	ND	ND	ND	ND
M3.8	0.42	< 0.001	ND	ND	ND	ND
FNG	1.90	0.001	3.79	0.003	0.84	0.004
M5	1.06	< 0.001	ND	ND	1.82	0.010
M6	ND	ND	ND	ND	ND	ND
MG	1.06	< 0.001	< 0.01	ND	2.28	0.004
DN	ND	ND	0.83	0.001	5.70	0.038
BCDN	2.39	0.001	1.10	0.001	0.38	0.002
M10	0.70	< 0.001	1.38	0.001	ND	ND
M11	ND	ND	ND	ND	0.76	0.004
M12	2.74	0.001	3.52	0.003	6.53	0.035
Non-resolved	5.43		5.82		5.93	
Total radioactive fractions	75.77	0.027	74.76	0.059	81.88	0.400

ND: not detected

Non-resolved: hexane extract of seeds

In the extracts of the foliage, up to eight radioactive fractions were detected. The pattern was similar for all treatment rates. The parent dinotefuran was found at levels of 0.025 mg/kg (11.3% TRR), 0.094 mg/kg (16.9% TRR) and 0.222 mg/kg (10.6% TRR) in foliage for doses 100 g ai/ha, 200 g ai/ha and 1000 g ai/ha, respectively.

Radioactive fractions M3 (at least eight compounds), MG and DN were found as significant metabolite fractions. In the extract of the 100 g ai/ha treated plants, the concentrations amounted to 0.120 mg/kg (53.4% TRR), 0.007 mg/kg (8.7% TRR) and 0.037 mg/kg (13.2% TRR) in foliage, respectively. In the extract of the 200 g ai/ha treated plants, the concentrations detected were 0.271 mg/kg (48.8% TRR), 0.010 mg/kg (4.9% TRR) and 0.105 mg/kg (15.0% TRR) in foliage, respectively. The concentrations determined in foliage of the 1000 g ai/ha treated plants amounted to 0.621 mg/kg (29.5% TRR), 0.088 mg/kg (11.5% TRR) and 0.461 mg/kg (17.4% TRR) for the three metabolite fractions M3, MG and DN, respectively. Furthermore detected were UF, MNG (one of the compounds in M3) and BCDN amounting to 0.143 mg/kg (8.7% TRR), 0.137 mg/kg (6.5% TRR) and 0.043 mg/kg (2.68% TRR), respectively. None of the other radioactive fractions exceeded 0.01% of TRR in the normal and high or 0.14% of TRR in the exaggerated dose treated plants.

Table 17 Distribution of dinotefuran and its metabolites in oil seed rape foliage following treatment with [^{14}C]dinotefuran

	100 g ai/ha		200 g ai/ha		1000 g ai/ha	
	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg
Dinotefuran	11.25	0.025	16.94	0.094	10.56	0.222
UF	ND	ND	ND	ND	8.68	0.143
PHP	ND	ND	ND	ND	ND	ND
M3.1–3.8	53.38	0.120	48.76	0.271	29.53	0.621
FNG	ND	ND	ND	ND	ND	ND
M5	ND	ND	< 0.01	ND	2.60	0.055
M6	ND	ND	ND	ND	ND	ND
MG	8.74	0.007	4.88	0.010	11.54	0.088
DN	13.15	0.037	14.97	0.105	17.36	0.461
BCDN	ND	ND	ND	ND	2.68	0.043
M10	ND	ND	ND	ND	ND	ND
M11	ND	ND	ND	ND	ND	ND
M12	ND	ND	ND	ND	ND	ND
Non-resolved	8.77		8.85		6.46	
Total radioactive fractions	95.28	0.189	94.39	0.480	95.95	1.770

ND: not detected

Non-resolved: Soxhlet extract of foliage

Up to three radioactive fractions detected in the pooled extracts of the rape roots. The parent dinotefuran was found at levels of 0.008 mg/kg (10.8% TRR), 0.021 mg/kg (18.4% TRR) and 0.100 mg/kg (13.1% TRR) roots for the treatment rates 100 g ai/ha (low dose), 200 g ai/ha (high dose) and 1000 g ai/ha (exaggerated dose), respectively. Radioactive fraction M3 was the only other fraction in the extracts of the low and high dose treated plants, amounting to 0.054 mg/kg (69.5% TRR) and 0.075 mg/kg (64.9% TRR), respectively. However, this very polar fraction consists of at least six components. In extracts of the exaggerated dosed plants, the concentration for M3 was determined to be 0.387 mg/kg (50.9% TRR) in roots. Additionally DN was detected and amounted to 0.065 mg/kg (6.7% TRR).

Although very low levels of radioactivity were present in the seed of oil seed rape plants treated at up to 1000 g/ha with [^{14}C]dinotefuran it was possible to confirm that dinotefuran was the major residue and that trace levels of the metabolites UF, PHP, FNG, MG, DN, and BCDN were also present.

Summary of plant metabolism

Metabolism of [^{14}C]dinotefuran labelled in the tetrahydrofuran and guanidine has been studied in apples, lettuce, potato, rice and oilseed rape, which are suitable to cover the crop groups pome fruits, leafy vegetables, root/tuber vegetables, cereals and pulses/oilseeds. Dinotefuran is the major component of the residues found in apples, lettuce, potato, rice (grain and straw) and oilseed rape (seed). UF, DN and MNG are also the major components of the residues in those plants. The following metabolic pathways were speculated in the plant metabolism studies available.

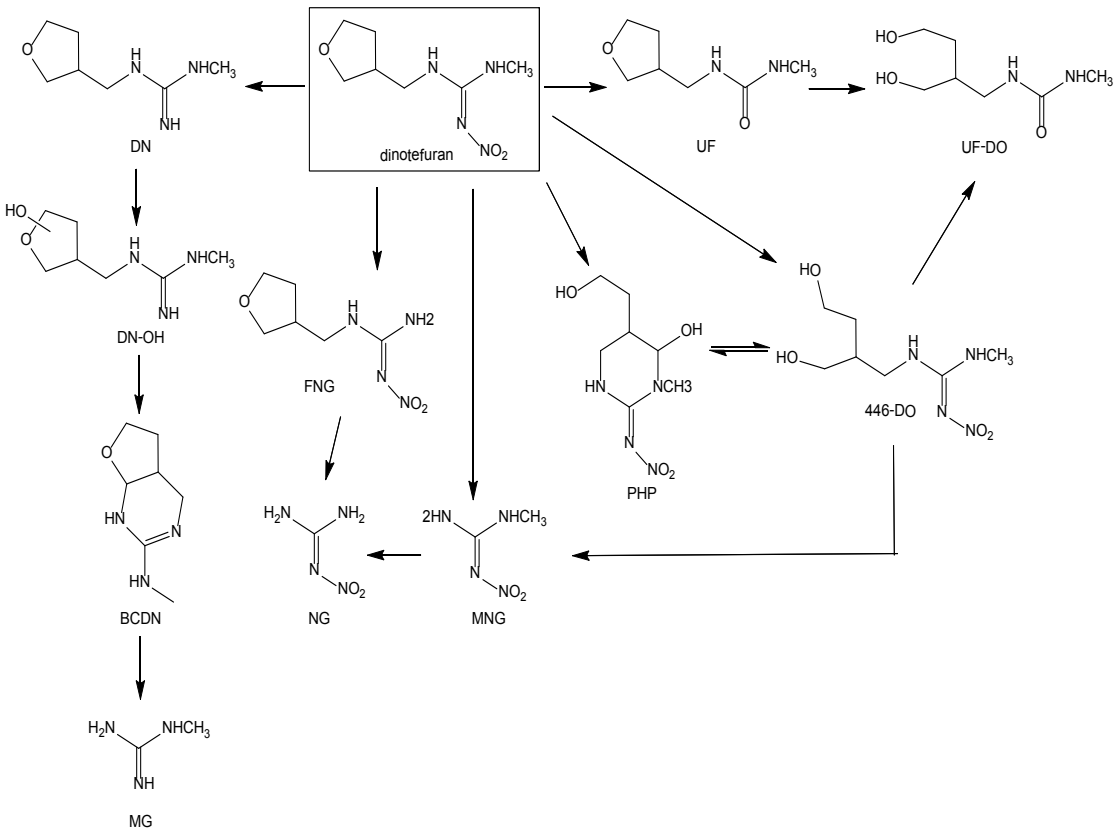


Figure 4 Proposed Metabolic Pathway of Dinotefuran in Plants

Environmental fate in soil

The Meeting received information on aerobic and anaerobic degradation in soil, photolysis on soil surface, rotational crop, degradation in water sediment and hydrolytic degradation study. Because dinotefuran is intend for use as foliar and soil treatment, aerobic degradation, soil photolysis, rotational crop and hydrolytic degradation study relevant to the current evaluations were reported below (FAO Manual 2009).

The fate and behaviour of dinotefuran in soils were investigated using [^{14}C]furanyl and [^{14}C]guanidine labelled compounds.

Aerobic degradation

Study 1

The metabolism and degradation rate (DT_{50} and DT_{90}) of dinotefuran was investigated in a silt loam soil incubated under aerobic conditions at 20 °C and 10 °C for a period of 120 days (Völkl, 2003: RCC 843175). A mixture of tetrafuryl ring and guanidine labelled [^{14}C]dinotefuran was applied to the soil samples at a concentration of about 0.3 mg/kg dry soil. This rate corresponds to about 0.3 kg ai/ha, assuming an even distribution of the test item in the top 10 cm of soil and a soil bulk density of 1 g/cm^3.

Table 18 Soil characteristics

Parameters	Soil (Gartenacker, Switzerland)
pH ($CaCl_2$)	7.2
Organic carbon (g/100 g soil)	1.8
Cation exchange capacity (meq/100 g soil)	13.1

Parameters	Soil (Gartenacker, Switzerland)
Soil type (USDA)	Silt loam
Clay (%)	10.3
Silt (%)	52.3
Sand (%)	37.4
Maximum water holding capacity (MWC; g/100 g soil)	67.5
40% MWC (g/100 g soil)	27.0
Biomass (mg microbial C/100 g dry soil)	
a) Start of incubation	30.3
b) Day 120 of incubation (20 °C)	27.4
c) Day 120 of incubation (10 °C)	32.5

Dinotefuran was detected until the end of incubation (day 120). It represented 91.2% of the applied radioactivity immediately after treatment and decreased further to 12.5% and 36.1% on day 28 of incubation at 20 °C and 10 °C, respectively. At the end of incubation (day 120), dinotefuran amounted to 0.6% and 3.7% of the applied radioactivity at 20 °C and 10 °C, respectively. Besides the test item, up to nine radioactive fractions were detected in the soil extracts. MNG and NG represented major degradation products. MNG amounted to a maximum of 15.6% (20 °C) of the applied radioactivity after 28 days of incubation, and 16.0% (10 °C) of the applied radioactivity after 62 days of incubation. Thereafter, it degraded continuously amounting to 7.3% and 14.9% on day 120 of incubation at 20 °C and 10 °C, respectively. NG represented a maximum of 5.4% of the applied radioactivity. Other radioactive fractions characterized as UF, FNG and six unknown degradation products were detected at trace amounts (< 2.4% of the applied radioactivity).

Table 19 Distribution of dinotefuran and metabolites in soil

		Incubation time (days)								
		0 [a]	3	7	10	14	21	28 [a]	62	120
20 °C										
Dinotefuran	%AR	91.2	72.9	57.4	47.2	34.4	21.1	12.5	2.4	0.6
	mg/kg	0.274	0.219	0.172	0.142	0.103	0.063	0.038	0.007	0.002
MNG	%AR	0.6	3.5	7.4	8.9	11.5	13.6	15.6	12.8	7.3
	mg/kg	0.002	0.010	0.022	0.027	0.035	0.041	0.047	0.038	0.022
NG	%AR	ND	0.2	1.0	1.2	2.0	3.2	3.6	5.2	4.3
	mg/kg	ND	0.001	0.003	0.004	0.006	0.010	0.011	0.016	0.013
UF	%AR	1.2	1.3	ND	0.9	0.7	0.5	ND	ND	0.1
	mg/kg	0.004	0.004	ND	0.003	0.002	0.001	ND	ND	< 0.001
FNG	%AR	1.0	0.9	0.8	0.7	0.7	0.6	0.5	0.2	0.1
	mg/kg	0.003	0.003	0.002	0.002	0.002	0.002	0.002	< 0.001	< 0.001
M7	%AR	ND	1.9	ND	0.2	0.3	0.2	0.1	ND	ND
	mg/kg	ND	0.006	ND	0.001	0.001	< 0.001	< 0.001	ND	ND
M8	%AR	ND	0.6	0.3	0.3	0.2	0.2	0.2	ND	< 0.1
	mg/kg	ND	0.002	0.001	0.001	0.001	< 0.001	< 0.001	ND	< 0.001
M9	%AR	ND	0.1	0.3	0.4	0.4	< 0.1	0.3	ND	ND
	mg/kg	ND	< 0.001	0.001	0.001	0.001	ND	0.001	ND	ND
M10	%AR	ND	0.7	1.4	1.0	1.0	0.9	0.9	0.7	0.5
	mg/kg	ND	0.002	0.004	0.003	0.003	0.003	0.003	0.002	0.001
M11	%AR	ND	0.6	0.8	0.7	0.7	0.9	0.7	0.6	0.3
	mg/kg	ND	0.002	0.002	0.002	0.002	0.003	0.002	0.002	0.001
Unextracted	%AR	1.9	5.9	8.9	11.9	13.6	16.1	17.8	20.9	25.7
	mg/kg	0.006	0.009	0.013	0.036	0.041	0.048	0.053	0.063	0.077
CO_2	%AR	–	6.5	15.9	21.4	27.6	35.1	39.7	49.3	52.1
	mg/kg	–	–	–	0.064	0.083	0.105	0.119	0.148	0.156
Other volatiles	%AR	–	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
	mg/kg	–	–	–	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
Total	%AR	95.9	95.0	94.3	95.0	93.1	92.2	91.7	92.1	91.0
	mg/kg	0.288	0.285	0.283	0.285	0.279	0.277	0.276	0.276	0.273

		Incubation time (days)								
		0 [a]	3	7	10	14	21	28 [a]	62	120
10 °C										
Dinotefuran	%AR	91.2	84.4	73.6	64.0	56.5	46.2	36.1	13.1	3.7
	mg/kg	0.274	0.253	0.221	0.192	0.169	0.139	0.108	0.039	0.011
MNG	%AR	0.6	1.6	4.1	5.6	7.5	9.5	12.4	16.0	14.9
	mg/kg	0.002	0.005	0.012	0.017	0.022	0.029	0.037	0.048	0.045
NG	%AR	ND	ND	0.3	0.5	0.6	1.5	1.6	3.8	5.4
	mg/kg	ND	ND	0.001	0.001	0.002	0.005	0.005	0.011	0.016
UF	%AR	1.2	ND	ND	0.8	1.1	0.9	ND	ND	0.1
	mg/kg	0.004	ND	ND	0.002	0.003	0.003	ND	ND	< 0.001
FNG	%AR	1.0	0.7	0.9	0.8	0.8	0.8	0.9	0.7	0.3
	mg/kg	0.003	0.002	0.003	0.002	0.002	0.002	0.003	0.002	0.001
M7	%AR	ND	ND	ND	0.3	0.4	0.2	0.3	ND	ND
	mg/kg	ND	ND	ND	0.001	0.001	0.001	0.001	ND	ND
M8	%AR	ND	0.8	0.7	0.8	0.4	0.4	0.4	0.2	0.1
	mg/kg	ND	0.002	0.002	0.002	0.001	0.001	0.001	0.001	< 0.001
M9	%AR	ND	ND	0.2	0.2	ND	ND	0.4	ND	ND
	mg/kg	ND	ND	< 0.001	0.001	ND	ND	0.001	ND	ND
M10	%AR	ND	1.0	0.8	1.3	0.4	1.1	1.1	0.9	0.8
	mg/kg	ND	0.003	0.002	0.004	0.007	0.003	0.004	0.003	0.002
M11	%AR	ND	0.7	1.3	1.3	0.4	1.1	1.1	0.9	0.8
	mg/kg	ND	0.002	0.004	0.004	0.001	0.003	0.003	0.003	0.002
Unextracted	%AR	1.9	3.9	6.0	7.4	8.3	10.1	12.2	16.6	19.9
	mg/kg	0.006	0.012	0.018	0.022	0.025	0.030	0.036	0.050	0.060
CO_2	%AR	–	3.0	7.3	11.2	12.0	21.8	26.7	37.5	43.7
	mg/kg	–	0.009	0.022	0.033	0.036	0.065	0.080	0.113	0.131
Other volatiles	%AR	–	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
	mg/kg	–	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
Total	%AR	95.9	96.1	95.2	94.1	90.3	93.6	93.4	89.8	89.7
	mg/kg	0.288	0.288	0.285	0.282	0.271	0.281	0.280	0.269	0.269

Values are given in % of applied radioactivity (%AR) and mg parent equivalents/kg dry soil (mg/kg).

[a] mean value of duplicate samples

ND: not detected

–: not performed

The calculated DT_{50} and DT_{90} values for dinotefuran in the soil, based on first-order kinetics, are shown in Table 20.

Table 20 Calculated DT_{50} and DT_{90} values for dinotefuran in aerobic soil

	20 ± 2 °C	10 ± 2 °C
DT_{50} (days)	10.2	21.1
DT_{90} (days)	33.9	70.0

Mineralisation of dinotefuran to carbon dioxide, microbial transformation to MNG and NG as well as incorporation of residues into the soil organic matter can be regarded as the major pathways of elimination of dinotefuran.

Study 2

The degradation rate of [^{14}C]dinotefuran was studied with the mixture of two radiolabelled materials ([^{14}C]furanyl-dinotefuran and [^{14}C]guanidine-dinotefuran) at a concentration of approximately 0.6 mg/kg in one soil under aerobic conditions at 25 ± 1 °C (Lentz, 2001: 013184-1). The bulk soil was sieved through a 2.0 mm sieve to remove gross debris. The soil was classified as loamy sand with soil organic matter of 1.1% and the pH was 6.9.

Table 21 Soil characteristics

Parameters	Soil (Gartenacker, Switzerland)
pH	6.9
Organic matter (%)	1.1
Cation exchange capacity (meq/100 g soil)	6.9
Soil type (USDA) Clay (%) Silt (%) Sand (%)	Loamy sand 6.0 12.0 82.0
Water holding capacity (%) Saturation 1/3 bar 15 bar	 29.6 7.5 3.8

The test soil was treated with [^{14}C]dinotefuran at a rate of approximately 0.6 mg/kg, which is equivalent to the proposed maximum use rate (at a seasonal application rate of 600 g ai/ha). Treated soil samples were incubated at 75% of 1/3 bar water-holding capacity at 25 °C in the dark. The soil samples were extracted and analysed by HPLC ay Days 0, 7, 14, 21, 30, 45, 62, 90, 120, 181 and 225. The volatiles traps were analysed at every sampling time starting with the day 7 time point. Material balance over the course of the study was maintained ranging from 90.7 to 103.3%. Extracted radiolabel decreased from an average of 99.4% at Day 0 to an average of 18.5% of the applied dose by the termination of the study at 225 days. Unextracted residues in the soil increased to a maximum average of 34.6% at day 225. Evolution of $^{14}CO_2$ was 41.8% of the applied dose at day 225.

Table 22 Balance of radioactivity in aerobic soil treated with [^{14}C]dinotefuran (25 °C incubation)

Sample	% Extract (average)	% in PES [a] (average)	% in 1.0 N NaOH traps	% VOC [b]	% Total (average)
Day 0	99.4	0.4	NA [c]	NA	99.8
Day 7	86.9	10.5	2.2	ND [d]	99.6
Day 14	81.6	15.4	2.8	< 0.1	99.8
Day 21	74.5	15.8	5.5	< 0.1	95.8
Day 30	72.8	19.5	9.1	< 0.1	101.3
Day 45	58.7	23.8	15.6	< 0.1	98.0
Day 62	50.3	23.2	20.2	< 0.1	93.7
Day 90	38.9	27.1	27.7	0.1	93.8
Day 120	32.8	29.7	33.4	0.1	96.0
Day 181	23.9	27.9	39.5	0.1	91.4
Day 225	18.5	34.6	41.8	0.1	94.9

All values are expressed as % of applied dose (%AD).

[a] PES = Post-extraction solids

[b] VOC = Volatile organic compounds trapped in the ethylene glycol trapping solution

[c] NA = Not analysed

[d] ND = Not detected

Dinotefuran declined from an average of 98.5% at Day 0 to 49.1% by Day 45 and was present at < 10% of the applied radiocarbon by study termination at Day 225. The major degradates formed were MNG and NG increasing to maximum average values of 8.7 and 3.6% of the applied radiocarbon, respectively, by Day 181. Both MNG and NG declined slightly to 6.6 and 3.4%, respectively, of the applied radiocarbon at Day 225. Several minor components were observed at retention times between 10–18 minutes in the HPLC chromatograms at Day 2–225. All of the individual minor components were less than 3% (average value) of the administered dose at every time point analysed.

Table 23 Distribution of radioactivity in aerobic soil treated with [^{14}C]dinotefuran (25 °C incubation)

Sample	Dinotefuran	MNG	NG	Others [a]
Day 0	98.5	ND [b]	ND	ND
Day 7	82.1	1.1	ND	3.6

Sample	Dinotefuran	MNG	NG	Others [a]
Day 14	75.8	1.4	ND	4.5
Day 21	66.0	4.1	ND	4.4
Day 30	64.3	2.7	0.2	5.6
Day 45	49.1	6.2	1.0	2.3
Day 62	38.5	7.9	1.7	2.3
Day 90	28.1	7.3	2.9	0.7
Day 120	21.1	8.3	2.7	0.7
Day 181	11.2	8.7	3.6	0.4
Day 225	8.3	6.6	3.4	0.2

All values are expressed as % of applied radiocarbon (%AR).

[a] All single area of radiocarbon < 4.6% of the AR at every sampling point.

[b] ND = Not detected

The DT_{50} and DT_{90} values (the time points as which 50% and 90%, respectively, of the initially applied parent material had degraded) were calculated for dinotefuran in soil. First-order kinetics was observed. The DT_{50} value was calculated to be 51.7 days and the DT_{90} value was 171.8 days for dinotefuran in aerobic soil.

Dinotefuran degraded in soil under the aerobic conditions employed, principally via cleavage of tetrahydromethyl portion of the molecule to MNG and demethylation of MNG to NG. No other significant degradate greater than 6% of the applied radiocarbon was detected using the HPLC method employed. Dinotefuran and degradates were metabolised and mineralized to CO_2 and immobilized as soil-bound residue.

Soil photolysis

The photolysis of dinotefuran was studied with the mixture of two radiolabelled materials ([^{14}C]furanyl-dinotefuran and [^{14}C]guanidine-dinotefuran) at a concentration of ca. 0.6 mg/kg on a loamy sand soil which was exposed to simulated sunlight with a 12 hour light/12 hour dark cycle for approximately 32 days at a temperature of 20 ± 1 °C (Hatzenbeler, 2001: 012788-1). The soil was obtained from the same location as the soil used for the aerobic soil metabolism study (Lentz, 2001: 013184-1).

Five grams of soil was packed into each photolysis vessel with a uniform surface approximately 1 mm thick. A total of 16 dark control and 16 light exposed samples were prepared. Three additional sample vessels each containing 15 g of dried soil were prepared for use in the mass balance portion of the study (1 dark control and 2 irradiated samples). The mass balance samples were incorporated into a flow-through system which consisted of a pump to draw humidified air over the samples at a rate of approximately 5–10 mL/minute through CO_2 (1 N NaOH) and volatile organics traps. The fortification solution was added to each sample jar onto the soil surface at a rate of approximately 0.56 mg/kg. The test samples were covered with quartz disks sealed to the lids of the dishes. They were then placed in the water bath at 20 ± 1 °C under the xenon arc lamp with only minor deviations outside of this temperature range.

In the dark control sample extracts, dinotefuran decreased from 98.7% (Day 0) to approximately 54.7% of the applied radioactivity (AR) by Day 32. The major degradate MNG was detected in Day 7 samples at 2.0% AR, and increased to 9.4% AR by Day 32. Minor metabolites were detected at levels of 1% or less. In the irradiated sample extracts, dinotefuran decreased from 98.7% (Day 0) to 60.2% of AR by Day 32. MNG did not appear in the irradiated samples. Minor metabolites were detected at levels of 3.4% or less.

The mean mass balance recoveries ranged from 98.2% for the dark control samples to 103.1% for the light-exposed samples. The percentage of radioactivity in the extractable fraction decreased from 99.1% (Day 0) to 66.3% (Day 32) for the dark controls and from 99.1% (Day 0) to 70.7% (Day 32) in the light-exposed samples. The levels of bound residue increased throughout the course of the study. It accounted for 16.3% (Day 32) in the dark control soil and for 23.3% (Day 32) in the irradiated soil samples. The cumulative amount of $^{14}CO_2$ found in the base trap was 9.4% AR for the

dark controls and 6.5% AR for the light-exposed samples. No radioactivity above the limit of detection was seen in the volatile organic traps.

The calculated half-life for dinotefuran under test conditions was 37 days for the dark control samples and 46 days for samples under illumination.

Table 24 Mass balance in samples throughout the study

Time	% Extract	% Bound	% Volatiles (CO_2)	% Recovery [a]
Dark control				
Day 0	99.1	0.9	NA [b]	100.0
Day 7	91.2	8.2	2.7	102.1
Day 14	83.5	11.3	4.5	99.3
Day 21	77.3	13.4	6.8	97.5
Day 32	66.3	16.3	9.4	92.0
Light exposed				
Day 0	99.1	0.9	NA	100.0
Day 7	93.6	9.7	2.5	105.9
Day 14	85.2	15.0	3.6	103.7
Day 21	82.7	17.5	5.0	105.2
Day 32	70.7	23.3	6.5	100.5

[a] Recovery = Extractable + Bound + Volatiles

[b] NA = Not analysed

Table 25 Distribution of radioactivity in samples throughout the study

Time	%TRR [a]				
	Dinotefuran	MNG	NG	DN	Others
Dark control					
Day 0	98.7	ND [b]	ND	ND	0.4
Day 7	85.7	2.0	ND	ND	3.5
Day 14	75.1	4.4	ND	ND	4.2
Day 21	65.1	9.0	1.0	ND	2.4
Day 32	54.7	9.4	1.0	ND	1.3
Light exposed					
Day 0	98.7	ND	ND	ND	0.4
Day 7	88.8	ND	ND	1.8	3.2
Day 14	76.4	ND	ND	2.5	6.5
Day 21	74.3	ND	ND	2.2	5.4
Day 32	60.2	ND	ND	3.4	7.5

[a] %TRR = % ^{14}C in designated fractions × % extractable

b ND = Not detected

The test compound, dinotefuran degraded in both the dark control and the light exposed samples. The major degradate in the dark control test system was MNG. MNG did not appear in the light-exposed samples. The minor metabolites in the dark control samples and in the light illuminated samples were detected at $\leq 3\%$ AR. Photolysis is not a significant degradation pathway for dinotefuran in soil incubated at 20 ± 1 °C, relative to the dark controls.

Residues in rotational crops

Confined rotational crop studies

Radiolabelled dinotefuran was applied to soil in California and rotational crops were planted at 30 and 120 days after treatment. The treated soil was aged for 30 days prior to planting (simulating a crop failure), and for 120 days (simulating a second crop planting in the same year). The test crops for the study were radish, lettuce, sorghum and wheat, which represent a root crop, leafy vegetable and small grain crop that could be exposed to the test material and consumed by humans and/or livestock (Hattermann, 2002: 41429C008). Soil samples collected from the plots prior to the application

showed that there was no radioactive residue present that would interfere with the analysis. The application formulation was mixed at the laboratory containing equal amounts of furanyl and guanidine labelled dinotefuran as well as unlabelled dinotefuran and blank formulation. This mixture was shipped to the field site in a dehydrated state. It was later mixed with water and was applied to the soil in the plots. The test substance was applied as one broadcast spray application at a rate of 600 g ai/ha in a volume of 1062 L/ha. At 30 days after the application, radish, lettuce and sorghum crops were planted. There were harvested at maturity and sent to the laboratory for analysis. At 120 days after the application, this procedure was repeated except that wheat was planted instead of sorghum.

Approximately one third to one half of the plants of each type in each plot were sampled at the immature growth stage (forage) and the remainder sampled at maturity. More than 12 plants were collected from random areas within each plot. Plant tissues were analysed first by combustion followed by scintillation counting. Then solvent extraction was carried out on selected matrices and the extracts analysed by HPLC and TLC. The amounts of dinotefuran and metabolites in the extracts are based on the chromatographic profiling by HPLC. One dimensional TLC served as a secondary analytical method.

The total radioactive residue (TRR) found in each of the plant fractions amounted to as much as 1.334 mg/kg in 30 day immature radish leaves to as little as 0.003 mg/kg in 120 day lettuce (mature and immature). The TRR was less than 0.01 mg/kg in all of the 120 day plant sample fractions except for the radish leaf samples where it was 0.035 mg/kg in the immature samples and 0.026 mg/kg in the mature samples. Given that the application rate was exaggerated at least two-fold over the expected rate for food crops. More than half and in some cases as much as 95% of the residue was extracted. There was much more radioactive residue detected in 30 day samples than was seen in 120 day samples (TRR as well as extractable residue). In the 30 day radish samples, the parent molecule was extracted from immature as well as mature leaf and root samples. It comprised the highest concentration of a single molecule from the immature leaf and root samples but not from the respective mature samples. The results showed several compounds present at above 0.01 mg/kg at the 30 day time interval, including the parent molecule, however, there were no individual components seen at concentrations above 0.006 mg/kg in any matrix in the 120 day samples including radish leaves.

Table 26 Total radioactive residues (TRRs) found in crops for confined rotational crop study

		30 days after the application				120 days after the application			
		Days between application and harvest	TRR (mg/kg)	%TRR		Days between application and harvest	TRR (mg/kg)	%TRR	
				Extractable	Non-Extractable			Extractable	Non-Extractable
Radish (immature)	Root	63	0.046	95.0	5.0	168	0.008	91.2	8.8
	Leaf	63	1.334	95.3	4.7	168	0.035	90.4	9.6
Radish (mature)	Root	98	0.024	63.5	36.5	196	0.005	94.9	5.1
	Leaf	98	0.354	91.8	8.2	196	0.026	91.6	8.4
Lettuce (immature)	Leaf	71	0.237	66.0	34.0	182	0.003	61.3	38.7
Lettuce (mature)	Leaf	107	0.423	86.9	13.1	204	0.003	73.8	26.2
Sorghum/Wheat (immature)	Forage	75	1.245	75.5	24.5	202	0.007	52.7	47.3
Sorghum (mature)	Forage	196	0.326	45.7	54.3	-	na	na	na
	Chaff	196	0.248	50.2	49.8				
	Grain	196	0.042	81.7	18.3				

na: not analysed due to the low residue in the immature forage

In the mature radish samples (leaf and root), DN was present at the highest concentration (0.074 and 0.007 mg/kg, respectively). In the lettuce samples, BCDN and UF metabolites were found at the highest concentration in immature samples (0.027 mg/kg for both) while PHP was found to be

present at the highest concentration in mature lettuce (0.083 mg/kg) followed by parent, BCDN and DN (0.064, 0.061 and 0.033 mg/kg, respectively). For the 30-day immature sorghum, the highest residue was found to be comprised of parent followed by BCDN, DN, PHP, 446-DO, UF and MNG (0.195, 0.191, 0.143, 0.126, 0.080, 0.069 and 0.017 mg/kg, respectively). The mature sorghum samples analyses indicated extractable residues of 0.149, 0.124 and 0.034 mg/kg in forage, chaff and grain. The extracted residues consisted of BCDN and DN in forage and chaff as well as an unidentified component known as M16. MNG was also present in chaff.

In the 120 day planted crops, there were no individual molecules found at concentrations higher than 0.006 mg/kg. This indicates that neither dinotefuran nor its metabolites should accumulate at concentrations greater than 0.01 mg/kg in crops planted 120 days after treatment. The only molecules found at concentrations above LOQ (0.001 mg/kg) in the immature radish root were parent, MNG and DN all at 0.001 mg/kg. In the immature radish leaf, parent as well as the metabolites MNG, PHP, BCDN, DN and UF were detected at concentrations just above LOQ. In mature radish samples, there was only 0.005 mg/kg residue in the root so the matrix was not fractionated due to the low radioactivity. In immature lettuce leaf samples, there were no extracted residues of parent or any metabolites detected above LOQ. Mature lettuce leaf samples were not fractionated since there was only 0.002 mg/kg radioactive residue detected. Immature wheat forage showed no residues of any of the molecules detected in other matrices. Extracted residue in this matrix was only 0.004 mg/kg. In the lettuce (immature as well as mature) and the immature wheat forage, the extracted residue was very low and the unextracted residue is one half to 75% of the extracted residue (i.e. 0.001 and 0.003 mg/kg).

Table 27 Residues of dinotefuran and metabolites in rotational crop matrices

	Radish				Lettuce		Sorghum/ Wheat			
	Immature		Mature		Immature	Mature	Immature	Mature		
	Root	Leaf	Root	Leaf	Leaf	Leaf	Forage	Forage	Chaff	Grain
30 days after the application										
Dinotefuran	42.1 (0.019)	24.5 (0.327)	9.3 (0.002)	7.3 (0.026)	7.1 (0.017)	15.1 (0.064)	15.7 (0.195)	ND	ND	ND
NG	ND [a]	7.8 (0.054)	ND	ND	ND	0.8 (0.002)	ND	ND	ND	ND
MNG	13.4 (0.004)	12.3 (0.095)	2.9 (< 0.001)	3.2 (0.007)	0.5 (0.001)	7.1 (0.017)	2.3 (0.017)	ND	1.1 (0.002)	ND
M3	ND	ND	ND	1.9 (0.007)	ND	ND	ND	ND	ND	ND
M4	6.1 (0.003)	ND	ND	ND	ND	ND	ND	ND	ND	ND
PHP	5.0 (0.002)	8.6 (0.122)	ND	2.8 (0.011)	5.5 (0.014)	18.5 (0.083)	9.5 (0.126)	ND	ND	ND
M6	ND	ND	ND	ND	1.3 (0.003)	ND	ND	ND	ND	ND
M7	ND	ND	ND	ND	1.6 (0.004)	ND	ND	ND	ND	ND
M8	ND	ND	1.6 (< 0.001)	4.9 (0.017)	2.3 (0.006)	ND	ND	ND	ND	ND
446-DO	ND	ND	ND	3.8 (0.015)	1.0 (0.003)	4.1 (0.019)	5.9 (0.080)	ND	ND	ND
UF	ND	7.0 (0.073)	ND	4.7 (0.013)	14.7 (0.027)	4.2 (0.014)	7.1 (0.069)	ND	ND	ND
M12	1.6 (0.001)	ND	ND	0.8 (0.003)	ND	ND	ND	ND	ND	ND
BCDN	5.7 (0.002)	13.7 (0.141)	ND	23.4 (0.064)	14.8 (0.027)	18.8 (0.061)	19.9 (0.191)	8.1 (0.020)	1.2 (0.002)	ND
DN	5.3 (0.002)	16.2 (0.168)	39.6 (0.007)	26.7 (0.074)	9.6 (0.018)	10.1 (0.033)	14.8 (0.143)	6.6 (0.017)	0.3 (0.001)	ND
M15	ND	ND	1.5 (< 0.001)	0.5 (0.002)	ND	ND	ND	ND	ND	ND
M16 [b]	ND	ND	ND	ND	ND	ND	ND	31.0 (0.101)	47.5 (0.118)	72.9 (0.031)
Non-	15.9	5.2	8.5	11.7	7.7	8.3	0.4	< 0.1	< 0.1	8.8

	Radish				Lettuce		Sorghum/ Wheat			
	Immature		Mature		Immature	Mature	Immature	Mature		
	Root	Leaf	Root	Leaf	Leaf	Leaf	Forage	Forage	Chaff	Grain
Resolved [c]	(0.007)	(0.070)	(0.002)	(0.042)	(0.018)	(0.035)	(0.005)	(< 0.001)	(< 0.001)	0.004
120 days after the application										
Dinotefuran	15.0 (0.001)	4.5 (0.002)	na	2.3 (0.001)	1.7 (< 0.001)	na	ND	na	na	na
NG	ND	ND		ND	ND		ND			
MNG	16.9 (0.001)	5.0 (0.001)		18.5 (0.003)	5.0 (< 0.001)		ND			
M3	ND	ND		ND	ND		ND			
M4	ND	ND		ND	ND		ND			
PHP	ND	8.0 (0.003)		ND	1.9 (< 0.001)		ND			
M6	ND	ND		ND	ND		ND			
M7	ND	ND		ND	ND		ND			
M8	4.9 (< 0.001)	ND		ND	ND		ND			
446-DO	2.0 (< 0.001)	0.5 (< 0.001)		8.0 (0.002)	ND		ND			
UF	ND	7.0 (0.002)		21.3 (0.004)	ND		ND			
M12	ND	ND		ND	ND		ND			
BCDN	5.7 (< 0.001)	8.5 (0.002)		10.0 (0.002)	11.8 (< 0.001)		ND			
DN	17.8 (0.001)	11.4 (0.003)		30.2 (0.006)	20.9 (< 0.001)		ND			
M15	ND	ND		1.3 (< 0.001)	0.8 (< 0.001)		ND			
Non-Resolved [c]	29.1 (0.002)	45.4 (0.016)		< 0.1 (< 0.001)	19.2 (< 0.001)		52.7 (0.004)			

The unknown radioactive fractions are given in mg parent equivalents/kg plant tissue. However, the known metabolites are given in mg metabolite/kg plant tissue.

[a] ND: not detected

[b] M16: Fraction remaining in the HPLC column or at the origin of TLC plate corresponding to radioactivity conjugated to the matrix.

[c] Non-resolved radioactivity corresponds to the extracted radioactivity which remained conjugated to the matrix.

[d] Not analysed due to the very low amount of radioactive residue

The radiochemical purity of the test substance mixture that was applied to the plots (the remainder of which was returned to the laboratory after the application) was determined to be 98%. Therefore, the stability of the test substance prior to application was confirmed. There was no residue found above background in the soil samples collected from the plots prior to the application or in the irrigation water.

An uptake of dinotefuran residues in rotational crop was detected. The total radioactive residue taken up decreased with increasing ageing time. Radioactive residue levels above 0.010 mg parent equivalent/kg plant part were detected in the plants originating from the 30-day field plot. However, the total radioactive residue was below 0.010 mg/kg for all plant parts originating from the plant grown in the 120-day field plot, except for the radish leaf which had residues of 0.035 mg/kg and 0.026 mg/kg for immature and mature leaf samples, respectively.

Environmental fate in water systems

Hydrolysis

The hydrolytic stability of dinotefuran as a function of pH was investigated between pH 4–13 (Sydney, 1998: 95/1216). Portions of dinotefuran (approximately 200 mg) were added to aliquots (100 mL) of each buffer solution in separate bottles to produce test solutions of nominal concentration 2 g/L. The test solutions were adjusted to within ± 0.05 pH units of the pH value, and then nitrogen

purged placed in a thermostatically controlled incubator at 50 °C in the dark until sampling (after a short incubation period of about 15 minutes and then at various other times up to 170 hours). At each sampling time, duplicate aliquots (1 mL) of the test solutions were diluted to volume (20 mL) with HPLC mobile phase for analysis by HPLC. Incubator temperature and the pH values of the test solutions were monitored throughout the course of the test. There were no significant changes in the pH value of 4, 7 and 9 test solutions with time. At pH 11 and 13 however, pH was observed to decrease with time.

Table 28 Hydrolytic degradation of dinotefuran in solutions buffered at pH4, 7, 9, 11 and 13 at 50 °C (the concentration of dinotefuran; mg/kg)

pH	Incubation period (hours)							
	0	2.4	24	95	96	118	120	170
4	1.85, 1.85	1.85, 1.85	1.87, 1.86	1.82, 1.83		1.85, 1.83		1.83, 1.83
7	1.91, 1.90	1.90, 1.88	1.92, 1.90			1.88, 1.87		1.85, 1.82
9	2.05, 2.04	2.08, 2.06	2.02, 1.99			1.94, 1.91		1.84, 1.84
11	2.13, 2.06	1.97, 1.97	1.34, 1.33		0.46, 0.46		0.32, 0.32	
13	2.06, 2.03	1.50, 1.45	0.04, 0.04					

At pH 4, 7 and 9 and 50 °C less than 10% hydrolysis was observed after 5 days, equivalent to an environmental half-life of greater than 1 year at each pH value investigated. At pH 11 and 13 and 50 °C, significant hydrolysis was observed, with respective half-lives of 45 hours 4.2 hours observed.

Dinotefuran is hydrolytically stable under acid, neutral and weak alkaline conditions, but unstable under more strongly basic conditions. The main hydrolysis product was confirmed by LC-MS analysis to be UF.

METHODS OF RESIDUE ANALYSIS

Analytical methods

Descriptions of analytical methods together with validation data for residues of dinotefuran and its metabolites (DN and UF) in plant and animal matrices were submitted to the Meeting. The methods rely on an initial extraction, usually with acetonitrile/water. After solvent partition and column clean-up, the dinotefuran and metabolite residues are prepared for LC analysis. Dinotefuran residues can be measured either by UV detector or mass selective detector (MSD), typically with an LOQ of 0.01 mg/kg. Determinations for the metabolites were conducted using HPLC with mass spectrometric detector (MS/MS). The LOQ for the metabolites were typically 0.01 mg/kg. Since the methods use standard extraction solvents and standard detection techniques, they have the potential to be incorporated into existing multi-residue methods.

Detailed descriptions of all these analytical methods are presented below.

Plant matrices

Lettuce (RCC 784945), Cotton (seeds) (RCC 774843)

Analyte:	Dinotefuran	LC-UV (270 nm)
LOQ:	0.01 mg/kg	
Description	Samples are extracted with acetonitrile/water (8:2, v/v). The acetonitrile/water phase is evaporated to aqueous remainder at reduced pressure. Liquid-liquid separation is carried out using hexane. The aqueous phase is transferred onto the Extrelut 20 column. Elution is performed using dichloromethane. The solution is evaporated to dryness and the residue is re-dissolved in methanol/water. The sample solution is transferred onto the ENVI Carb cartridge and eluted using acetonitrile/water (8:2, v/v). The aqueous phase is transferred onto the Extrelut 20 column and eluted using dichloromethane. The solution is evaporated to dryness and the residue is re-dissolved in distilled water. Residues of dinotefuran are determined by HPLC with an UV detector.	

Cotton (seeds, gin trash, meal, hulls and refined oil) (RCC 841464)

Analyte:	Dinotefuran (m/z 203→129) and UF (m/z 159→102)	LC-MS/MS
LOQ:	0.05 mg/kg for seeds, gin trash, meal and hulls, 0.01 mg/kg for refined oil	

Description Samples are extracted with acetonitrile/water (8:2 v/v) under acid condition. Liquid-liquid separation is carried out using hexane. The acetonitrile/water phase is evaporated to aqueous remainder at reduced pressure. The pH value of the aqueous residue is adjusted to pH 8 using buffer solution. Liquid-liquid separation is carried out using hexane The aqueous solution is evaporated to remove any remaining hexane. The solution is transferred onto the Extrelut NT3 column and eluted using ethyl acetate. The solution is evaporated to dryness, re-dissolved in water and filtered. Residues of dinotefuran and UF are determined after HPLC separation using an MS/MS detector.

Analyte: DN (m/z 158→102) LC-MS/MS

LOQ: 0.05 mg/kg for seeds, gin trash, meal and hulls, 0.01 mg/kg for refined oil

Description Samples are extracted with acetonitrile/water (8:2 v/v) under acid condition. Liquid-liquid separation is carried out using hexane. The acetonitrile/water phase is evaporated to aqueous remainder at reduced pressure. The pH value of the aqueous residue is adjusted to pH 8 using buffer solution. The sample solution is transferred onto the Bond Elut CBA cartridge and rinsed using distilled water followed by methanol. Elution is performed using hydrochloric acid (0.1 N). The solution is evaporated to dryness, re-dissolved in hydrochloric acid (0.1 N) and filtered. Residues of DN are determined by HPLC using an MS/MS detector.

Lettuce (RCC 836111)

Analyte: UF (m/z 159→102) LC-MS/MS

LOQ: 0.01 mg/kg

Description Samples are extracted with acetonitrile/water (8:2 v/v) under acid condition. Liquid-liquid separation is carried out using hexane. The acetonitrile/water phase is evaporated to aqueous remainder at reduced pressure. The pH value of the aqueous residue is adjusted to pH 7–8 using buffer solution. Liquid-liquid separation is carried out using hexane The aqueous solution is evaporated to remove any remaining hexane. The solution is transferred onto the Extrelut NT3 column and eluted using ethyl acetate. The solution is evaporated to dryness, re-dissolved in water and filtered. Residues of dinotefuran and UF are determined after HPLC separation using an MS/MS detector.

Analyte: DN LC-MS

LOQ: 0.01 mg/kg

Description Samples are extracted with acetonitrile/water (8:2 v/v) under acid condition. Liquid-liquid separation is carried out using hexane. The acetonitrile/water phase is evaporated to aqueous remainder at reduced pressure. The pH value of the aqueous residue is adjusted to pH 7–8 using buffer solution. The sample solution is transferred onto the Bond Elut CBA cartridge and rinsed using distilled water followed by methanol. Elution is performed using hydrochloric acid (0.1 N). The solution is evaporated to dryness, re-dissolved in hydrochloric acid (0.1 N) and filtered. Residues of DN are determined by HPLC using an MS detector.

Lettuce (RCC 795306), Celery and Spinach (RCC 810055)

Analyte: Dinotefuran LC-UV (270 nm)

LOQ: 0.01 mg/kg

Description Samples were extracted with an acetonitrile/water (8:2, v/v) mixture, the phase was evaporated to aqueous remainder, and liquid-liquid separation was carried out using hexane. After transferring the aqueous solution onto an Extrelut 20 column, elution was performed using dichloromethane. The solution was evaporated to dryness and re-dissolved in methanol/water. After clean-up with an ENVI Carb SPE cartridge, the second liquid-liquid partition was performed. The concentration of dinotefuran was then quantified, after HPLC separation, by UV detection.

Analyte: DN (m/z 158→102) and UF (m/z 159→102) LC-MS and MS/MS

LOQ: 0.01 mg/kg

Description Samples were extracted with an acetonitrile/water (8:2, v/v) mixture and the phase was evaporated to aqueous remainder.

DN: An aliquot of the solution was buffered and centrifuged. Clean-up was performed with a Bond Elut CBA cartridge. The solution was filtered, and the concentration of DN was determined, after HPLC separation, using a MS detector.

UF: An aliquot of the solution was cleaned with hexane. Solution was transferred onto an Extrelut NT3 column and elution was performed with ethyl acetate. The solution was evaporated to dryness and re-dissolved in water. The solution was filtered, and the concentration of UF was determined, after HPLC separation, using a MS/MS detector.

Rice (husked rice), Tomato

Analyte: Dinotefuran LC-UV (270 nm)

LOQ: 0.01 mg/kg

Description: Samples are added of distilled water and soaked for 2 hours. Residues of dinotefuran are extracted from the sample matrix by maceration with acetonitrile/water (8:2, v/v). The concentrated solution is transferred onto porous diatomite column (Chem Elut 1020) and rinsed with hexane. The eluate with ethyl acetate is transferred onto graphite-carbon column (ENVI-Carb) and rinsed with methanol and water. The eluate was further cleaned up on Chem Elut 1020 with water/acetonitrile (8:2, v/v) . Determination of dinotefuran is conducted using HPLC employing UV (UVD) detection.

Rice (grain, straw, bran and hulls), Tomato, Melon, Potato, Grape, Broccoli, Lettuce
Analyte: Dinotefuran (m/z 203→129) and UF (m/z 159→102) LC-MS/MS
LOQ: 0.01 mg/kg
Description: Samples are extracted with acetonitrile/water (8:2, v/v) and partitioned with hexane. The acetonitrile/water phase is concentrated and adjusted to pH 8 using a buffer solution. The aqueous solution is transferred onto Extrelut NT3 column and the residues are eluted with ethyl acetate. The eluted residues are evaporated and reconstituted in 0.1 N HCl. The solution is analysed by HPLC with mass spectrometric (MS/MS) detection.
Analyte: DN (m/z 158→102) LC-MS/MS
LOQ: 0.01 mg/kg
Description: Samples are extracted with acetonitrile/water (8:2, v/v) and partitioned with hexane. The acetonitrile/water phase is concentrated and adjusted to pH 8 using a buffer solution. The aqueous solution is transferred onto BondElut CBA column and the cartridge is rinsed with water and methanol. The residues are eluted with 0.1 N HCl. The solution is analysed by HPLC with mass spectrometric (MS/MS) detection.

Multi Residue Method

Lettuce (non-fatty food) 236C-103
Analyte: Dinotefuran GC-NPD Method: PAM
LOQ: 0.01 mg/kg
Description Sample is extracted with acetone followed by partition with petroleum ether/methylene chloride. The organic phase is concentrated, and residues of dinotefuran are determined by gas chromatography with a nitrogen/phosphorus selective detection (NPD).

Cotton meal, Cotton oil (fatty food) 236C-103
Analyte: Dinotefuran GC-ECD Method: PAM
LOQ: 0.01 mg/kg
Description: 100 g of Sample is extracted with 200 mL of petroleum ether, 150 mL of ethyl ether/petroleum ether (1:1) and 150 mL of ethanol. The extracts are shaken with water. The aqueous phase is added to petroleum ether and mixed. The organic phase is washed with water and concentrated. The residue is analysed by gas chromatography with an electron captured detector (ECD).

The recoveries of residues from plant matrices are summarized in Tables 29 and 30.

Table 29 Summary of recovery data of dinotefuran from fortified plant matrices

Commodity	Fortification mg/kg	N	Range Recovery (%)	Mean recovery (%)	% RSD	Reference
Peach	0.01	3	99–102	100	1.5	IR-4 09548
	0.50	3	106–109	107	1.6	
	5.0	3	112–114	113	1.0	
Cranberry	0.01	3	90–94	91	2.5	IR-4 09832
	0.10	3	98–101	100	1.7	
Onion, Bulb	0.01	3	81–88	85	4.2	IR-4 08645
	0.10	3	94–106	98	7.1	
	1.0	3	100–115	105	7.9	
Onion, green	0.01	5	85–103	93	6.9	IR-4 09550
	0.10	3	85–95	90	5.5	
	1.0	3	97–102	99	2.6	
	4.0	3	80–101	89	12.3	
Lettuce	0.01	5	82.4–91.7	88.1	4.6	RCC 784945
	0.10	5	74.2–92.3	80.7	9.1	
	5.0	5	73.6–106.6	85.7	15.7	
Lettuce	0.01	3	83.3–100	91	9.27	236C-107
	0.10	3	85.2–90.7	88.3	3.18	(ILV)
	0.50	3	82.4–88.2	86.2	3.79	
Watercress	0.01	3	89–105	95	8.9	IR-4 09514
	0.10	3	86–94	91	5.1	
	1.0	3	90–94	92	2.2	
	4.0	3	91–104	97	6.8	
Rice, husked	0.01	3	94–99	96	2.8	JFRL
	0.40	3	84–101	92	9.3	

Commodity	Fortification mg/kg	N	Range Recovery (%)	Mean recovery (%)	% RSD	Reference
Rice, husked	0.01 0.40	3 3	94–115 103–104	104 103	10.1 0.6	
Rice, grain	0.01 0.10	7 3	90.5–97.3 102–106	94.2 104	2.83 1.76	236C-162
Rice, straw	0.10 1.0	7 3	101–110 102–112	105 107	2.89 4.60	
Rice, bran	0.01 0.10	7 3	97.2–108 96.9–107	103 104	3.46 5.63	
Rice, hulls	0.01 0.10	7 3	96.7–108 102–105	103 105	3.70 1.89	
Cotton, seeds	0.01 0.10 0.30	5 5 5	101.8–109.7 87.3–95.4 78.9–105.3	105.3 91.0 84.7	2.8 4.1 13.5	RCC 774843
Cotton, seeds	0.05 0.50	5 5	78.6–93.0 90.7–101.5	86.0 96.6	6.0 4.7	RCC 841464
Cotton, gin trash	0.05 0.50	5 5	109.3–110.4 82.6–98.8	109.7 94.9	0.4 7.3	
Cotton, meal	0.05 0.50	5 5	71.7–87.6 72.8–85.7	79.0 79.0	8.5 6.4	
Cotton, hulls	0.05 0.50	5 5	95.1–110.1 74.0–95.6	100.1 90.0	6.1 10.3	
Cotton, refined oil	0.01 0.10	5 5	74.9–95.2 80.0–94.2	83.2 86.6	11.6 7.2	
Cotton, seeds	0.05 0.50 2.50	3 3 3	103–106 104–106 99.1–102	104 105 100	1.47 0.95 1.64	236C-108 (ILV)
Cotton, gin trash	0.05 0.50 2.50	3 3 3	102–107 98.6–104 87.2–92.8	104 101 90.5	2.41 2.67 3.24	
Cotton, meal	0.05 0.50 2.50	3 3 3	109–116 108–118 94.4–100	113 112 97.8	3.12 4.57 3.03	
Cotton, oil	0.01 0.10 0.50	3 3 3	100–102 100–101 99.7–102	101 100 101	1.14 0.575 1.14	
Tomato	0.01 1.0	5 5	105–113 107–115	110	2.7	236C-113
Melon	0.01 1.0	5 5	82.1–88.8 84.8–89.2	85.5	3.1	
Potato	0.01 1.0	5 5	97.6–106 108–115	107	5.8	
Grape	0.01 1.0	5 5	89.2–101 100–111	100	7.2	
Broccoli	0.01 1.0	5 5	89.3–97.4 101–110	99.4	6.4	
Lettuce	0.01 1.0	5 5	94.0–116 104–109	109	5.9	
Tomato, paste	0.01 1.0	5 5	89.9–107 85.6–98.0	94.4	8.3	
Tomato, puree	0.01 1.0	5 5	102–110 87.6–95.7	99.0	8.8	
Potato, flakes	0.01 1.0	4 5	102–113 85.7 –110	102	8.8	
Potato, chips	0.01 1.0	5 5	88.1–98.1 93.1–102	95.2	4.9	
Potato, wet peel	0.01 1.0	5 5	70.6–90.2 72.7–106	87.3	14.1	
Raisins	0.01 1.0	5 5	87.5–95.3 88.5–101	92.3	5.0	
Grape, juice	0.01 1.0	5 5	92.5–107 100 –110	103	6.3	

Table 30 Summary of recovery data of metabolites from fortified into plant matrices

Commodity	Fortification mg/kg	N	Range Recovery (%)	Mean recovery (%)	% RSD	Reference
Peach	0.01	3	88–97	92	5.1	IR-4 09548
DN	0.50	3	96–99	97	1.5	
	5.0	3	93–96	95	1.8	
Peach	0.01	3	108–113	110	2.4	
UF	0.50	3	106–107	106	0.5	
	5.0	3	108–113	110	2.3	
Cranberry	0.01	3	84–88	86	2.4	IR-4 09832
DN	0.10	3	92–94	93	1.2	
Cranberry	0.01	3	105–107	106	1.1	
UF	0.10	3	107–111	109	1.8	
Onion, Bulb	0.01	3	87–95	91	4.5	IR-4 08645
DN	0.10	3	98–102	100	2.0	
	1.0	3	97–100	98	1.6	
Onion, Bulb	0.01	3	98–114	108	7.9	
UF	0.10	3	103–108	106	2.7	
	1.0	3	106–109	107	1.4	
Onion, green	0.01	5	87–105	96	9.1	IR-4 09550
DN	0.10	3	87–106	96	9.9	
	1.0	3	78–106	96	16.5	
	4.0	3	85–98	90	8.1	
Onion, green	0.01	5	94–102	98	3.1	
UF	0.10	3	89–95	93	3.4	
	1.0	3	95–100	98	2.6	
	4.0	3	79–103	89	14.0	
Lettuce	0.01	5	102.2–108.9	105.2	2.3	RCC 836111
DN	0.10	5	94.7–109.9	104.6	6.0	
	0.50	5	93.8–102.7	98.2	4.15	
Lettuce	0.01	5	83.8–102.4	92.0	8.1	
UF	0.10	5	85.0–99.3	90.0	6.4	
	0.50	5	77.4–95.7	90.2	8.6	
Lettuce	0.01	3	87.4–106	96.1	9.74	236C-107
DN	0.10	3	88.9–99.0	94.8	5.57	(ILV)
	0.50	3	84.9–87.8	86.4	1.68	
Lettuce	0.01	3	77.7–84.3	81.8	4.40	
UF	0.10	3	85.7–95.2	91.3	5.43	
	0.50	3	87.5–90.8	89.5	1.96	
Watercress	0.01	3	97–105	102	4.1	IR-4 09514
DN	0.10	3	73–107	90	19.0	
	1.0	3	99–104	102	2.8	
	4.0	3	83–102	93	10.2	
Watercress	0.01	3	77–102	88	14.5	
UF	0.10	3	87–99	92	6.8	
	1.0	3	87–93	90	3.3	
	4.0	3	92–106	97	8.1	
Rice, grain	0.01	7	89.3–97.4	93.8	3.60	236C-162
DN	0.10	3	90.3–98.9	94.0	4.70	
Rice, grain	0.01	7	90.3–98.6	95.1	2.72	
UF	0.10	3	95.0–98.6	96.3	2.08	
Rice, straw	0.10	7	92.7–100	98.0	2.69	
DN	1.0	3	96.2–99.8	98.5	2.06	
Rice, straw	0.10	7	96.8–104	101	2.76	
UF	1.0	3	98.7–106	103	3.82	
Rice, bran	0.01	7	83.7–100	89.6	6.45	
DN	0.10	3	70.6–75.8	73.1	3.52	
Rice, bran	0.01	7	84.7–91.8	88.1	2.99	
UF	0.10	3	93.7–97.3	95.7	1.90	
Rice, hulls	0.01	7	112–122	116	3.38	
DN	0.10	3	101–109	106	3.91	
Rice, hulls	0.01	7	96.6–105	102	2.66	
UF	0.10	3	93.3–98.5	96.5	2.95	

Commodity	Fortification mg/kg	N	Range Recovery (%)	Mean recovery (%)	% RSD	Reference
Cotton, seeds	0.05	5	84.3–102.5	93.9	6.9	RCC 841464
DN	0.50	5	84.4–95.2	90.6	4.5	
Cotton, seeds	0.05	5	70.9–79.7	73.5	4.9	
UF	0.50	5	80.9–93.4	86.7	5.2	
Cotton, gin trash	0.05	5	87.4–97.7	91.6	5.7	
DN	0.50	5	78.2–104.4	89.5	10.7	
Cotton, gin trash	0.05	5	70.3–92.9	81.6	10.5	
UF	0.50	5	78.4–90.8	87.4	6.0	
Cotton, meal	0.05	5	71.8–87.4	80.6	7.0	
DN	0.50	5	71.5–83.8	77.7	6.6	
Cotton, meal	0.05	5	76.3–84.3	81.2	3.7	
UF	0.50	5	84.9–94.8	87.6	4.7	
Cotton, hulls	0.05	5	72.4–81.5	75.1	4.9	
DN	0.50	5	69.7–76.5	72.4	3.8	
Cotton, hulls	0.05	5	70.7–89.2	78.5	8.7	
UF	0.50	5	84.2–89.1	86.6	2.7	
Cotton, refined oil	0.01	5	86.9–107.2	93.6	9.4	
DN	0.10	5	85.1–102.0	95.5	6.6	
Cotton, refined oil	0.01	5	74.8–97.0	82.9	11.3	
UF	0.10	5	79.4–89.1	84.4	5.3	
Cotton, seeds	0.05	3	98.7–104	102	2.64	236C-108
DN	0.50	3	89.3–93.7	91.7	2.42	(ILV)
	2.50	3	90.8–93.4	92.3	1.47	
Cotton, seeds	0.05	3	94.4–96.6	95.4	1.16	
UF	0.50	3	92.2–98.7	95.2	3.45	
	2.50	3	88.5–96.7	93.7	4.83	
Cotton, gin trash	0.05	3	101–102	101	0.570	
DN	0.50	3	94.5–96.1	95.1	0.894	
	2.50	3	97.1–99.6	98.2	1.31	
Cotton, gin trash	0.05	3	86.9–90.3	88.9	2.02	
UF	0.50	3	91.7–94.2	93.3	1.52	
	2.50	3	84.0–88.9	86.7	2.86	
Cotton, meal	0.05	3	100–103	101	1.51	
DN	0.50	3	94.5–95.6	95.1	0.598	
	2.50	3	91.9–97.8	94.1	3.43	
Cotton, meal	0.05	3	93.6–99.6	97.1	3.23	
UF	0.50	3	95.0–102	97.7	3.85	
	2.50	3	87.0–92.4	90.5	3.33	
Cotton, oil	0.01	3	95.9–103	98.9	3.73	
DN	0.10	3	92.1–99.6	97.0	4.40	
	0.50	3	102–104	103	1.12	
Cotton, oil	0.01	3	100–106	103	2.98	
UF	0.10	3	96.6–99.1	98.0	1.32	
	0.50	3	97.3–99.8	98.9	1.38	
Tomato	0.01	5	87.5–97.8	94.4	4.6	236C-113
DN	1.0	5	93.4–99.2			
Tomato	0.01	5	89.0–95.4	98.5	7.4	
UF	1.0	5	104–106			
Melon	0.01	5	95.1–108	101	4.0	
DN	1.0	5	98.5–102			
Melon	0.01	5	90.9–98.7	100	6.2	
UF	1.0	5	104–107			
Potato	0.01	5	86.3–90.1	90.0	2.8	
DN	1.0	5	90.0–93.3			
Potato	0.01	5	102–104	104	2.1	
UF	1.0	5	99.7–107			
Grape	0.01	5	84.5–87.5	87.3	1.8	
DN	1.0	5	85.3–90.1			
Grape	0.01	5	98.5–105	104	2.7	
UF	1.0	5	101–108			
Broccoli	0.01	5	88.1–91.1	89.9	1.7	
DN	1.0	5	88.8–93.2			

Commodity	Fortification mg/kg	N	Range Recovery (%)	Mean recovery (%)	% RSD	Reference
Broccoli UF	0.01 1.0	5 5	97.9–104 95.7–104	101	3.3	
Lettuce DN	0.01 1.0	5 5	109–118 99.0–104	108	6.1	
Lettuce UF	0.01 1.0	5 5	106–116 105–116	111	3.3	
Tomato, paste DN	0.01 1.0	5 5	78.5–91.1 72.3–76.0	78.6	8.3	
Tomato, paste UF	0.01 1.0	5 5	85.6–96.1 93.5–99.3	92.5	4.5	
Tomato, puree DN	0.01 1.0	5 5	91.2–103 85.5–98.5	94.6	5.2	
Tomato, puree UF	0.01 1.0	5 5	90.9–100 91.2–103	96.1	4.2	
Potato, flakes DN	0.01 1.0	5 5	90.9–98.0 88.7–93.8	93.0	2.9	
Potato, flakes UF	0.01 1.0	5 5	76.4–96.6 74.8–104	91.8	10	
Potato, chips DN	0.01 1.0	5 5	86.9–92.6 90.0–94.2	90.6	3.1	
Potato, chips UF	0.01 1.0	5 5	85.7–94.7 88.0–96.6	91.1	4.1	
Potato, wet peel DN	0.01 1.0	5 5	98.5–107 100–111	104	3.6	
Potato, wet peel UF	0.01 1.0	5 5	86.1–105 85.2–107	97.0	8.7	
Raisins DN	0.01 1.0	5 5	72.3–78.3 67.0–73.3	72.7	4.2	
Raisins UF	0.01 1.0	5 5	78.2–89.8 86.7–97.4	87.1	6.5	
Grape, juice DN	0.01 1.0	5 5	74.3–116 77.9–93.1	88.1	13	
Grape, juice UF	0.01 1.0	5 5	101–108 103–111	105	3.1	

The Meeting received a study for the efficiency of the extraction method validated for the determination of dinotefuran in cotton (RCC 774843). The test system consisted of the samples obtained from the dinotefuran plant metabolism study on oilseed rape (RCC 767880). Plant parts (seeds and foliage) were extracted at room temperature using acetonitrile/water (8:2; v/v). After extraction, the residual debris was dried and combusted. The results are summarized in Table 31.

Table 31 Efficiency of the extraction method validated for the determination of dinotefuran in cotton

Sample	Seeds			Foliage		
Method	Plant metabolism method	Residue analytical method		Plant metabolism method	Residue analytical method	
	% TRR	% TRR	mg/kg [a]	% TRR	% TRR	mg/kg [a]
Extractables	81.9	81.4	0.567	94.4	89.4	2.10
Non-extractables	18.1	18.5	0.129	5.6	10.6	0.249
Total	100	100	0.696	100	100	2.35

[a] parent dinotefuran equivalents

Animal matrices

Milk, Cream, Bovine tissues and Hen eggs (MTU250/024447)

Analyte:	Dinotefuran (m/z 203→129), DN (m/z 158→102)and UF (m/z 159→102)	LC-MS/MS
LOQ:	0.01 mg/kg	

Description Samples are extracted with acidified acetonitrile/water (80:20 containing 0.2% hydrochloric acid). Hexane extract is added to the extract. After partitioning the hexane phase is discarded. The extract is transferred to a round bottom flask, acetone is added and the sample is evaporated to remove the organic phase. The aliquot is purified through a C_{18} cartridge. Determination of dinotefuran is conducted using HPLC employing mass spectrometric (MS/MS) detection. Samples requiring further dilution are diluted using 0.1 N hydrochloric acid.

Milk, Muscle, Liver, Kidney and Fat (017472-1)
Analyte: Dinotefuran (m/z 203→129), DN (m/z 158→102) and UF (m/z 159→102) LC-MS/MS
LOQ: 0.01 mg/kg
Description Samples are extracted with acetonitrile/water (8:2) and centrifuged to remove particulates. Milk samples are extracted with the same solvent, but are sonicated and then centrifuged. The extract was transferred to another tube and partitioned with hexane. The hexane is discarded and the acetonitrile/water phase is brought to a larger volume with the addition of acetone. The extract is evaporated to water and is reconstituted to 6 mL with water. Half of this sample (3 mL) is transferred to a new tube and methanol is added. This half is then added to a preconditioned 1 gram C_{18} solid phase extraction cartridge (SPE) and the eluent is collected in a vial. An additional methanol/water (1:1) is added to further elute the compounds from SPE cartridge. The eluent is evaporated to aqueous and reconstituted with 0.1 N HCl. All samples are analysed using HPLC with MS/MS.

The recovery data from animal matrices are summarized in Tables 32 and 33.

Table 32: Summary of recovery data of dinotefuran from fortified animal matrices

Commodity	Fortification mg/kg	N	Range of Recovery (%)	Mean recovery (%)	% RSD	Reference
Bovine liver	0.01 1.0	5 5	71–93 75–81	86 79	10.1 2.9	MTU 250/024447
Bovine kidney	0.01 1.0	5 5	72–94 72–87	81 80	10.5 8.5	
Bovine muscle	0.01 1.0	5 5	70–78 70–83	74 75	4.9 7.6	
Bovine fat	0.01 1.0	5 5	75–98 74–85	84 80	13.7 6.2	
Bovine milk	0.01 1.0	5 5	88–110 72–84	98 78	8.4 6.5	
Bovine cream	0.01 1.0	5 5	77–100 80–105	89 89	10.3 10.5	
Hen eggs	0.01 1.0	5 5	71–110 79–88	83 83	18.9 4.6	
Bovine fat	0.01 1.0	5 5	91.4–113 80.9–89.8	105 85.1	9.7 3.2	017472-1
Bovine cream	0.01 1.0	5 5	68–99.7 89.4–105	92.7 98.9	13.8 6.5	
Bovine milk	0.01 1.0	5 5	92.0–108 88.9–95.6	102 92.5	6.2 4.2	
Bovine muscle	0.01 1.0	5 5	96.9–106 91.7–98.7	102 95.0	4.4 3.2	
Bovine liver	0.01 1.0	5 5	80.9–102 89.1–92.6	88.7 90.4	7.7 1.4	
Bovine kidney	0.01 1.0	5 5	89.1–96.8 89.0–90.9	91.5 89.7	3.0 0.9	

Table 33 Summary of recovery data of metabolites from fortified animal matrices

Commodity	Fortification mg/kg	N	Range of Recovery (%)	Mean recovery (%)	% RSD	Reference
Bovine liver DN	0.01 1.0	5 5	72–104 86–106	84 99	18.2 8.0	MTU 250/024447
Bovine liver UF	0.01 1.0	5 5	72–102 78–91	88 86	14.4 6.2	
Bovine kidney DN	0.01 1.0	5 5	72–86 98–103	80 101	8.5 1.9	

Commodity	Fortification mg/kg	N	Range of Recovery (%)	Mean recovery (%)	% RSD	Reference
Bovine kidney	0.01	5	74–96	86	11.5	
UF	1.0	5	74–86	81	6.7	
Bovine muscle	0.01	5	74–85	78	5.7	
DN	1.0	5	80–107	92	14.0	
Bovine muscle	0.01	5	72–98	85	12.5	
UF	1.0	5	75–88	80	8.1	
Bovine fat	0.01	5	73–106	89	14.1	
DN	1.0	5	84–102	90	8.1	
Bovine fat	0.01	5	76–91	84	7.4	
UF	1.0	5	74–84	81	4.9	
Bovine milk	0.01	5	70–105	89	15.5	
DN	1.0	5	77–96	90	8.5	
Bovine milk	0.01	5	81–85	83	1.8	
UF	1.0	5	73–93	85	8.9	
Bovine cream	0.01	5	91–107	98	6.9	
DN	1.0	5	95–108	100	5.7	
Bovine cream	0.01	5	81–104	93	10.7	
UF	1.0	5	83–96	90	5.6	
Hen eggs	0.01	5	73–108	93	19.2	
DN	1.0	5	91–105	98	5.5	
Hen eggs	0.01	5	70–103	81	16.1	
UF	1.0	5	75–90	83	6.7	
Bovine fat	0.01	5	88.5–105	99.9	7.6	017472-1
DN	1.0	5	72.3–77.4	75.4	2.3	
Bovine fat	0.01	5	110–124	119	7.3	
UF	1.0	5	97.0–106	101	3.3	
Bovine cream	0.01	5	64.1–97.6	84.5	12.7	
DN	1.0	5	90.2–93.4	92.2	1.3	
Bovine cream	0.01	5	78.1–124	109	18.7	
UF	1.0	5	102–108	104	2.2	
Bovine milk	0.01	5	100–133	118	15.1	
DN	1.0	5	86.6–93.2	90.4	3.1	
Bovine milk	0.01	5	104–121	111	8.0	
UF	1.0	5	93.3–105	99.5	4.4	
Bovine muscle	0.01	5	105–116	110	5.4	
DN	1.0	5	89.2–92.1	91.0	1.2	
Bovine muscle	0.01	5	106–113	110	2.8	
UF	1.0	5	91.1–99.2	94.7	3.4	
Bovine liver	0.01	5	76.8–85.5	81.6	3.7	
DN	1.0	5	96.9–111	103	5.6	
Bovine liver	0.01	5	101–126	108	9.9	
UF	1.0	5	88.8–97.7	94.3	3.5	
Bovine kidney	0.01	5	110–117	113	2.9	
DN	1.0	5	96.8–100	98.8	1.3	
Bovine kidney	0.01	5	108–116	111	4.2	
UF	1.0	5	93.7–100	96.8	2.7	

Stability of residues in stored analytical samples

The Meeting received data on the stability of dinotefuran residues in plants (apple, tomato, lettuce, potato, cotton and rice) samples and animal commodities during frozen storage, and the data on frozen storage stability which were conducted concurrently with the supervised trials on peach, grape, cranberry, bulb onion, green onion, broccoli, melon, tomato, lettuce, watercress and potato.

The stability study of dinotefuran was conducted on apple, tomato, lettuce, potato and cotton seeds at about -20 °C (Wais, 2002: 827537). Samples of untreated homogenized raw agricultural commodities (RAC) were fortified with dinotefuran at 0.50 mg/kg (50 times LOQ) prior to storage. Samples were analysed after storage at about -20 °C (0, 3, 5 (lettuce and cotton seeds) 6, 9 and 12 months). The residues of dinotefuran were determined using LC-UVD. The LOQ was 0.01 mg/kg.

Up to 12 months, the mean percentages of residues remaining in the stored samples were ≥ 70% of the theoretical value. Dinotefuran can be regarded as stable over a storage period of at least 12 months at about -20 °C.

Table 34 Recovery of dinotefuran from stored fortified samples

Storage period	Recovery (%) [0.50 mg/kg fortification]		
	Procedural	% remaining	Mean
Apple			
0 month	70.1	73.6, 79.7	76.7
1 month	75.6	73.2, 74.3, 79.0	75.5
3 months	71.7	70.0, 84.5, 88.3	80.9
6 months	72.6	69.3, 71.7, 78.6	73.2
12 months	– [a]	71.4, 74.6, 77.7	74.6
Tomato			
0 month	81.4	79.4, 89.4	84.4
1 month	75.7	82.2, 84.8, 89.1	85.4
3 months	97.9	81.3, 88.8, 97.3	89.1
6 months	88.6	90.9, 91.1, 95.0	92.3
9 months	77.9	82.0, 83.2, 83.4	82.9
12 months	99.4	97.7, 99.2, 101.4	93.4
Lettuce			
0 month	77.0	74.6, 87.1	80.9
1 month	85.1	80.6, 85.0, 98.8	88.1
3 months	90.3	91.0, 91.1, 92.8	91.6
5 months	89.4	64.2, 79.4, 90.8	78.1
9 months	73.7	75.7, 78.2, 82.4	78.8
12 months	70.4	68.7, 71.1, 72.0	70.6
Potato			
0 month	79.4	77.3, 78.9	78.1
1 month	78.4	72.9, 75.7, 82.9	77.2
3 months	82.3	68.1, 77.7, 93.5	79.8
6 months	79.5	73.8, 78.8, 81.7	78.1
9 months	84.7	73.8, 75.7, 78.4	76.0
12 months	72.6	69.1, 73.4, 77.8	73.4
Cotton seeds			
0 month	70.5	71.7, 76.0	73.9
1 month	70.8	72.5, 75.7	74.1
3 months	79.3	79.3, 79.6, 81.7	80.2
5 months	67.5	65.9, 71.1, 72.3	70.0
9 months	-*	67.6, 76.6, 82.9	75.7
12 months	77.1	78.6, 80.7, 81.0	80.1

[a] During analysis of the 12-months apple and 9-months cotton seeds samples, the freshly fortified sample produced meaningless results; however all three recovery values of the stored samples were > 70% and within a range of previous recoveries that would indicate approximately 100% relative recovery.

The storage stability of dinotefuran, UF and DN added to rice grain, straw, hulls and bran was assessed under freezer storage conditions (MacGregor, 2011: 236C-163). At initiation of the freezer storage assessment, sample sets were prepared to confirm the nominal fortification concentration and sets were prepared for subsequent analysis following freezer-storage intervals. The initial sample sets for each substrate were analysed both as method validation samples and as the analytical sets to initiate stability testing. The initial sample sets, shared with method validation, consisted of two controls and three procedural recoveries. Sample sets for each subsequent storage interval consisted of three control (untreated) samples and two fortified at a concentration of 0.10 mg/kg for rice grain, bran and hulls and 1.0 mg/kg for rice straw. For the second storage interval, the storage period was approximately four months (115 to 119 days), which encompassed the freezer storage interval for processed commodities, hulls and bran, collected in a residue program. The last interval consisted only of analyses of rice grain and straw extending the freezer stability assessment to 434 days for grain and 431 days for straw.

Table 35 Recovery of dinotefuran, DN and UF from stored fortified samples of rice

Storage interval (days)		% Recovery (0.10 mg/kg fortification for grain, bran and hulls, 1.0 mg/kg for straw)		
		Procedural	% remaining	Mean
Grain				
Dinotefuran	0	102, 103, 106		
	30	82.8, 91.5	87.5, 94.4	91.0
	118	89.1, 91.7	91.3, 94.2	92.8
	434	90.3, 92.8	97.6, 97.8	97.7
DN	0	90.3, 92.9, 98.9		
	30	87.7, 89.4	89.2, 90.6	89.9
	118	90.1, 90.1	91.1, 92.0	91.6
	434	90.9, 91.4	89.7, 92.3	91.0
UF	0	95.0, 95.3, 98.6		
	30	92.1, 98.5	94.8, 98.9	96.9
	118	81.4, 86.8	85.7, 89.4	87.6
	434	96.8, 99.6	100, 101	101
Straw				
Dinotefuran	0	102, 108, 112		
	32	93.0, 93.8	96.9, 98.1	97.5
	116	99.8, 105	106, 106	106
	431	99.6, 101	104, 106	105
DN	0	96.2, 99.6, 99.8		
	32	94.9, 97.6	98.5, 104	101
	116	90.7, 97.9	100, 108	104
	431	98.0, 98.6	96.9, 97.5	97.2
UF	0	98.7, 103, 106		
	32	98.7, 99.3	100, 100	100
	116	94.1, 98.6	98.3, 99.4	98.9
	431	103, 106	102, 103	103
Bran				
Dinotefuran	0	96.9, 107, 107		
	30	114, 118	114, 116	115
	119	112, 122	118, 124	121
DN	0	70.6, 72.8, 75.8		
	30	49.0, 49.2	46.9, 56.3	51.6
	119	72.5, 81.1	76.5, 78.4	77.5
UF	0	93.7, 95.9, 97.3		
	30	98.7, 104	102, 103	103
	119	91.7, 104	95.6, 99.2	97.4
Hulls				
Dinotefuran	0	103, 105, 107		
	30	104, 106	101, 105	103
	115	97.2, 107	106, 111	108
DN	0	101, 106, 109		
	30	96.9, 100	103, 104	104
	115	88.7, 97.9	88.8, 90.1	89.5
UF	0	93.3, 97.9, 98.5		
	30	92.5, 95.6	85.8, 94.7	90.2
	115	89.7, 91.7	90.1, 91.5	90.8

The storage stability study of dinotefuran, DN and UF in cotton (seeds, gin trash, hulls, meal and oil) was conducted at about -20 ºC (Wolf, 2003: 842365). Samples of untreated homogenized cotton seeds, gin trash, hulls and meal were fortified with dinotefuran, DN and UF at 0.50 mg/kg (50× LOQ) prior to storage. Sample of untreated oil was fortified with dinotefuran, DN and UF at 0.10 mg/kg (10× LOQ). Samples were analysed after storage at about -20 °C (1, 3, 6 and 12 months).

Table 36 Recovery of dinotefuran, DN and UF from stored fortified samples of cotton

Storage interval (months)	% Recovery (0.10 mg/kg fortification for grain, bran and hulls, 1.0 mg/kg for straw)		
	Procedural	% remaining	Mean
Seeds			

Storage interval (months)		% Recovery (0.10 mg/kg fortification for grain, bran and hulls, 1.0 mg/kg for straw)		
		Procedural	% remaining	Mean
Dinotefuran	1	91.2	88.5, 90.0, 92.3	90.3
	3	88.9	71.2, 87.5, 93.3	84.0
	6	101	91.1, 96.0, 98.3	95.1
	12	98.1	98.9, 102, 102	101
DN	1	73.4	75.1, 75.4, 83.1	77.9
	3	80.2	73.3, 75.9, 80.6	76.6
	6	73.9	88.9, 88.9, 97.3	91.7
	12	71.7	85.7, 86.1, 90.6	87.5
UF	1	84.7	92.4, 93.9, 97.1	94.5
	3	82.4	79.4, 88.2, 91.5	86.4
	6	88.6	77.6, 92.2, 94.5	88.1
	12	92.0	90.2, 95.7, 104	96.5
Gin trash				
Dinotefuran	1	114	92.3, 94.7, 95.1	94.0
	3	109	92.2, 92.9, 94.3	93.1
	6	101	84.3, 86.3, 107	92.5
	12	106	104, 104	104
DN	1	79.6	75.7, 78.5, 81.8	78.6
	3	102	95.3, 104, 105	101
	6	73.4	77.6, 87.2, 90.4	85.1
	12	85.6	94.2, 94.2, 96.7	95.0
UF	1	97.5	86.0, 88.6, 95.7	90.1
	3	106	103, 103, 105	104
	6	108	85.7, 101, 104	96.8
	12	94.2	87.6, 92.5, 104	94.6
Meal				
Dinotefuran	1	99.7	94.8, 101, 101	98.9
	3	97.1	83.1, 88.9	86.0
	6	77.6	90.3, 100	95.3
	12	110	109, 114	112
DN	1	81.9	74.8, 82.2, 86.3	81.1
	3	75.2	73.4, 76.9, 82.7	77.6
	6	84.8	89.0, 95.6, 98.5	94.4
	12	82.7	88.5, 90.0, 92.3	90.3
UF	1	102	95.2, 96.4, 102	97.9
	3	101	72.0, 95.4, 103	90.2
	6	78.8	85.2, 90.3, 91.7	89.1
	12	107	107, 110, 114	110
Hulls				
Dinotefuran	1	87.0	88.0, 90.2, 90.4	89.5
	3	89.0	82.3, 84.6, 87.2	84.7
	6	76.6	73.5, 75.8, 77.9	75.7
	12	88.5	80.4, 94.9	87.6
DN	1	70.7	71.1, 71.4, 71.6	71.3
	3	90.8	86.0, 88.6, 107	93.9
	6	78.5	81.6, 90.8, 104	91.9
	12	76.5	70.7, 74.6	72.7
UF	1	87.0	89.1, 89.6, 90.7	89.8
	3	85.7	78.1, 83.0, 86.4	82.5
	6	75.2	67.8, 76.0	71.9
	12	82.8	72.7, 80.8	76.7
Oil				
Dinotefuran	1	96.8	83.7, 94.4	94.9
	3	101	91.6, 96.0, 104	97.1
	6	99.6	81.8, 87.1, 98.9	89.3
	12	107	105, 106, 106	106
DN	1	102	75.1, 82.4	78.8
	3	81.3	85.3, 86.5, 89.4	87.1
	6	74.4	86.2, 96.9, 99.9	90.9
	12	90.1	99.9, 101, 103	101

Storage interval (months)		% Recovery (0.10 mg/kg fortification for grain, bran and hulls, 1.0 mg/kg for straw)		
		Procedural	% remaining	Mean
UF	1	87.5	79.0, 89.8	84.4
	3	96.5	81.2, 88.6, 102	90.6
	6	78.0	82.3, 93.4, 102	92.7
	12	95.8	101, 101, 103	101

The storage stability testing of dinotefuran and its metabolites residues in peaches was performed after 787 days of storage (Dorschner, 2011: IR-4 09548). The samples were fortified with dinotefuran, UF and DN at 0.5 mg/kg, respectively. The fortified samples were stored frozen (generally -20 ºC) until being analysed.

Table 37 Recovery of dinotefuran, UF and DN from stored fortified samples of peach

Storage period 787 days	% Recovery (0.5 mg/kg fortification)		
	Concurrent	% remaining	Mean
Dinotefuran	101	103, 104, 104	104
UF	111	101, 103, 105	103
DN	94	92, 93, 94	93

The stability of residues, under freezer storage conditions, was assessed by fortification of untreated grape matrix (control) at a concentration of 1.0 mg/kg for dinotefuran, UF and DN (Hattermann, 2003: 43403A018). Control samples of grape were fortified and analysed following freezer storage at three intervals including the day of preparation (Day 0) and two subsequent intervals (Day 51 and 139).

Table 38 Recovery of dinotefuran, UF and DN from stored fortified samples of grape

Storage interval (days)		% Recovery (1.0 mg/kg fortification)		
		Procedural	% remaining	Mean
Dinotefuran	0	98.2, 102		
	51	97.0, 97.1	94.3, 97.7	96.0
	139	103, 103	96.5, 102	99.3
UF	0	99.5, 102		
	51	98.2, 98.8	98.3, 98.3	98.3
	139	109, 109	99.5, 104	102
DN	0	83.8, 83.9		
	51	89.6, 89.8	87.1, 89.5	88.3
	139	95.3, 95.4	89.8, 96.9	93.4

The storage stability of dinotefuran and its metabolites residues in cranberries was investigated by fortifying with dinotefuran at 0.10 mg/kg, UF at 0.095 mg/kg, and DN at 0.10 mg/kg (Samoil, 2010: IR-4 09832). The storage stability samples were held in frozen storage under similar conditions to the field generated samples. After 286 days of freezer storage, the storage stability sample were analysed for dinotefuran and its metabolites.

Table 39 Recovery of dinotefuran, UF and DN from stored fortified samples of cranberry

Storage period 286 days	% Recovery (0.10 mg/kg for dinotefuran and DN, 0.095 mg/kg for UF)		
	Concurrent	% remaining	Mean
Dinotefuran	98	96, 99, 102	99
UF	101	94, 98, 99	97
DN	86	88, 89, 90	89

The storage stability of dinotefuran and its metabolites was tested by fortifying onion (dry bulb) and analysing the samples after 749 days of frozen storage (Leonard, 2010: IR-4 08645). Samples of onion were fortified separately at 0.1 mg/kg dinotefuran, UF and DN.

Table 40 Recovery of dinotefuran, UF and DN from stored fortified samples of onion (dry bulb)

Storage period 749 days	% Recovery (Spike level: 0.10 mg/kg, Storage condition: < -20 ºC)		
	Concurrent	% remaining	Mean
Dinotefuran	107	109, 110, 112	110
UF	117	105, 107, 108	107
DN	93	90, 91, 91	90

The storage stability of dinotefuran and its metabolites was tested by fortifying green onion and analysing the samples after 679 days of frozen storage (Leonard, 2010: IR-4 09550). Storage stability samples were fortified at 0.1 mg/kg dinotefuran, 0.08 mg/kg for UF and 0.10 mg/kg for DN.

Table 41 Recovery of dinotefuran, UF and DN from stored fortified samples of green onion

Storage period 679 days	% Recovery (Spike level: 0.10 and 0.08 mg/kg, Storage condition: -40 to -6 ºC)		
	Concurrent	% remaining	Mean
Dinotefuran	98	73, 90, 90	84
UF	93	82, 84, 86	84
DN	86	72, 95, 101	89

Stability of residues under freezer storage conditions was assessed by fortification of untreated broccoli matrix (control) at a concentration of 1.0 mg/kg for dinotefuran, UF and DN (Hummel, 2003: 43414A013). Control samples of broccoli, a representative commodity of Brassica vegetables, were fortified and analysed following freezer storage at three intervals including the day of preparation (Day 0) and two subsequent intervals (Day 51 and 139).

Table 42 Recovery of dinotefuran, UF and DN from stored fortified samples of broccoli

Storage interval (days)		% Recovery (1.0 mg/kg fortification)		
		Procedural	% remaining	Mean
Dinotefuran	0	94.4, 98.1		
	51	93.9, 99.9	90.6, 91.3	91.0
	139	92.0, 97.0	91.5, 91.8	91.7
UF	0	91.3, 95.7		
	51	94.0, 100	91.7, 93.7	92.7
	139	100, 101	99.6, 99.8	99.7
DN	0	84.5, 86.9		
	51	92.5, 98.5	90.9, 93.8	92.4
	139	92.5, 97.6	96.7, 97.3	97.0

Stability of residues under freezer storage conditions was assessed by fortification of untreated melon matrix (control) at a concentration of 1.0 mg/kg for dinotefuran, UF and DN (Hummel, 2003: 43413A014). Control samples of melon, a representative commodity of cucurbit vegetables, were fortified and analysed following freezer storage at a minimum of three intervals including the day of preparation (Day 0) and two subsequent intervals (Day 53 and 161).

Table 43 Recovery of dinotefuran, UF and DN from stored fortified samples of melon

Storage interval (days)		% Recovery (1.0 mg/kg fortification)		
		Procedural	% remaining	Mean
Dinotefuran	0	102, 107		
	53	96.5, 109	109, 114	112
	161	101, 104	97.9, 100	99.0
UF	0	107, 110		
	53	91.6, 93.7	91.4, 102	96.7
	161	104, 105	102, 104	103
DN	0	98.2, 105		
	53	104, 111	104, 105	105
	161	96.3, 96.6	90.2, 93.6	91.9

Stability of residues under freezer storage conditions was assessed by fortification of untreated tomato matrix (control) at a concentration of 1.0 mg/kg for dinotefuran, UF and DN

(Hummel, 2003: 43415A010). Control samples of tomato, representative of both tomato and pepper, were fortified and analysed following freezer storage at three intervals including the day of preparation (Day 0) and two subsequent intervals (Day 53 and 161).

Table 44 Recovery of dinotefuran, UF and DN from stored fortified samples of tomato

Storage interval (days)		% Recovery (1.0 mg/kg fortification)		
		Procedural	% remaining	Mean
Dinotefuran	0	108, 113		
	53	101, 111	106, 107	107
	161	99.0, 99.2	90.5, 102	96.3
UF	0	105, 111		
	53	107, 107	98.6, 106	102
	161	103, 104	97.3, 106	102
DN	0	98.6, 102		
	53	108, 109	97.3, 107	102
	161	95.2, 99.1	90.6, 93.7	92.2

Stability of residues under freezer storage conditions was assessed by fortification of untreated lettuce matrix (control) at a concentration of 1.0 mg/kg for dinotefuran, UF and DN (Hummel, 2003: 43409A015). Control samples of lettuce were fortified and analysed following freezer storage at a minimum of three intervals including the day of preparation (Day 0) and two subsequent intervals (Day 53 and 161). The initial analysis set consisted of a control and two samples fortified at 1.0 mg/kg with each analyte.

Table 45 Recovery of dinotefuran, UF and DN from stored fortified samples of lettuce

Storage interval (days)		% Recovery (1.0 mg/kg fortification)		
		Procedural	% remaining	Mean
Dinotefuran	0	91.9, 110		
	53	88.5, 88.9	92.4, 96.3	94.4
	161	99.7, 102	97.0, 99.0	98.0
UF	0	107, 107		
	53	96.2, 97.7	89.4, 91.4	90.4
	161	105, 105	101, 104	103
DN	0	97.4, 98.7		
	53	98.2, 109	94.8, 100	97.4
	161	95.0, 96.0	94.1, 95.0	94.6

The storage stability of dinotefuran and its metabolites was tested by fortifying watercress and analysing the samples after 608 days of frozen storage (Dorschner, 2009: IR-4 09514). Samples of onion were fortified separately at 0.10 mg/kg dinotefuran, UF and DN.

Table 46 Recovery of dinotefuran, UF and DN from stored fortified samples of watercress

Storage period 608 days	% Recovery (Spike level: 0.10 mg/kg, Storage condition: < -20 ºC)		
	Concurrent	% remaining	Mean
Dinotefuran	90	86, 90, 96	91
UF	93	81, 85, 88	85
DN	97	90, 94, 96	93

Stability of residues under freezer storage conditions was assessed by fortification of untreated potato matrix (control) at a concentration of 1.0 mg/kg for dinotefuran, UF and DN. Control samples of potatoes were fortified and analysed following freezer storage at three intervals including the day of preparation (Day 0) and two subsequent intervals (Day 51 and 139).

Table 47 Recovery of dinotefuran, UF and DN from stored fortified samples of potato

Storage interval (days)		% Recovery (1.0 mg/kg fortification)		
		Procedural	% remaining	Mean
Dinotefuran	0	100, 102		
	51	97.7, 101	95.2, 96.0	95.6

Storage interval (days)		% Recovery (1.0 mg/kg fortification)		
		Procedural	% remaining	Mean
	139	104, 104	101, 104	103
UF	0	99.8, 102		
	51	99.4, 100	96.7, 97.3	97.0
	139	105, 107	107, 107	107
DN	0	84.1, 93.1		
	51	95.7, 95.7	92.3, 93.8	93.1
	139	90.5, 96.2	90.7, 96.3	93.5

The stability study of dinotefuran, UF and DN was conducted in bovine tissues (liver, kidney, muscle and fat) when stored at approximately -20 ºC for periods of up to 60 days and in bovine milk and cream, and hen eggs at approximately +4 ºC for periods of up to 4 days. Untreated sub-samples of bovine tissues (liver, kidney, muscle and fat) were fortified at 1.0 mg/kg and analysed immediately (day 0) and after 30 and 60 days freezer storage. Untreated sub-samples of homogenised bovine milk and cream, and hen eggs were fortified at 1.9 mg/kg (mg/L for milk) and analysed immediately (day 0) and after 2 and 4 days freezer storage.

Table 48 Recovery of dinotefuran, DN and UF from stored fortified samples of bovine tissues

Storage interval (days)		Recovery (fortification level: 1.0 mg/kg, Storage condition: -20 ºC)		
		Procedural (%)	Stored sample (mg/kg)	Mean (%)
Liver				
Dinotefuran	0		0.72, 0.72	72
	30	101	1.03, 1.03	103
	60	107	1.00, 1.05	103
UF	0		0.77, 0.79	78
	30	100	0.90, 0.97	94
	60	89	1.02, 1.03	103
DN	0		0.79, 0.91	85
	30	92	1.01, 1.04	103
	60	81	0.73, 0.92	83
Kidney				
Dinotefuran	0		0.79, 0.83	81
	30	104	0.80, 0.90	85
	60	86	0.71, 0.78	75
UF	0		0.72, 0.74	73
	30	104	1.00, 1.04	102
	60	85	0.79, 0.89	84
DN	0		0.85, 0.86	86
	30	88	0.83, 0.99	91
	60	101	0.75, 0.84	80
Muscle				
Dinotefuran	0		0.81, 0.84	83
	30	98	0.93, 0.95	94
	60	75	0.71, 0.86	79
UF	0		0.78, 0.82	80
	30	95	0.84, 0.91	88
	60	83	0.78, 0.86	82
DN	0		0.75, 0.76	76
	30	97	1.00, 1.01	101
	60	106	0.74, 0.91	83
Fat				
Dinotefuran	0		0.73, 0.81	77
	30	86	0.70, 0.75	73
	60	73	0.71, 0.82	77
UF	0		0.72, 0.74	73
	30	77	0.77, 0.80	79
	60	71	0.71, 0.73	72
DN	0		0.70, 0.70	70
	30	74	0.74, 0.89	82
	60	102	0.89, 0.90	90

Table 49 Recovery of dinotefuran, DN and UF from stored fortified samples of bovine milk, cream and hen eggs

Storage interval (days)		Recovery (fortification level: 1.0 mg/kg, Storage condition: +4 ºC)		
		Procedural (%)	Stored sample (mg/kg)	Mean (%)
Milk				
Dinotefuran	0		0.82, 1.00	91
	2	109	1.06, 1.09	108
	4	108	0.81, 0.90	86
UF	0		0.89, 1.02	96
	2	109	1.07, 1.08	108
	4	100	0.70, 0.73	72
DN	0		0.93, 1.07	100
	2	107	0.91, 0.97	94
	4	109	0.70, 0.71	71
Cream				
Dinotefuran	0		0.89, 1.10	100
	2	101	0.98, 1.08	103
	4	75	1.08, 1.10	109
UF	0		0.98, 1.07	103
	2	84	0.82, 1.02	92
	4	110	1.09, 1.10	110
DN	0		0.84, 1.09	97
	2	98	0.80, 0.85	83
	4	110	0.72, 1.06	89
Eggs				
Dinotefuran	0		0.75, 1.00	88
	2	93	0.91, 0.95	93
	4	76	0.74, 0.74	74
UF	0		0.91, 0.91	91
	2	76	0.60, 0.85	73
	4	75	0.72, 0.96	84
DN	0		0.77, 0.94	86
	2	97	0.93, 0.97	95
	4	93	0.74, 0.75	75

USE PATTERN

Dinotefuran is registered in some countries for the control of biting and sucking insects on fruits, vegetables, cereals and oilseed etc. It is applied as foliar and soil treatment (irrigation). The Meeting received labels in Japan, Korea and the USA. The information available to Meeting on registered uses of dinotefuran is summarized in the Table below.

Table 50 Registered uses of dinotefuran relevant to the review

Crop	Country	Formulation		Application					PHI days
		Type	Conc. of dinotefuran	Method	Rate kg ai/ha	Volume L/ha	Spray conc. kg ai/hl	Number max	
Stone fruits									
Peach Nectarine	Japan	SG	200 g/kg	Foliar		2000–7000	0.010	3	1
Peach Nectarine	USA	SG	200 g/kg	Foliar	0.10–0.20	> 46.8 (air) > 468 (ground)	Do not apply more than a total of 0.30 kg ai/ha per season		3
				Soil treatment	0.30	> 935	Do not apply more than a total of 0.30 kg ai/ha per season		21

Crop	Country	Formulation		Application					PHI days
		Type	Conc. of dinotefuran	Method	Rate kg ai/ha	Volume L/ha	Spray conc. kg ai/hl	Number max	
				Regardless of application method, do not exceed 0.40 kg ai/ha per season					
Peach Nectarine	USA	SG	700 g/kg	Foliar	0.10–0.20	> 46.8 (air) > 468 (ground)	Do not apply more than a total of 0.30 kg ai/ha per season		3
				Soil treatment	0.30	> 935	Do not apply more than a total of 0.30 kg ai/ha per season		21
				Regardless of application method, do not exceed 0.39 kg ai/ha per season					
Berries and other small fruits									
Grape	Japan	SG	200 g/kg	Foliar		2000–7000	0.0067–0.020	2	7
				Paint on the surface of trunk	20–40 g/tree		20	1	30
Grape	Korea	WG	200 g/kg	Foliar			0.010	2	21
Grape	USA	SG	200 g/kg	Foliar	0.050–0.15	28–94 (air) 94–467 (ground)	Do not apply more than a total of 0.30 kg ai/ha per season		1
				Soil treatment	0.25–0.37			1	28
				Regardless of application method, do not exceed 0.59 kg ai/ha per season					
Berry and Small fruit Subgroup: Small fruit vine climbing, except Fuzzy kiwifruit	USA	SG	200 g/kg	Foliar	0.050–0.15	47–94 (air) 467–2802 (ground)	Do not apply more than a total of 0.30 kg ai/ha per season		1
				Soil treatment	0.25–0.38	94–935		1	28
				Regardless of application method, do not exceed 0.61 kg ai/ha per season					
	USA	SG	700 g/kg	Foliar	0.050–0.15	47–94 (air) 467–2802 (ground)	Do not apply more than a total of 0.30 kg ai/ha per season		1
				Soil treatment	0.25–0.38	94–935		1	28
				Regardless of application method, do not exceed 0.59 kg ai/ha per season					
Berry and Small fruit Subgroup: Low growing berry subgroup, except strawberry	USA	SG	200 g/kg	Foliar	0.10–0.20	> 47 (air) > 280 (ground)	Do not apply more than a total of 0.40 kg ai/ha per season		7
	USA	SG	700 g/kg	Foliar	0.10–0.20	> 47 (air) > 280 (ground)	Do not apply more than a total of 0.40 kg ai/ha per season		7
Bulb vegetables									
Onion, Bulb and Green	USA	SG	200 g/kg	Foliar	0.10–0.20	> 47 (air) > 187 (ground)	Do not apply more than a total of 0.30 kg ai/ha per season		1
				Soil treatment	0.25–0.30	> 94	Do not apply more than a total of 0.30 kg ai/ha per season		at planting
				Regardless of application method, do not exceed 0.43 kg ai/ha per season					
	USA	SG	700 g/kg	Foliar	0.10–0.20	> 47 (air) > 187 (ground)	Do not apply more than a total of 0.30 kg ai/ha per season		1

Crop	Country	Formulation		Application					PHI days
		Type	Conc. of dinotefuran	Method	Rate kg ai/ha	Volume L/ha	Spray conc. kg ai/hl	Number max	
				Soil treatment	0.25–0.30	> 94	Do not apply more than a total of 0.30 kg ai/ha per season		at planting
				Regardless of application method, do not exceed 0.42 kg ai/ha per season					
Green Onion	Japan	SG	200 g/kg	Foliar		1000–3000	0.010	2	3
				Irrigation		0.5 L/ nursery box	0.40	1	at planting
						0.4 L/m^2	0.05	1	14
Brassica (cole or cabbage) vegetables, Head cabbages, Flowerhead cabbages									
Broccoli	Japan	SG	200 g/kg	Foliar		1000–3000	0.0067–0.010	2	3
				Irrigation		0.5 L/ nursery box	0.20	1	at planting
Broccoli	Korea	WG	200 g/kg	Irrigation			0.20	1	Before transplanting
Cabbage	Japan	SG	200 g/kg	Foliar		1000–3000	0.0067–0.010	2	3
				Irrigation		0.5 L/ nursery box	0.20–0.40	1	at planting
Cabbage	Korea	WG	200 g/kg	Irrigation		10 mL/plant (nursery box)	0.20	1	Before transplanting
Chinese Cabbage	Japan	SG	200 g/kg	Foliar		1000–3000	0.0067–0.010	2	3
Chinese cabbage	Korea	WG	200 g/kg	Foliar			0.010	2	7
				Irrigation		10 mL/plant (nursery box)	0.20	1	Before transplanting
	Korea	WP	100 g/kg	Irrigation		4 mL/plant (nursery box)	0.20	1	Before transplanting
Head and Stem Brassica	USA	SG	200 g/kg	Foliar	0.050–0.20	28–94 (air) 187–374 (ground)	Do not apply more than a total of 0.30 kg ai/ha per season		1
				Soil treatment	0.25–0.37		Do not apply more than a total of 0.60 kg ai/ha per season		21
	USA	SG	700 g/kg	Foliar	0.050–0.20	28–94 (air) 187–374 (ground)	Do not apply more than a total of 0.30 kg ai/ha per season		1
				Soil treatment	0.25–0.37	94–935	Do not apply more than a total of 0.60 kg ai/ha per season		21
Fruiting vegetables, Cucurbits									
Bitter gourd	Japan	SG	200 g/kg	Foliar		1000–3000	0.010	2	1
Cucumber	Japan	SG	200 g/kg	Foliar		1000–3000	0.0067–0.010	2	1
Cucumber	Korea	WG	200 g/kg	Foliar			0.010–0.020	3	2
	Korea	WP	100 g/kg	Foliar			0.010	2	2
				Irrigation		4 mL/plant (nursery box)	0.20	1	Before transplanting
Cucurbits for pickles	Japan	SG	200 g/kg	Foliar		1000–3000	0.0067–0.010	2	7
Melons	Japan	SG	200 g/kg	Foliar		1000–3000	0.0067–0.010	2	3

Crop	Country	Formulation		Application					PHI days
		Type	Conc. of dinotefuran	Method	Rate kg ai/ha	Volume L/ha	Spray conc. kg ai/hl	Number max	
Oriental melon	Japan	SG	200 g/kg	Foliar		1000–3000	0.0067	1	3
Oriental melon	Korea	WG	200 g/kg	Foliar			0.010	2	7
Pumpkins	Japan	SG	200 g/kg	Foliar		1000–3000	0.010	2	1
Squash (including pumpkin)	Korea	WP	100 g/kg	Foliar			0.010	3	3
Watermelon	Japan	SG	200 g/kg	Foliar		1000–3000	0.0067–0.010	2	7
Watermelon	Korea	WG	200 g/kg	Foliar			0.010	3	7
	Korea	WP	100 g/kg	Foliar			0.010	3	7
Cucurbits	USA	SG	200 g.kg	Foliar	0.050–0.20	28–94 (air) 187–374 (ground)	Do not apply more than a total of 0.30 kg ai/ha per season		1
				Soil treatment	0.25–0.37		Do not apply more than a total of 0.60 kg ai/ha per season		21
	USA	SG	700 g.kg	Foliar	0.050–0.20	28–94 (air) 187–374 (ground)	Do not apply more than a total of 0.30 kg ai/ha per season		1
				Soil treatment	0.25–0.37	94–935	Do not apply more than a total of 0.60 kg ai/ha per season		21
Fruiting vegetables, other than Cucurbits									
Chili peppers	Japan	SG	200 g/kg	Foliar		1000–3000	0.0067–0.010	2	1
Chili pepper (including sweet pepper)	Korea	SL	100 g/L	Foliar			0.010	3	2
	Korea	WG	200 g/kg	Foliar			0.010	3	5
				Irrigation		10 mL/plant (nursery box)	0.20	1	Before transplanting
	Korea	WP	100 g/kg	Foliar			0.010	3	3
				Irrigation		8 mL/plant (nursery box)	0.20	1	Before transplanting
Egg plant	Japan	SG	200 g/kg	Foliar		1000–3000	0.0067–0.010	2	1
Tomato, Cherry tomato	Japan	SG	200 g/kg	Foliar		1000–3000	0.0067–0.010	2	1
				Irrigation		0.5 L/ nursery box	0.20	1	at planting
Tomato	Korea	WG	200 g/kg	Foliar			0.010	3	2
Tomato (including cherry tomato)	Korea	WP	100 g/kg	Foliar			0.010	2	2
Okra	Japan	SG	200 g/kg	Foliar		1000–3000	0.010	2	1
Paprika	Korea	WG	200 g/kg	Irrigation		150 mL/ plant (nursery box)	0.0033	2	7
Sweet pepper	Japan	SG	200 g/kg	Foliar		1000–3000	0.0067–0.010	2	1
Sweet pepper	Korea	WP	100 g/kg	Irrigation		150 mL/ plant (nursery box)	0.0033	2	30

Crop	Country	Formulation		Application					PHI days
		Type	Conc. of dinotefuran	Method	Rate kg ai/ha	Volume L/ha	Spray conc. kg ai/hl	Number max	
Fruiting vegetables Do not apply to varieties of tomatoes which are less than 2 inches in size, such as cherry or grape tomatoes.	USA	SG	200 g/kg	Foliar	0.050–0.20	28–94 (air) 187–374 (ground)	Do not apply more than a total of 0.30 kg ai/ha per season		1
				Soil treatment	0.25–0.37		Do not apply more than a total of 0.60 kg ai/ha per season		21
	USA	SG	700 g.kg	Foliar	0.050–0.20	28–94 (air) 187–374 (ground)	Do not apply more than a total of 0.30 kg ai/ha per season		1
				Soil treatment	0.25–0.37	94–935	Do not apply more than a total of 0.60 kg ai/ha per season		21
Leafy vegetables (including Brassica leafy vegetables)									
Komatsuna	Japan	SG	200 g/kg	Foliar		1000–3000	0.0067–0.010	2	14
Lettuce, Head	Japan	SG	200 g/kg	Foliar		1000–3000	0.0067–0.010	2	3
				Irrigation		0.5 L/ nursery box	0.20–0.40	1	at planting
Lettuce, Leaf	Japan	SG	200 g/kg	Foliar		1000–3000	0.0067–0.010	2	7
				Irrigation		0.5 L/ nursery box	0.20–0.40	1	at planting
Mizuna	Japan	SG	200 g/kg	Foliar		1000–3000	0.0067–0.010	2	7
Pak-choi	Japan	SG	200 g/kg	Foliar		1000–3000	0.0067–0.010	2	3
Spinach	Japan	SG	200 g/kg	Foliar		1000–3000	0.0067	2	3
Watercress	Japan	SG	200 g/kg	Foliar		1000–3000	0.0067	3	3
Watercress	USA	SG	200 g/kg	Foliar	0.10–0.20	47–94 (air) 468–2806 (ground)	Do not apply more than a total of 0.40 kg ai/ha per season		1
	USA	SG	700 g/kg	Foliar	0.10–0.20	47–94 (air) 468–2806 (ground)	Do not apply more than a total of 0.40 kg ai/ha per season		1
Leafy Brassica Greens	USA	SG	200 g/kg	Foliar	0.099–0.15	28–94 (air) 187–374 (ground)	Do not apply more than a total of 0.30 kg ai/ha per season		1
	USA	SG	700 g/kg	Foliar	0.099–0.15	28–94 (air) 187–374 (ground)	Do not apply more than a total of 0.30 kg ai/ha per season		1
Leafy vegetables	USA	SG	200 g/kg	Foliar	0.050–0.15	28–94 (air) 187–374 (ground)	Do not apply more than a total of 0.30 kg ai/ha per season		7
				Soil treatment	0.25–0.37		Do not apply more than a total of 0.60 kg ai/ha per season		21
	USA	SG	700 g/kg	Foliar	0.050–0.15	28–94 (air) 187–374 (ground)	Do not apply more than a total of 0.30 kg ai/ha per season		7

Crop	Country	Formulation		Application					PHI days
		Type	Conc. of dinotefuran	Method	Rate kg ai/ha	Volume L/ha	Spray conc. kg ai/hl	Number max	
				Soil treatment	0.25–0.37	94–935	Do not apply more than a total of 0.60 kg ai/ha per season		21
Root and tuber vegetables									
Carrot	Japan	SG	200 g/kg	Foliar		1000–3000	0.010	2	30
				Irrigation		0.4 L/m^2	0.050	1	45
Ginseng	Korea	WG	200 g/kg	Foliar			0.010	3	7
Potato	Japan	SG	200 g/kg	Foliar		1000–3000	0.010	2	7
Potato	Korea	WG	200 g/kg	Foliar			0.010	2	14
Potato	USA	SG	200 g/kg	Foliar	0.056–0.074	28–94 (air) 94-467 (ground)	Do not apply more than a total of 0.22 kg ai/ha per season		7
				Soil treatment	0.31–0.37			1	at pre-plant
Radish	Japan	SG	200 g/kg	Foliar		1000–3000	0.0067	2	7
Sugar beet	Japan	SG	200 g/kg	Foliar		1000–3000	0.0067–0.020	2	7
				Irrigation		2.5–3.0 L/m^2	0.083–0.20	1	Before trans–planting
Turnip	Japan	SG	200 g/kg	Foliar		1000–3000	0.0067	2	3
Tuberous and Corm vegetables	USA	SG	200 g/kg	Foliar	0.050–0.076	28–94 (air) 94–468 (ground)	Do not apply more than a total of 0.23 kg ai/ha per season		7
				Soil treatment	0.33–0.38	94–935	Do not apply more than a total of 0.38 kg ai/ha per season		at pre-plant
	USA	SG	700 g/kg	Foliar	0.050–0.076	28–94 (air) 94–468 (ground)	Do not apply more than a total of 0.23 kg ai/ha per season		7
				Soil treatment	0.33–0.38	94–935	Do not apply more than a total of 0.38 kg ai/ha per season		at pre-plant
Stalk and stem vegetables									
Celery	Japan	SG	200 g/kg	Foliar		1000–3000	0.010	2	14
Fuki	Japan	SG	200 g/kg	Foliar		1000–3000	0.010	2	7
Cereal grains									
Rice	Japan	SG	200 g/kg	Foliar		600–1500	0.0067–0.010	3	7
Rice	Korea	WG	200 g/kg	Irrigation		0.5 L/ nursery box	0.20	1	Before planting
	Korea	WP	100 g/kg	Foliar			0.010	3	14
	Korea	SL	100 g/L	Foliar			0.010	3	14
						8	1.25		
						30	0.33		
Rice	USA	SG	200 g/kg	Foliar	0.11–0.15			2	7
Oilseed									
Cotton	USA	SG	200 g/kg	Foliar	0.050–0.15	28–94 (air) 94–468 (ground)	Do not apply more than a total of 0.30 kg ai/ha per season		14
	USA	SG	700 g/kg	Foliar	0.050–0.15	28–94 (air) 94–468 (ground)	Do not apply more than a total of 0.30 kg ai/ha per season		14

The US uses for head and stem Brassica, cucurbits, fruiting vegetables, leafy vegetables and tuberous and corm vegetables specifies: 'Do not combine foliar applications with soil applications, or vice versa. Only use one application method'.

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

The Meeting received information on dinotefuran supervised field trials for the following crops.

Group	Commodity	Table No.
Stone fruits	Peach	51
Berries and other small fruits	Grape	52
	Cranberry	53
Bulb vegetables	Bulb Onion	54
	Green onion	55
Brassica vegetables	Broccoli and Cauliflower	56
	Cabbage	57
Fruiting vegetables, Cucurbits	Cucumber	58
	Melon	59
	Summer squash and Zucchini	60
Fruiting vegetables, other than Cucurbits	Pepper	61, 62
	Tomato	63, 64
Leafy vegetables	Lettuce	65
	Spinach	66
	Watercress	67
Root and tuber vegetables	Potato	68
Stalk and stem vegetables	Celery	69
Cereal grains	Rice	70
Oilseed	Cotton seed	71

Dinotefuran SG formulation was applied for foliar spray and soil treatment. Each of the field trial sites generally consisted of untreated control plot and treated plot. Application rates and spray concentrations have generally been rounded to two significant figures.

Residue values from the trials, which have been used for the estimation of maximum residue levels, STMRs and HRs, are underlined.

Laboratory reports included method validation with procedural recoveries from spiking at residue levels similar to those occurring in samples from the supervised trials. Date of analyses and duration of residue sample storage were also provided. Although trials included control plots, no control data are recorded in the tables except when residues were found in samples from control plots. Residue data are not corrected for percentage recovery.

Conditions of the supervised residue trials were generally well reported in detailed field reports. Most field reports provided data on the sprayers used, plot size, field sample size and sampling date.

The residue concentrations are reported for dinotefuran, UF and DN. Since the residue values were expressed as mg of the analyte/kg sample, UF and DN need to be converted into dinotefuran equivalent. The conversion factors are 1.3 (202.21/158.20 = 1.28) for UF and 1.3 (202.21/157.22 = 1.29) for DN. Residues of < LOQ for both analytes are not converted.

Total residues for estimation of STMRs are calculated by summing up the concentrations of dinotefuran, UF and DN. In case that the residues of dinotefuran were found at high levels, UF and DN were also detected and the ratio of UF and DN depends on the commodity.

The method for calculation of the total residues for plant commodities is illustrated below.

Dinotefuran	UF	DN	Total
0.14	0.016	0.010	0.17 (0.14 + 0.016 × 1.3 + 0.010 × 1.3)
0.056	0.011	< 0.01	0.080 (0.056 + 0.011 × 1.3 + 0.01)
0.051	< 0.01	< 0.01	0.071 (0.051 + 0.01 + 0.01)
< 0.01	< 0.01	< 0.01	< 0.03 (0.01 + 0.01 + 0.01)

Stone fruits

Peaches

The Meeting received 12 trials on peaches which were conducted in the USA (Dorschner, 2011: IR-4 09548). In each trial, the test substance was applied in two foliar directed applications of approximately 0.20 kg ai/ha each, for a total of approximately 0.40 kg ai/ha. The applications were made at 6- to 10-day intervals and timed so that mature peaches could be collected 2 to 3 days following the final application. Samples for decline determination were collected from the trial in North Carolina. In addition to the two foliar directed applications at the trials in California, an additional plot was treated with one soil directed application of approximately 0.30 kg ai/ha. The application was made 21 days prior to harvest.

The maximum storage interval for field-treated samples was 746 days (approximately 24 months). Storage stability testing was performed after 787 days of storage.

Table 51 Dinotefuran and metabolites residues on peaches from supervised trials in USA

Peaches country, year (variety)	Application				PHI Days	Residues, mg/kg			
	Form	kg ai/ha	water, L/ha	no.		Dinotefuran	UF	DN	Total
GAP, USA	SG SG	0.30 (soil) 0.20 (foliar)	> 935 > 46.8	[a] [b]	21 3				
USA, 2007 Lyons/NY (GloHaven)	SG	0.20 (foliar)	747 767	2	3	0.20, 0.21 mean 0.21	0.02, 0.02	0.01, 0.01	mean 0.25
USA, 2007 Bridgeton/NJ (Sentry)	SG	0.20 (foliar)	810 805	2	3	0.19, 0.20 mean 0.20	0.01, 0.01	< 0.01, 0.01	mean 0.23
USA, 2007 Bridgeton/NJ (Blake)	SG	0.20 (foliar)	857 575	2	3	0.24, 0.37 mean 0.31	0.02, 0.02	< 0.01, 0.02	mean 0.35
USA, 2007 Madera/CA (Springcrest)	SG	0.20 (foliar)	378 375	2	3	0.43, 0.50 mean 0.47	0.05, 0.05	0.03, 0.03	mean 0.57
	SG	0.30 (soil)	1170	1	21	0.01, 0.01 mean 0.01	< 0.01, < 0.01	< 0.01, < 0.01	mean 0.03
USA, 2007 Madera/CA (Angelos)	SG	0.20 (foliar)	705 707	2	3	0.09, 0.16 mean 0.13	< 0.01, < 0.01	< 0.01, < 0.01	mean 0.15
USA, 2007 Parlier/CA (Fairtime)	SG	0.20 (foliar)	869 879	2	3	0.17, 0.19 mean 0.18	< 0.01, < 0.01	< 0.01, < 0.01	mean 0.20
	SG	0.30 (soil)	1132	1	21	< 0.01, < 0.01	< 0.01, < 0.01	< 0.01, < 0.01	mean < 0.03
USA, 2007 Parlier/CA (Flavorcrest)	SG	0.20 (foliar)	666 647	2	3	0.17, 0.25 mean 0.21	< 0.01, < 0.01	< 0.01, < 0.01	mean 0.23
USA, 2007 Fennville/MI (Elberta)	SG	0.20 (foliar)	718 701	2	2	0.47, 0.54 mean 0.51	0.05, 0.06	0.03, 0.04	mean 0.64
USA, 2008 Jackson springs /NC (Contender)	SG	0.20 (foliar)	751 729	2	1 3 8 15	0.12, 0.20 mean 0.16 0.09, 0.13 mean 0.11 0.05, 0.13 mean 0.09 0.07, 0.07 mean 0.07	< 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	mean 0.18 mean 0.13 mean 0.11 mean 0.09
USA, 2008 Jackson springs	SG	0.20 (foliar)	686 738	2	3	0.22, 0.25 mean 0.24	0.01, 0.02	< 0.01, < 0.01	mean 0.28

Peaches country, year (variety)	Application				PHI Days	Residues, mg/kg			
	Form	kg ai/ha	water, L/ha	no.		Dinotefuran	UF	DN	Total
/NC (Emery)									
USA, 2008 Troy/TN (Red Skin)	SG	0.20 (foliar)	573 579	2	3	0.08, 0.10 mean 0.09	0.01, 0.01	< 0.01, < 0.01	mean 0.11
USA, 2007 Fredericksburg /TX (Red Globe)	SG	0.20 (foliar)	400 399	2	3	0.32, 0.38 mean 0.35	0.01, 0.01	< 0.01, < 0.01	mean 0.37

Analytical portion: Fruit without pits. Only the higher residue value was considered from trials conducted side-by side.

[a] Do not apply more than a total of 0.30 kg ai/ha per season

[b] Regardless of application method, do not exceed 0.40 kg ai/ha per season

Berries and other small fruits

Grapes

The study was conducted at seven trial locations in the major regions of commercial grape production in the USA (Hattermann, 2003: 43403A018). The 200 g/kg SG formulation was used in all of the trials. The 100 g/L SL formulation was also used in two of the trials in plots treated concurrently with the 200 g/kg SG formulation to determine if there were any residue differences between the two formulations. One soil application was made with the 200 g/kg SG formulation at a target rate of 0.30 kg ai/ha followed by two foliar applications at 0.15 kg ai/ha. For the 100 g/L SL formulation, two foliar applications were done at 0.15 kg ai/ha. The soil application was done 29 days prior to harvest while the foliar applications were done 14 days apart with the last application done 1 day prior to harvest. The soil applications were done by drip irrigation or directed soil applications. The total target application rates after all applications done was 0.60 kg ai/ha for the soil applications (200 g/kg SG formulation only) and 0.30 kg ai/ha for the foliar applications. The maximum storage interval for field-harvested samples was 117 days.

Six residue trials were established in typical grape growing areas in the USA, using commercially viable varieties of grapes (Steams, 2003: V-24862). The 200 g/kg SG formulation was applied in a three-application program. The first application was made to the soil at the base of the grapes, using either drip irrigation equipment or backpack sprayers directed in a 12–18" band at the base of grape vines, applying 0.30 kg ai/ha, approximately 29 days before normal harvest. The second and third applications were foliar applications, applying approximately 0.15 kg ai/ha and were made at 14 ± 2 day intervals, using airblast or backpack sprayers. At one trial location, an additional treated plot was established using 2× rates (0.60 kg ai/ha as a soil application followed by 2× ca. 0.30 kg ai/ha foliar applications). Grapes were sampled 1 day after the last application at each trial. In one trial, additional samples were taken at 0, 3 and 6 days after the application to determine the decline rate of dinotefuran.

Samples from this study were extracted for analysis within 194 days (79–194 days) of sampling, and were maintained under frozen storage conditions (approximately -20 °C) from sampling until analysis. Storage stability studies in grapes have demonstrated that dinotefuran, UF and DN are all stable in/on grapes with quantitative recovery at 139 days when stored under frozen conditions. The storage stability study in high water content commodities (peach and cranberry) also demonstrated that dinotefuran, UF and DN were stable after 286–787 days of freezer storage (-20 °C).

Table 52 Dinotefuran and metabolites residues on grapes from supervised trials in USA

Grapes country, year (variety)	Application				PHI Days	Residues, mg/kg			
	Form	kg ai/ha	water, L/ha	no.		Dinotefuran	UF	DN	Total
GAP, USA	SG	0.38 (soil) 0.15 (foliar)		1 [a]	28 1				

Grapes country, year (variety)	Application				PHI Days	Residues, mg/kg			
	Form	kg ai/ha	water, L/ha	no.		Dinotefuran	UF	DN	Total
Hattermann, 2003: 43403A018									
USA, 2002 Ephrata/WA (White Reisling)	SG	0.30 (soil) + 0.15 (foliar)	187 1496–1518	1+ 2	1	0.11, 0.13 mean 0.12	< 0.01, < 0.01	< 0.01, < 0.01	mean 0.14
USA, 2002 Richgrove/CA (Red Globe)	SG	0.30 (soil) + 0.15 (foliar)	6090 1852–1864	1+ 2	1	0.11, 0.11 mean 0.11	< 0.01, < 0.01	< 0.01, < 0.01	mean 0.13
USA, 2002 Dinuba/CA (Ruby Red Wine)	SG	0.30 (soil) + 0.15 (foliar)	6090 1870–1921	1+ 2	1	0.22, 0.23 mean 0.22	< 0.01, < 0.01	< 0.01, < 0.01	mean 0.24
USA, 2002 Plainview/CA (Crimson Grape)	SG	0.30 (soil) + 0.15 (foliar)	6090 1853–1858	1+ 2	1	0.18, 0.22 mean 0.20	< 0.01, < 0.01	< 0.01, < 0.01	mean 0.22
	SL	0.15 (foliar)	1851–1856	2	1	0.17, 0.17 mean 0.17	< 0.01, < 0.01	< 0.01, < 0.01	mean 0.19
USA, 2002 Terra Bella/CA (Emperor Table Grapes)	SG	0.30 (soil) + 0.15 (foliar)	186 800–814	1+ 2	1	0.082, 0.093 mean 0.087	< 0.01, < 0.01	< 0.01, < 0.01	mean 0.11
USA, 2002 Hart/CA (Crimson Seedless Grapes)	SG	0.30 (soil) + 0.15 (foliar)	187 819–823	1+ 2	1	0.16, 0.18 mean 0.17	< 0.01, < 0.01	< 0.01, < 0.01	mean 0.19
USA, 2002 Dundee/NY (Vidal Blanc)	SG	0.30 (soil) + 0.15 (foliar)	254 942	1+ 2	0 0.5 1 3 5 7 10 14 21 28 35	0.30 0.32 0.34, 0.37 mean 0.36 0.34 0.40 0.30 0.26 0.32 0.18 0.20 0.16	0.011 0.015 0.015, 0.016 0.025 0.033 0.031 0.031 0.037 0.033 0.029 0.039	< 0.01 < 0.01 < 0.01, < 0.01 0.014 0.017 0.017 0.017 0.016 0.010 0.011 < 0.01	0.32 0.35 mean 0.39 0.39 0.47 0.36 0.32 0.39 0.24 0.25 0.22
	SL	0.15 (foliar)	944	2	1	0.35, 0.42 mean 0.38	0.014, 0.018	< 0.01, < 0.01	mean 0.41
Steams, 2003: V-24862									
USA, 2002 Dundee/NY (DeChaunac)	SG	0.30 (soil) + 0.15 (foliar)	937–945	1+ 2	1	0.22, 0.31 mean 0.27	0.01, < 0.01	< 0.01, < 0.01	mean 0.29
USA, 2002 Terra Bella/CA (Thompson)	SG	0.30 (soil) + 0.15 (foliar)	945–955	1+ 2	1	0.09, 0.10 mean 0.10	< 0.01, < 0.01	< 0.01, < 0.01	mean 0.12
	SG	0.60 (soil) + 0.30 (foliar)	926–951	1+ 2	1	0.26, 0.32 mean 0.29	0.01, 0.02	< 0.01, < 0.01	mean 0.33
USA, 2002 Mecca/CA (Flame Seedless)	SG	0.30 (soil) + 0.15 (foliar)	932–933	1+ 2	1	0.26, 0.28 mean 0.27	0.01, 0.01	< 0.01, < 0.01	mean 0.29
USA, 2002 Gonzolas/CA (Chardonnay)	SG	0.30 (soil) + 0.15 (foliar)	912–930	1+ 2	0 1 3 6	0.44, 0.56 mean 0.50 0.44, 0.48 mean 0.46 0.49, 0.60 mean 0.55 0.37, 0.52	0.02, 0.04 0.03, 0.03 0.05, 0.04 0.03, 0.05	0.01, 0.02 0.02, 0.02 0.03, 0.04 0.02, 0.03	mean 0.57 mean 0.53 mean 0.67

Grapes country, year (variety)	Application				PHI Days	Residues, mg/kg			
	Form	kg ai/ha	water, L/ha	no.		Dinotefuran	UF	DN	Total
						mean 0.45			mean 0.54
USA, 2002 Weiser/ID (Concord)	SG	0.30 (soil) + 0.15 (foliar)	938–940	1+ 2	1	0.13, 0.18 mean 0.16	< 0.01, < 0.01	< 0.01, < 0.01	mean 0.18
USA, 2002 Ukiah/CA (Grenache)	SG	0.30 (soil) + 0.15 (foliar)	932–940	1+ 2	1	0.34, 0.69 mean 0.52	0.02, 0.03	< 0.01, 0.01	mean 0.57

Analytical portion: fruit

[a] Do not apply more than a total of 0.30 kg ai/ha per season. Regardless of application method, do not exceed 0.61 kg ai/ha per season

Cranberry

Residue data have been collected from five field trials located in the USA (Samoil, 2010: IR-4 09832). At each trial, two broadcast applications of the test substance were made 12–14 days apart. The application rate was 0.20 kg ai/ha per treatment with a total rate of 0.40 kg ai/ha per season. All applications were made using appropriate spray equipment, and the spray volume was sufficient to provide adequate dispersal of the test substance. The formulation used in the trials was 200 g/kg SG formulation. A non-ionic surfactant was included in the spray mixes in the Oregon and Wisconsin trials. Sampling started in the untreated control plot and ended in the treated plot. At all the field trials, samples were harvested 6–7 days after the last application.

The maximum storage interval for field-treated samples in the trials was 280 days. Storage stability samples were fortified with dinotefuran at 0.10 mg/kg, UF at 0.095 mg/kg, and DN at 0.10 mg/kg soon after the receipt of the samples. After 286 days of freezer storage, the storage stability samples were analysed for dinotefuran and its metabolites which were found to be stable.

Table 53 Dinotefuran and metabolites residues on cranberry from supervised trials in USA

Cranberry country, year (variety)	Application				PHI Days	Residues, mg/kg			
	Form	kg ai/ha	water, L/ha	no.		Dinotefuran	UF	DN	Total
GAP, USA	SG	0.10–0.20	> 280	[a]	7				
USA, 2008 Plymouth/MA (Stevens)	SG	0.20	514	2	6	0.01, 0.01 mean 0.01	< 0.01, < 0.01	< 0.01, < 0.01	mean 0.03
USA, 2008 Wareham/MA (Early Black)	SG	0.20	514	2	6	0.03, 0.05 mean 0.04	< 0.01, < 0.01	< 0.01, < 0.01	mean 0.06
USA, 2008 Langlois/OR (Pilgrim)	SG	0.20	570–588	2	7	0.04, 0.05 mean 0.05	< 0.01, < 0.01	< 0.01, 0.01	mean 0.07
USA, 2008 Warrens/WI (Stevens)	SG	0.20	635–654	2	6	0.06, 0.06 mean 0.06	< 0.01, < 0.01	0.02, 0.02	mean 0.10
USA, 2008 Warrens/WI (Ben Lear)	SG	0.20	654	2	6	0.04, 0.06 mean 0.05	< 0.01, < 0.01	< 0.01, 0.01	mean 0.07

Analytical portion: fruit

[a] Do not apply more than a total of 0.40 kg ai/ha per season

Bulb vegetables

Onion, Bulb

Eight field trials on onion (dry bulb) were conducted using a 200 g/kg SG formulation during the 2007, 2008 and 2009 growing seasons in the USA (Leonard, 2010: IR-4 08645). At each trial, there were a control and two treated plots. One treated plot received either one in-furrow soil application (if onion seed was planted) or one drench application (if transplants were used) of test substance at planting/transplanting of approximately 0.29 kg ai/ha. The in-furrow applications used 93.4–187 L/ha and the drench applications with 467–934 L/ha. The drench/in furrow application plots were harvested at normal onion harvest. The second treated plot received two foliar (broadcast or directed) applications at 7–8 day intervals of test substance each at approximately 0.20 kg ai/ha and timed to be applied at 8 days and 1 day prior to harvest. The foliar applications used 187–934 L/ha spray solution. Onion samples were harvested at 1 day (2days at Oregon site only) after the final application in the foliar application plots. Additionally, residue decline samples were taken at 0, 3 and 7 days after the second application in the foliar application plot.

The maximum storage interval for field crop samples in the trials was 695 days. Samples of onion fortified separately at 0.1 mg/kg dinotefuran, UF and DN and analysed after 749 days of frozen storage demonstrated stability.

Table 54 Dinotefuran and metabolites residues on onion (dry bulb) from supervised trials in USA

Onion country, year (variety)	Application				PHI Days	Residues, mg/kg			
	Form	kg ai/ha	water, L/ha	no.		Dinotefuran	UF	DN	Total
GAP, USA	SG	0.30 (soil) 0.20 (foliar)		[a] [b]	[a] 1				
USA, 2007 Freeville/NY (BGS 233)	SG	0.29 (drench)	873	1	96	< 0.01, < 0.01 mean < 0.01	< 0.01, < 0.01	< 0.01, < 0.01	mean < 0.03
	SG	0.20 (foliar)	273–278	2	1	0.01, 0.02 mean 0.02	< 0.01, < 0.01	< 0.01, < 0.01	mean 0.04
USA, 2007 Arlington/WI (Montero)	SG	0.39 (drench)	927	1	80	0.01, 0.01 mean 0.01	< 0.01, < 0.01	< 0.01, < 0.01	mean 0.03
	SG	0.20 (foliar)	508–531	2	1	< 0.01, < 0.01 mean < 0.01	< 0.01, < 0.01	< 0.01, < 0.01	mean < 0.03
USA, 2007 Weslaco/TX (Cougar)	SG	0.29 (drench)	484	1	71	0.01, 0.01 mean 0.01	< 0.01, < 0.01	< 0.01, < 0.01	mean 0.03
	SG	0.20 (foliar)	201	2	1	0.03, 0.04 mean 0.04	< 0.01, 0.01	< 0.01, < 0.01	mean 0.06
USA, 2009 Las Cruces/NM (Nu Mex Freedom)	SG	0.31 (in furrow)	176	1	145	< 0.01, < 0.01 mean < 0.01	< 0.01, < 0.01	< 0.01, < 0.01	mean < 0.03
	SG	0.20 (foliar)	289–332	2	1	< 0.01, < 0.01 mean ≤ 0.01	< 0.01, < 0.01	< 0.01, < 0.01	mean ≤ 0.03
USA, 2007 Holtville/CA (Serengeti)	SG	0.30 (drench)	144	1	156	< 0.01, < 0.01 mean < 0.01	< 0.01, < 0.01	< 0.01, < 0.01	mean < 0.03
	SG	0.20 (foliar)	511–513	2	1	0.05, 0.06 mean 0.06	< 0.01, 0.01	< 0.01, 0.01	mean 0.09
USA, 2007 Parma/ID (Vaquero)	SG	0.29 (in furrow)	141	1	147	< 0.01, < 0.01 mean < 0.01	< 0.01, < 0.01	< 0.01, < 0.01	mean < 0.03
	SG	0.20 (foliar)	280–281	2	1	< 0.01, < 0.01 mean ≤ 0.01	< 0.01, < 0.01	< 0.01, < 0.01	mean ≤ 0.03
USA, 2008 Salem/OR (Red Ball)	SG	0.30 (drench)	765	1	84	< 0.01, < 0.01 mean < 0.01	< 0.01, < 0.01	< 0.01, < 0.01	mean < 0.03

Onion country, year (variety)	Application				PHI Days	Residues, mg/kg			
	Form	kg ai/ha	water, L/ha	no.		Dinotefuran	UF	DN	Total
	SG	0.20 (foliar)	461–472	2	2	0.02, 0.02 mean 0.02	< 0.01, < 0.01	< 0.01, < 0.01	mean 0.04
USA, 2007 Parlier/CA (Candy)	SG	0.30 (in furrow)	169	1	145	< 0.01, < 0.01 mean < 0.01	< 0.01, < 0.01	< 0.01, < 0.01	mean < 0.03
	SG	0.20 (foliar)	284–286	2	0	< 0.01, < 0.01 mean < 0.01	< 0.01, < 0.01	< 0.01, < 0.01	mean < 0.03
					1	0.01, 0.02 mean 0.02	< 0.01, < 0.01	< 0.01, < 0.01	mean 0.04
					3	< 0.01, < 0.01 mean < 0.01	< 0.01, < 0.01	< 0.01, < 0.01	mean < 0.03
					7	< 0.01, < 0.01 mean < 0.01	< 0.01, < 0.01	< 0.01, ,0.01	mean < 0.03

Analytical portion: bulbs

[a] Do not apply more than a total of 0.30 kg ai/ha per season at planting

[b] Regardless of application method, do not exceed 0.43 kg ai/ha per season

Onion, green

Five trials were conducted using a 200 g/kg SG formulation during the 2006–2007 growing season in the USA (Leonard, 2010: IR-4 9550). At each trial, two foliar applications of approximately 0.20 kg ai/ha was made at a 7 day interval with the last application 1 day before harvest. For the Georgia site, there were two treated plots with one plot receiving two foliar treatments of approximately 0.20 kg ai/ha applied at a 7 day interval with the last application 1 day before harvest and the second plot receiving a single broadcast soil application of approximately 0.30 kg ai/ha immediately after planning. Green onion whole plant samples were collected 1 day after the final application at all sites and also at normal harvest for the Georgia site plot which was treated 1 day post planting and were analysed after. The roots and the leaf were trimmed with a knife before analysis.

The maximum storage interval for field-treated samples in the trials was 644 days. Storage stability samples were fortified at 0.10 mg/kg with dinotefuran, 0.08 mg/kg for UF and 0.10 mg/kg for DN, and were analysed after 679 days of frozen storage.

Table 55 Dinotefuran and metabolites residues on green onion from supervised trials in USA

Green onion country, year (variety)	Application				PHI Days	Residues, mg/kg			
	Form	kg ai/ha	water, L/ha	no.		Dinotefuran	UF	DN	Total
GAP, USA	SG	0.30 (soil) 0.20 (foliar)		[a] [b]	[a] 1				
USA, 2006 Charleston/SC (White Onion set)	SG	0.20 (foliar)	486–504	2	1	0.41, 0.64 mean 0.52	0.066, 0.12	0.21, 0.21	mean 0.91
USA, 2006 Tifton/GA (White Spear)	SG	0.30 (soil)	177	1	93	< 0.01, < 0.01	< 0.01, < 0.01	< 0.01, < 0.01	mean < 0.01
	SG	0.20 (foliar)	299	2	1	1.1, 1.5 mean 1.3	0.037, 0.058	0.047, 0.11	mean 1.5

Green onion country, year (variety)	Application				PHI Days	Residues, mg/kg			
	Form	kg ai/ha	water, L/ha	no.		Dinotefuran	UF	DN	Total
USA, 2006 Willard/OH (Ishikura Improved)	SG	0.20 (foliar)	411–495	2	1	0.084, 0.088 mean 0.086	< 0.01, < 0.01	0.021, 0.013	mean 0.12
USA, 2007 Weslaco/TX (Calera)	SG	0.20 (foliar)	346–355	2	1	1.6, 2.2 mean 1.9	0.13, 0.11	0.25, 0.20	mean 2.3
USA, 2006 Salinas/CA (Oasis)	SG	0.20 (foliar)	467–476	2	1	0.22, 0.23 mean 0.22	0.016, 0.020	0.27, 0.26	mean 0.59

[a] Do not apply more than a total of 0.30 kg ai/ha per season at planting

[b] Regardless of application method, do not exceed 0.43 kg ai/ha per season

Brassica (cole or cabbage) vegetables, Head cabbages, Flowerhead brassicas

The trials were conducted at 12 locations in the major regions of commercial broccoli, cauliflower and cabbage production in the USA (Hummel, 2003: 43414A013). Two foliar applications of the test substance (dinotefuran 200 g/kg SG formulation) were made at a foliar rate of 0.20 kg ai/ha in a calibrated volume of 205–347 L/ha to obtain the maximum labelled per season rate of 0.40 kg ai/ha. There were 7 day intervals between applications and 1 day between the last application and harvest (i.e. 1 day PHI). Two applications of the test substance were made to soil at a rate of 0.30 kg ai/ha to obtain the maximum labelled per season rate of 0.60 kg ai/ha. The first application was made within 3 days after planting, and the second 21–22 days prior to harvest. In addition, two trials included an additional plot treated with a 100 g/L SL formulation applied according to equivalent rate, timing and spray parameters compared to the foliar treatment of the SG formulation.

Stability of residues under freezer storage was assessed by fortification of untreated broccoli matrix (control) at a concentration of 1.0 mg/kg for dinotefuran, UF and DN. Control samples of broccoli were fortified and analysed following freezer storage at three intervals including the day of preparation (Day 0) and two subsequent intervals (Day 51 and 139).

Broccoli and Cauliflower

Samples were stored for varying lengths of time upon receipt at the analytical laboratory prior to analysis. All treated samples were analysed for dinotefuran, UF and DN within 165 days (44–149 days for broccoli and 2–165 days for cauliflower) after collection.

Table 56 Dinotefuran and metabolites residues on broccoli and cauliflower from supervised trials in the USA

country, year (variety)	Application				PHI Days	Residues, mg/kg			
	Form	kg ai/ha	water, L/ha	No.		Dinotefuran	UF	DN	Total
GAP, USA (Head and Stem Brassica)	SG	0.37 (soil) [a] 0.20 (foliar) [b]			21 1				
USA, 2002 Colony/OK Broccoli (Packman)	SG	0.30 (soil)		2	22	0.052, 0.067 mean 0.059	< 0.01, < 0.01	< 0.01, < 0.01	mean 0.079
	SG	0.20 (foliar)	205–219	2	1	0.97, 1.1 mean 1.0	0.010, 0.011	0.024, 0.029	mean 1.0
USA, 2002 King City/CA Broccoli (Patriot)	SG	0.20 (foliar)	278–283	2	0 1	0.81 0.34, 0.63 mean 0.49	0.021 0.014, 0.021	0.068 0.034, 0.044	0.93 mean 0.56

country, year (variety)	Application				PHI Days	Residues, mg/kg			
	Form	kg ai/ha	water, L/ha	No.		Dinotefuran	UF	DN	Total
					3	0.41	0.029	0.065	0.53
					5	0.31	0.022	0.050	0.40
					7	0.24, 0.24 mean 0.24	0.026, 0.033	0.084, 0.079	mean 0.38
					9	0.23	0.025	0.069	0.35
					11	0.15	0.022	0.062	0.26
					14	0.12	0.022	0.065	0.23
					21	0.041	0.018	0.066	0.15
					28	0.015	< 0.01	0.038	0.074
					35	0.025	0.013	0.062	0.12
USA, 2002 Porterville/CA Broccoli (Liberty)	SG	0.30 (soil)		2	21	< 0.01, < 0.01 mean < 0.01	< 0.01, < 0.01	< 0.01, < 0.01	mean < 0.03
	SG	0.20 (foliar)	285–288	2	1	0.90, 1.2 mean 1.0	0.032, 0.030	0.061, 0.068	mean 1.1
USA, 2002 Casa Grande /AZ Cauliflower (Quasar)	SG	0.20 (foliar)	286–290	2	1	0.083, 0.089 mean 0.086	< 0.01, < 0.01	< 0.01, < 0.01	mean 0.11
USA, 2002 Greenfield/CA Cauliflower (Apex)	SG	0.20 (foliar)	283–286	2	1	0.33, 0.38 mean 0.36	0.015, 0.013	0.026, 0.026	mean 0.41
USA, 2002 Vernon/WA Cauliflower (Ruvella)	SG	0.20 (foliar)	280–282	2	1	0.044, 0.067 mean 0.056	< 0.01, < 0.01	< 0.01, < 0.01	mean 0.076
	SL	0.20 (foliar)	281–282	2	1	0.34, 0.065 mean 0.20	< 0.01, < 0.01	< 0.01, < 0.01	mean 0.22

GAP information: Do not combine foliar applications with soil applications, or vice versa. Only use one application method.

[a] Do not apply more than a total of 0.60 kg ai/ha per season

[b] Do not apply more than a total of 0.30 kg ai/ha per season

Cabbages, Head

Samples were stored for varying lengths of time upon receipt at the analytical laboratory prior to analysis. All treated samples were analysed for dinotefuran, UF and DN within 110 days (14–110 days) after collection.

Table 57Dinotefuran and metabolites residues on cabbage from supervised trials in USA

Cabbage country, year (variety)	Application				PHI Days	Residues, mg/kg			
	Form	kg ai/ha	water, L/ha	no.		Dinotefuran	UF	DN	Total
GAP, USA (Head and Stem Brassica)	SG	0.37 (soil) [a] 0.20 (foliar) [b]			21 1				
USA, 2002 North Rose/NY	SG	0.30 (soil)		2	21	0.14, 0.20 mean 0.17	0.020, 0.048	0.057, 0.14	mean 0.34

Cabbage country, year (variety)	Application				PHI Days	Residues, mg/kg			
	Form	kg ai/ha	water, L/ha	no.		Dinotefuran	UF	DN	Total
(Blue Thunder)	SG	0.20 (foliar)	235–239	2	1	0.71, 1.0 mean 0.85	0.057, 0.10	0.077, 0.13	mean 1.1
USA, 2002 Suffolk/VA (Bravo)	SG	0.20 (foliar)	319–347	2	0	0.22	< 0.01	< 0.01	0.24
					1	0.090, 0.13 mean 0.11	< 0.01, < 0.01	< 0.01, < 0.01	mean 0.13
					3	0.16	< 0.01	0.027	0.21
					5	0.099	< 0.01	0.017	0.13
					7	0.098, 0.15 mean 0.13	< 0.01, < 0.01	0.018, 0.026	mean 0.17
					9	0.14	< 0.01	0.026	0.18
					11	0.25	0.012	0.086	0.38
					14	0.047	< 0.01	< 0.01	0.067
					21	0.029	< 0.01	< 0.01	0.049
					28	0.027	< 0.01	< 0.01	0.047
					35	0.027	< 0.01	< 0.01	0.047
USA, 2002 Zellwood/FL (Bravo)	SG	0.20 (foliar)	274–281	2	1	0.58, 0.97 mean 0.78	0.026, 0.035	0.053, 0.071	mean 0.90
USA, 2002 Comstock Park /MI (Strukton)	SG	0.20 (foliar)	235–236	2	1	0.062, 0.37 mean 0.22	< 0.01, 0.019	< 0.01, 0.047	mean 0.28
	SL	0.20 (foliar)	231–234	2	1	0.030, 0.083 mean 0.056	< 0.01, < 0.01	< 0.01, 0.013	mean 0.082
USA, 2002 Colony/OK (Gourmet)	SG	0.20 (foliar)	222–223	2	1	0.013, 0.013 mean 0.013	< 0.01, < 0.01	< 0.01, < 0.01	mean 0.033
USA, 2002 Greenfield/CA (Grenadler)	SG	0.20 (foliar)	306–315	2	1	0.026, 0.046 mean 0.036	< 0.01, < 0.01	< 0.01, < 0.01	mean 0.056

GAP information: Do not combine foliar applications with soil applications, or vice versa. Only use one application method.

[a] Do not apply more than a total of 0.60 kg ai/ha per season

[b] Do not apply more than a total of 0.30 kg ai/ha per season

Fruiting vegetables, Cucurbits

The trials were conducted at 18 locations in the major regions of commercial cucumber, muskmelon and summer squash production in the USA (Hummel, 2003: 43413A014). Two foliar applications of the test substance (dinotefuran 200 g/kg SG formulation) were made at a rate of 0.20 kg ai/ha in a calibrated volume of 214–308 L/ha to obtain the maximum labelled per season rate of 0.40 kg ai/ha. There were 7 day intervals between each application and 1 day between the last application and harvest (i.e., 1 day PHI). Two soil applications of the test substance were made at a rate of 0.30 kg ai/ha to obtain the maximum labelled per season rate of 0.60 kg ai/ha. The first application was made within 1 day after planting, and the second 21 days prior to harvest. In addition, two trials included an additional plot treated with a 100 g/L SL formulation applied according to equivalent rate, timing and spray parameters compared to the foliar treatment of the SG formulation.

Stability of residues under freezer storage conditions was assessed by fortification of untreated melon matrix (control) at a concentration of 1.0 mg/kg for dinotefuran, UF and DN.

Recoveries of residues of dinotefuran, UF and DN from melons, fortified and stored with field-harvested samples, did not show indications of significant degradation under freezer conditions for 161 days.

Cucumber

The maximum storage period for any field-collected sample was 139 days (27–139 days).

Table 58 Dinotefuran and metabolites residues on cucumber from supervised trials in the USA

Cucumber country, year (variety)	Application				PHI Days	Residues, mg/kg			
	Form	kg ai/ha	water, L/ha	no.		Dinotefuran	UF	DN	Total
GAP, USA (Cucurbits)	SG	0.37 (soil) 0.20 (foliar)		[a] [b]	21 1				
USA, 2002 Ty Ty/GA (General Lee)	SG	0.20 (foliar)	279	2	1	0.12, 0.14 mean 0.13	0.027, 0.030	0.059, 0.077	mean 0.26
USA, 2002 Jennings/FL (Thunder)	SG	0.20 (foliar)	282–288	2	1	0.18, 0.19 mean 0.18	0.022, 0.025	0.038, 0.044	mean 0.26
USA, 2002 Chula/GA (Lightning)	SG	0.30 (soil)		2	21	0.052, 0.055 mean 0.053	< 0.01, < 0.01	< 0.01, < 0.01	mean 0.073
	SG	0.20 (foliar)	241–243	2	1	0.17, 0.18 mean 0.18	0.017, 0.019	0.034, 0.046	mean 0.26
	SL	0.20 (foliar)	243	2	1	0.16, 0.16 mean 0.16	0.016, 0.016	0.040, 0.037	mean 0.23
USA, 2002 Campbell/MN (Speedway)	SG	0.20 (foliar)	282	2	1	0.17, 0.25 mean 0.21	0.012, 0.013	0.037, 0.044	mean 0.28
USA, 2002 Conklin/MI (Marketmore 76)	SG	0.20 (foliar)	220–221	2	1	0.12, 0.16 mean 0.14	< 0.01, 0.011	0.017, 0.020	mean 0.18
USA, 2002 Comstock Park /MI (Marketmore 76)	SG	0.20 (foliar)	214–220	2	1	0.17, 0.22 mean 0.20	0.025, 0.029	0.031, 0.035	mean 0.28
USA, 2002 Colony/OK (Long Green)	SG	0.20 (foliar)	215–223	2	0	0.19	0.012	0.048	0.27
					1	0.13, 0.17 mean 0.15	0.011, 0.015	0.047, 0.056	mean 0.23
					3	0.14	0.013	0.068	0.25
					5	0.17	0.016	0.11	0.33
					7	0.14, 0.14 mean 0.14	0.012, 0.013	0.10, 0.12	mean 0.30
					9	0.16	0.012	0.12	0.33
					11	0.10	< 0.01	0.091	0.23
					14	0.068	< 0.01	0.095	0.20
					21	0.031	< 0.01	0.066	0.13
					28	0.014	< 0.01	0.044	0.081

Cucumber country, year (variety)	Application				PHI Days	Residues, mg/kg			
	Form	kg ai/ha	water, L/ha	no.		Dinotefuran	UF	DN	Total
					35	< 0.01	< 0.01	0.034	0.054

GAP information: Do not combine foliar applications with soil applications, or vice versa. Only use one application method.

[a] Do not apply more than a total of 0.60 kg ai/ha per season

[b] Do not apply more than a total of 0.30 kg ai/ha per season

Melons

The maximum storage period for any field-collected sample was 150 days (52–150 days).

Table 59 Dinotefuran and metabolites residues on melons from supervised trials in USA

Melons country, year (variety)	Application				PHI Days	Residues, mg/kg			
	Form	kg ai/ha	water, L/ha	No.		Dinotefuran	UF	DN	Total
GAP, USA (Cucurbits)	SG	0.37 (soil)[a] 0.20 (foliar)[b]			21 1				
USA, 2002 Ty Ty/GA Cantaloupe (Athena)	SG	0.20 (foliar)	277–280	2	1	0.17, 0.20 mean 0.18	0.026, 0.032	0.011, 0.013	mean 0.23
USA, 2002 Campbell/MN Muskmelon (Minerva)	SG	0.20 (foliar)	282	2	1	< 0.01, 0.074 mean 0.042	< 0.01, 0.012	< 0.01, 0.016	mean 0.073
USA, 2002 East Bernard /TX Cantaloupe (Tam Uvalde)	SG	0.20 (foliar)	214–248	2	1	0.11, 0.30 mean 0.20	0.022, 0.063	0.026, 0.077	mean 0.32
USA, 2002 Porterville/CA Cantaloupe (Hale's Rest Jumbo)	SG	0.20 (foliar)	284–285	2	0 1 3 5 7 9 11 14 21 28 35	0.11 0.059, 0.11 mean 0.082 0.068 0.062 0.060, 0.087 mean 0.074 0.080 0.081 0.059 0.055 0.061 0.039	0.014 0.014, 0.024 0.023 0.028 0.025, 0.042 0.040 0.039 0.027 0.027 0.031 0.025	< 0.01 < 0.01, 0.014 0.012 0.015 0.011, 0.016 0.017 0.021 0.010 0.012 0.013 < 0.01	0.14 mean 0.12 0.11 0.12 mean 0.14 0.15 0.16 0.11 0.11 0.12 0.082
USA, 2002 Porterville/CA Honeydew (Green Flesh)	SG	0.30 (soil)		2	21	0.039, 0.040 mean 0.040	< 0.01, < 0.01	< 0.01, < 0.01	mean 0.060
	SG	0.20 (foliar)	282–302	2	1	0.043, 0.064 mean 0.054	0.021, 0.022	0.017, 0.021	mean 0.11
USA, 2002	SG	0.20 (foliar)	278–	2	1	0.079, 0.13	0.017, 0.027	0.016, 0.025	

Melons country, year (variety)	Application				PHI Days	Residues, mg/kg			
	Form	kg ai/ha	water, L/ha	No.		Dinotefuran	UF	DN	Total
Maricopa/AZ Cantaloupe (Esteem)			285			mean 0.10			mean 0.15
	SL	0.20 (foliar)	274–284	2	1	0.13, 0.16 mean 0.15	0.026, 0.043	0.024, 0.041	mean 0.24

GAP information: Do not combine foliar applications with soil applications, or vice versa. Only use one application method.

[a] Do not apply more than a total of 0.60 kg ai/ha per season

[b] Do not apply more than a total of 0.30 kg ai/ha per season

Summer squash and Zucchini

The maximum storage period for any field-collected sample was 144 days (80–144 days).

Table 60 Dinotefuran and metabolites residues on summer squash and zucchini from supervised trials in USA

Summer squash country, year (variety)	Application				PHI Days	Residues, mg/kg			
	Form	kg ai/ha	water, L/ha	no.		Dinotefuran	UF	DN	Total
GAP, USA (Cucurbits)	SG	0.37 (soil) 0.20 (foliar)		[a] [b]	21 1				
USA, 2002 Germansville /PA Summer squash (Superpik)	SG	0.30 (soil)		2	21	0.085, 0.089 mean 0.087	0.012, 0.011	< 0.01, < 0.01	mean 0.11
	SG	0.20 (foliar)	293–294	2	1	0.16, 0.21 mean 0.18	0.051, 0.063	0.045, 0.056	mean 0.32
USA, 2002 Suffolk/VA Summer squash (Early Summer Crookneck)	SG	0.20 (foliar)	297–308	2	1	0.13, 0.17 mean 0.15	0.047, 0.046	0.066, 0.073	mean 0.30
USA, 2002 Madison/FL Summer squash (Early Summer Crookneck)	SG	0.30 (soil)		2	21	< 0.01, 0.073 mean 0.041	< 0.01, < 0.01	< 0.01, 0.012	mean 0.065
	SG	0.20 (foliar)	279–282	2	1	0.081, 0.10 mean 0.092	0.027, 0.027	0.050, 0.040	mean 0.19
USA, 2002 Campbell/MN Zucchini (Spineless Beauty)	SG	0.20 (foliar)	282	2	1	0.098, 0.10 mean 0.10	0.011, 0.014	0.015, 0.019	mean 0.14
USA, 2002 Porterville/CA Zucchini (Golden Rod 3)	SG	0.20 (foliar)	277–285	2	1	0.11, 0.20 mean 0.15	0.028, 0.032	0.022, 0.031	mean 0.22

GAP information: Do not combine foliar applications with soil applications, or vice versa. Only use one application method.

[a] Do not apply more than a total of 0.60 kg ai/ha per season

[b] Do not apply more than a total of 0.30 kg ai/ha per season

Fruiting vegetables, other than Cucurbits

Peppers

The trials were conducted at 10 locations in the major regions of commercial fruiting vegetable production in the USA (Hattermann, 2003: 43415A010). Two applications of the test substance (dinotefuran 200 g/kg SG formulation) were made to each of the treated plots at a rate of 0.20 kg ai/ha (foliar applications) or 0.30 kg ai/ha (soil applications) to obtain the maximum labelled per season rate of 0.40 kg ai/ha for foliar applications and 0.60 kg ai/ha for soil applications. There were *5* to 8 day intervals between each foliar application with samples collected at 1 and 7 day PHI for the foliar trials. For the trials with soil applications, the first application was made at planting or within 7 days of planting and the second application was made 21 days prior to harvest. The soil applications were made by various methods including irrigation, soil directed applications and injection.

The maximum storage period for any field-collected sample was 116 days (48–116 days). Stability of residues under freezer storage conditions was assessed by fortification of untreated tomato matrix (control) at a concentration of 1.0 mg/kg for dinotefuran, UF and DN. Recoveries of residues of dinotefuran, UF and DN from tomatoes, fortified and stored with field-harvested samples, did not show indications of significant degradation under freezer conditions for 161 days.

Table 61 Dinotefuran and metabolites residues on peppers from supervised trials in the USA

Peppers country, year (variety)	Application				PHI Days	Residues, mg/kg			
	Form	kg ai/ha	water, L/ha	No.		Dinotefuran	UF	DN	Total
GAP, USA (Fruiting vegetables)	SG	0.37 (soil) [a] 0.20 (foliar)[b]			21 1				
USA, 2002 Chula/GA Sweet pepper (Capistrano)	SG	0.30 (soil)	277–285	2	21	0.091, 0.096 mean 0.094	< 0.01, < 0.01	< 0.01, < 0.01	mean 0.11
	SG	0.20 (foliar)	275–282	2	1	0.041, 0.043 mean 0.042	< 0.01, < 0.01	< 0.01, < 0.01	mean 0.062
USA, 2002 Jennings/FL Sweet pepper (Aristotle)	SG	0.20 (foliar)	281	2	1	0.12, 0.16 mean 0.14	0.012, 0.021	0.010, < 0.01	mean 0.17
USA, 2002 Madison/FL Sweet pepper (Missela)	SG	0.20 (foliar)	281	2	1	0.24, 0.25 mean 0.25	< 0.01, 0.013	0.017, 0.016	mean 0.29
USA, 2002 New Holland /OH Sweet pepper (Crusader)	SG	0.20 (foliar)	216–239	2	1	0.13, 0.16 mean 0.14	0.015, 0.017	0.012, 0.013	mean 0.18
USA, 2002 Delavan/WI Sweet pepper (Jupiter)	SG	0.20 (foliar)	239–240	2	1	0.029, 0.031 mean 0.030	< 0.01, < 0.01	< 0.01, < 0.01	mean 0.050
USA, 2002 Colony/OK Chili pepper (Habanero)	SG	0.20 (foliar)	215–222	2	1	0.12, 0.21 mean 0.17	< 0.01, 0.015	< 0.01, 0.025	mean 0.21
	SL	0.20 (foliar)	207–220	2	1	0.20, 0.25 mean 0.23	0.010, 0.013	< 0.01, 0.012	mean 0.26
USA, 2002 Maricopa/AZ Chili pepper (Grande)	SG	0.20 (foliar)	278–285	2	1	0.36, 0.46 mean 0.41	0.054, 0.067	0.037, 0.057	mean 0.55

Peppers country, year (variety)	Application				PHI Days	Residues, mg/kg			
	Form	kg ai/ha	water, L/ha	No.		Dinotefuran	UF	DN	Total
USA, 2002 Porterville/CA Sweet pepper (Jupiter)	SG	0.30 (soil)	218–220	2	21	0.017, 0.030 mean 0.024	< 0.01, < 0.01	< 0.01, < 0.01	mean 0.044
	SG	0.20 (foliar)	285–287	2	0	0.34	0.075	0.060	0.52
					1	0.23, 0.23 mean 0.23	0.055, 0.043	0.045, 0.035	mean 0.35
					3	0.27	0.091	0.083	0.50
					5	0.17	0.077	0.061	0.35
					7	0.13, 0.15 mean 0.14	0.050, 0.067	0.039, 0.053	mean 0.28
					9	0.11	0.047	0.035	0.22
					11	0.086	0.039	0.027	0.17
					14	0.085	0.030	0.019	0.15
					21	0.079	0.026	0.015	0.13
					28	0.065	0.021	< 0.01	0.10
					35	0.070	0.015	< 0.01	0.10

GAP information: Do not combine foliar applications with soil applications, or vice versa. Only use one application method.

[a] Do not apply more than a total of 0.60 kg ai/ha per season

[b] Do not apply more than a total of 0.30 kg ai/ha per season

Two residue trials were carried out in Japan. All trials received an application of dinotefuran 10 g/kg GR formulation at 2 g/plant in plant hole, and two foliar applications of dinotefuran 200 g/kg SG formulation at a concentration of 0.010 kg ai/hL with a 7 days interval.

The samples were homogenized and shaken with acetonitrile-water (8:2) solution. The extract was filtered, partitioned with hexane and concentrated. The extract was subject to SPE clean-up steps. Then, dinotefuran was analysed by HPLC-UV. The limit of detection (LOD) of 0.005 mg/kg was determined. Samples were analysed with procedural recovery (= concurrent fortification) samples at levels of 0.005 and 0.20 mg/kg. Procedural recoveries were 73–112%.

The maximum storage period for any field-collected sample was 247 days (121–247 days). Stability of residues under freezer storage conditions was assessed by fortification of untreated tomato matrix (control) at a concentration of 1.0 mg/kg for dinotefuran. Recoveries of residues of dinotefuran from green peppers, fortified and stored with field-harvested samples, did not show indications of significant degradation under freezer conditions for 146–259 days (recoveries 86–101%).

Table 62 Dinotefuran residues on green peppers from supervised trials in Japan

Green pepper country, year (variety)	Application				PHI Days	Residues, mg/kg	Ref
	Form	kg ai/hL	water, L/ha	no.			
GAP, Japan	SG	0.0067–0.010	1000–3000	2	1		
Japan, 2000 (Kyou-yutaka)	GR + SG	0.02 g/plant + 0.010	2000	1 + 2	1 3 7 14	0.43 0.36 0.28 0.080	Komatsu, 2001 JFRL
Japan, 2000 (Kyou-midori)	GR + SG	0.02 g/plant + 0.010	2000	1 + 2	1 3 7 14	1.2 1.1 0.85 0.65	

Tomatoes

The trials were conducted at 17 locations in the major regions of commercial fruiting vegetable production in the USA (Hattermann, 2003: 43415A010). Two applications of the test substance (dinotefuran 200 g/kg SG formulation) were made to each of the treated plots at a rate of 0.20 kg ai/ha (foliar applications) or 0.30 kg ai/ha (soil applications) to obtain the maximum labelled per season rate of 0.40 kg ai/ha for foliar applications and 0.60 kg ai/ha for soil applications. There were *5* to 8 day intervals between each foliar application with samples collected at 1 and 7 day PHI for the foliar trials. For the trials receiving soil applications, the first application was made at planting or within 7 days of planting and the second application was made 21 days prior to harvest. The soil applications were made by various methods including irrigation, soil directed applications and injection.

The maximum storage period for any field-collected sample was 141 days (48–141 days).

Table 63 Dinotefuran and metabolites residues on tomato from supervised trials in the USA

Tomato country, year (variety)	Application				PHI Days	Residues, mg/kg			
	Form	kg ai/ha	water, L/ha	No.		Dinotefuran	UF	DN	Total
GAP, USA (Fruiting vegetables)	SG	0.37 (soil) 0.20 (foliar)		[a] [b]	21 1				
USA, 2002 North Rose/NY (Rutgers)	SG	0.30 (soil)	1020–1048	2	21	0.012, 0.018 mean 0.015	< 0.01, < 0.01	< 0.01, < 0.01	mean 0.035
	SG	0.20 (foliar)	234	2	1	0.11, 0.15 mean 0.13	< 0.01, < 0.01	< 0.01, < 0.01	mean 0.15
USA, 2002 Germansville /PA SG: (Mountain Pride) SL: (Mountain Spring)	SG	0.20 (foliar)	290	2	1	0.15, 0.17 mean 0.16	0.012, < 0.01	0.013, 0.012	mean 0.19
	SL	0.20 (foliar)	290	2	1	0.081, 0.087 mean 0.084	< 0.01, < 0.01	< 0.01, < 0.01	mean 0.10
USA, 2002 Chula/GA (Mountain Spring)	SG	0.20 (foliar)	295	2	1	0.069, 0.10 mean 0.084	< 0.01, < 0.01	< 0.01, < 0.01	mean 0.10
USA, 2002 Doswell/VA (Sun Brite)	SG	0.20 (foliar)	266	2	1	0.040, 0.062 mean 0.051	< 0.01, < 0.01	< 0.01, < 0.01	mean 0.071
USA, 2002 Zellwood/FL (Florida 91)	SG	0.20 (foliar)	278–281	2	1	0.039, 0.039 mean 0.039	< 0.01, < 0.01	< 0.01, < 0.01	mean 0.059
USA, 2002 Jennings/FL (Florida 47)	SG	0.20 (foliar)	276–385	2	1	0.086, 0.0.17 mean 0.13	< 0.01, < 0.01	< 0.01, < 0.01	mean 0.15
USA, 2002 Delavan/WI (Celebrity)	SG	0.20 (foliar)	234–242	2	1	0.13, 0.15 mean 0.14	0.014, 0.017	0.015, 0.021	mean 0.18
USA, 2002 New Holland /OH (TR 12)	SG	0.20 (foliar)	221–222	2	0	0.11	< 0.01	< 0.01	0.13
					1	0.044, 0.064 mean 0.054	< 0.01, 0.012	< 0.01, < 0.01	mean 0.078
					3	0.069	0.016	< 0.01	0.10
					7	0.021, 0.022 mean 0.022	< 0.01, < 0.01	< 0.01, < 0.01	mean 0.042
					11	0.012	< 0.01	< 0.01	0.032
					21	0.021	< 0.01	< 0.01	0.041
					28	< 0.01	< 0.01	< 0.01	< 0.03

Tomato country, year (variety)	Application				PHI Days	Residues, mg/kg			
	Form	kg ai/ha	water, L/ha	No.		Dinotefuran	UF	DN	Total
					35	0.011	< 0.01	< 0.01	0.031
USA, 2002 Porterville/CA (3155)	SG	0.30 (soil)	159–1017	2	21	0.024, 0.066 mean 0.045	< 0.01, < 0.01	< 0.01, < 0.01	mean 0.065
	SG	0.20 (foliar)	287–289	2	1	0.15, 0.17 mean 0.16	0.011, 0.018	0.015, 0.021	mean 0.20
USA, 2002 Kings City/CA (Mt, Fresh)	SG	0.20 (foliar)	285–287	2	1	0.052, 0.074 mean 0.063	< 0.01, < 0.01	< 0.01, < 0.01	mean 0.083
	SL	0.20 (foliar)	286–287	2	1	0.055, 0.083 mean 0.069	< 0.01, < 0.01	< 0.01, < 0.01	mean 0.089
USA, 2002 Pixley/CA (Calebrity)	SG	0.20 (foliar)	278–287	2	1	0.14, 0.15 mean 0.15	0.013, 0.015	0.011, 0.015	mean 0.19
USA, 2002 Porterville/CA (Early Girl)	SG	0.20 (foliar)	282–283	2	1	0.044, 0.068 mean 0.056	< 0.01, 0.011	< 0.01, < 0.01	mean 0.080
USA, 2002 Porterville/CA (Floramerica)	SG	0.20 (foliar)	282–283	2	1	0.078, 0.12 mean 0.097	< 0.01, 0.013	< 0.01, < 0.01	mean 0.12
USA, 2002 Leemore/CA (Stam 55)	SG	0.20 (foliar)	282–283	2	1	0.060, 0.060 mean 0.060	< 0.01, < 0.01	< 0.01, < 0.01	mean 0.080
USA, 2002 Maricopa/AZ (Shady Lady)	SG	0.20 (foliar)	279–291	2	1	0.063, 0.080 mean 0.071	< 0.01, < 0.01	< 0.01, < 0.01	mean 0.091

GAP information: Do not combine foliar applications with soil applications, or vice versa. Only use one application method.

[a] Do not apply more than a total of 0.60 kg ai/ha per season

[b] Do not apply more than a total of 0.30 kg ai/ha per season

Two residue trials were carried out in Japan. All trials received an application of dinotefuran 10 g/kg GR formulation at 2 g/plant in plant hole, and two foliar applications of dinotefuran 200 g/kg SG formulation at a concentration of 0.010 kg ai/hL with 5 or 7 days intervals.

The samples were homogenized and shaken with acetonitrile-water (8:2) solution. The extract was filtered, partitioned with hexane and concentrated. The extract was subject to SPE clean-up steps. Then, dinotefuran was analysed by HPLC-UV. The limit of detection (LOD) of 0.005 mg/kg was determined. Samples were analysed with procedural recovery (= concurrent fortification) samples at levels of 0.20 mg/kg. Procedural recoveries were 90–96%.

The maximum storage period for any field-collected sample was 146 days (138–146 days). Stability of residues under freezer storage conditions was assessed by fortification of untreated tomato matrix (control) at a concentration of 1.0 mg/kg for dinotefuran. Recoveries of residues of dinotefuran from tomatoes, fortified and stored with field-harvested samples, did not show indications of significant degradation under freezer conditions for 146–152 days (recoveries 85–90%).

Table 64 Dinotefuran residues on tomatoes from supervised trials in Japan

Tomato country, year (variety)	Application				PHI Days	Residues, mg/kg	Ref
	Form	kg ai/hl	water, L/ha	no.			
GAP, Japan	SG	0.20 0.0067-0.010	0.5 L/plant 1000–3000	1 2	at planting 1		
Japan, 1998 (Momotaro eight)	G + SG	0.02 g/plant + 0.010	 3000	1 + 2	1 3 7	0.084 0.094 0.086	Komatsu, 2000 JFRL
Japan, 1998 (Momotaro)	G + SG	0.02 g/plant + 0.010	 2000	1 + 2	1 3 7	0.22 0.35 0.25	

Leafy vegetables (including Brassica leafy vegetables)

Lettuce

The trials were conducted at 12 locations in the major regions of commercial leaf lettuce and head lettuce production in the USA (Hummel, 2002: 41409A005). Two applications of the test substance (dinotefuran 200 g/kg SG formulation) were made to each of the treated plots at a rate of 0.15 kg ai/ha in a calibrated volume of 187–369 L/ha to obtain the maximum labelled per season rate of 0.30 kg ai/ha. There were 14 day intervals between each application and seven days between the last application and harvest (7 day PHI). Leaf lettuce was sampled at 1, 3, 5, 7, 10 and 14 days after the final application in the residue decline trial.

Samples were stored upon receipt at the analytical laboratory for varying lengths of time prior to analysis. However, all treated samples were analysed for dinotefuran within 99 days, for DN within 232 days, and for UF within 234 days after collection. Up to 12 months all mean recoveries for dinotefuran in the stored lettuce samples were found to be ≥ 70% of the theoretical value (RCC study 827537). For DN and UF, up to 12 months all recoveries in the stored lettuce samples were found to be > 90% of the value on day 0 (RCC study 834388).

The trials were conducted to compare residue resulting from foliar applications versus residue resulting from soil applications at three locations in the representative regions of commercial lettuce production in the USA (Hummel, 2003: 43409A015). At each location, two foliar applications of the test substance were made at a rate of 0.15 kg ai/ha in a calibrated volume 228–324 L/ha to obtain the maximum labelled per season rate of 0.30 kg ai/ha. There were 14 day intervals between each application and 7 days between the last application and harvest (7 day PHI). At each location, two soil applications of the test substance were made at a rate of 0.30 kg ai/ha to obtain the maximum labelled per season rate of 0.60 kg ai/ha. The first application was applied within 5 days after planting, and the second application was applied 21 days prior to harvest (21 day PHI).

Stored control and fortified samples were removed from storage and analysed following 53 and 161 days. Residues of dinotefuran, UF and DN did not show any indication of degradation under freezer conditions for at least 161 days. The maximum storage interval for field-harvested samples was 112 days (2–112 days).

Table 65 Dinotefuran and metabolites residues on lettuce from supervised trials in USA

Lettuce country, year (variety)	Application				PHI Days	Residues, mg/kg			
	Form	kg ai/ha	water, L/ha	No.		Dinotefuran	UF	DN	Total
GAP, USA (Leafy vegetables)	SG	0.37 (soil) 0.15 (foliar)		[a] [b]	21 7				
Hummel, 2002: 41409A005									
USA, 2001 Porterville/CA Leaf lettuce (Two Star)	SG	0.15 (foliar)	279–285	2	1	2.2, 2.7 mean 2.5	0.25, 0.21	0.16, 0.08	mean 3.0
					3	1.6, 1.8 mean 1.7	0.27, 0.22	0.26, 0.19	mean 2.3
					5	0.81, 0.84 mean 0.83	0.23, 0.28	0.19, 0.41	mean 1.6
					7	1.1, 1.2 mean <u>1.1</u>	0.17, 0.18	0.41, 0.18	mean <u>1.7</u>
					10	0.58, 0.77 mean 0.68	0.15, 0.09	0.36, 0.40	mean 1.3
					14	0.50, 0.65 mean 0.58	0.07, 0.14	0.38, 0.35	mean 1.2
USA, 2001 Porterville/CA	SG	0.15 (foliar)	279–289	2	7	0.17, 0.25 mean <u>0.21</u>	0.14, 0.13	0.20, 0.15	mean <u>0.63</u>

Lettuce country, year (variety)	Application				PHI Days	Residues, mg/kg			
	Form	kg ai/ha	water, L/ha	No.		Dinotefuran	UF	DN	Total
Leaf lettuce (Big Red)									
USA/CA, 2001 Leaf lettuce (New Red Fire)	SG	0.15 (foliar)	286–310	2	7	0.30, 0.34 mean 0.32	0.10, 0.11	0.14, 0.15	mean 0.66
USA, 2001 Stanfield/AZ Leaf lettuce (Darkland)	SG	0.15 (foliar)	268–275	2	7	0.19, 0.21 mean 0.20	0.18, 0.15	0.22, 0.25	mean 0.73
USA, 2001 Suffolk/VA Leaf lettuce (Black Seeded Simpson)	SG	0.15 (foliar)	187–201	2	7	0.20, 0.38 mean 0.29	0.06, 0.05	0.39, 0.29	mean 0.81
USA, 2001 Oviedo/FL Leaf lettuce (Bibb)	SG	0.15 (foliar)	279–280	2	7	0.14, 0.16 mean 0.15	0.02, 0.03	0.09, 0.12	mean 0.33
USA, 2001 Porterville/CA Head lettuce (Bayview)	SG	0.15 (foliar)	281–282	2	7	0.17, 0.18 mean 0.18	0.25, 0.19	0.31, 0.24	mean 0.83
USA, 2001 Porterville/CA Head lettuce (Big Ben)	SG	0.15 (foliar)	282	2	7	0.52, 0.54 mean 0.53	0.20, 0.39	0.24, 0.43	mean 1.4
USA, 2001 San Ardo/CA Head lettuce (Bayview)	SG	0.15 (foliar)	282	2	7	0.08, 0.16 mean 0.12	0.03, 0.03	0.10, 0.10	mean 0.29
USA, 2001 Stanfield/AZ Head lettuce (Green Lightning)	SG	0.15 (foliar)	270–291	2	7	0.06, 0.09 mean 0.08	0.03, 0.05	0.09, 0.09	mean 0.25
USA, 2001 North Rose/NY Head lettuce (Ithaca)	SG	0.15 (foliar)	231–236	2	7	0.12, 0.20 mean 0.16	0.06, 0.06	0.15, 0.19	mean 0.46
USA, 2001 Belle Glade/FL Head lettuce (Gator)	SG	0.15 (foliar)	356–370	2	7	0.13, 0.19 mean 0.16	0.01, 0.02	0.07, 0.08	mean 0.29
Hummel, 2003: 43409A015									
USA, 2002 North Rose/NY Head lettuce (Crispino)	SG	0.30 (soil)	843–864	2	21	0.016, 0.016 mean 0.016	< 0.01, < 0.01	0.019, 0.017	mean 0.049
	SG	0.15 (foliar)	228–235	2	7	0.16, 0.22 mean 0.19	0.051, 0.047	0.21, 0.14	mean 0.49
USA, 2002 Porterville/CA Leaf lettuce (Tiarra)	SG	0.30 (soil)	217–224	2	21	0.095, 0.13 mean 0.11	< 0.01, 0.013	0.043, 0.075	mean 0.20
	SG	0.15 (foliar)	320–324	2	7	0.63, 1.2 mean 0.91	0.17, 0.28	0.43, 0.72	mean 2.0
USA/AZ, 2002 Casa Grande	SG	0.30 (soil)		1	21	0.015, 0.017 mean 0.016	< 0.01, < 0.01	< 0.01, 0.010	mean 0.039

Lettuce country, year (variety)	Application				PHI Days	Residues, mg/kg			
	Form	kg ai/ha	water, L/ha	No.		Dinotefuran	UF	DN	Total
/AZ Leaf lettuce (BOS 9021 G)	SG	0.15 (foliar)	285–303	2	7	2.4, 2.4 mean 2.4	0.38, 0.30	0.36, 0.28	mean 3.3

GAP information: Do not combine foliar applications with soil applications, or vice versa. Only use one application method.

[a] Do not apply more than a total of 0.60 kg ai/ha per season

[b] Do not apply more than a total of 0.30 kg ai/ha per season

Spinach

The trials were conducted at six locations in the major regions of commercial spinach production in the USA (Hummel, 2002: 41409A006). Two applications of the test substance (dinotefuran 200 g/kg SG formulation) were made to each of the treated plots at a rate of 0.15 kg ai/ha in a calibrated volume of 221–307 L/ha to obtain the maximum labelled per season rate of 0.30 kg ai/ha. There were 14 day intervals between each application and seven days between the last application and harvest (7 day PHI). A residue decline trial was sampled at 1, 3, 5, 7, 10 and 14 days after the final application.

Samples were stored upon receipt at the analytical laboratory for varying lengths of time prior to analysis. However, all treated samples were analysed for dinotefuran within 159 days, for DN within 238 days, and for UF within 236 days after collection. Up to 12 months all mean recoveries for dinotefuran in the stored lettuce samples were found to be ≥ 70% (RCC study 827537). For DN and UF, up to 12 months all recoveries in the stored lettuce samples were found to be > 90% of the value on day 0 (RCC study 834388).

The trials were conducted to compare residue resulting from foliar applications versus residue resulting from soil applications at one location in the representative regions of commercial spinach production in the USA (Hummel, 2003: 43409A015). Two foliar applications of the test substance were made at a rate of 0.15 kg ai/ha in a calibrated volume 111–331 L/ha to obtain the maximum labelled per season rate of 0.30 kg ai/ha. There were 14 day intervals between each application and 7 days between the last application and harvest (7 day PHI). Two soil applications of the test substance were made at a rate of 0.30 kg ai/ha to obtain the maximum labelled per season rate of 0.60 kg ai/ha. The first application was made within 5 days after planting, and the second 21 days prior to harvest (21 day PHI).

Stored control and fortified samples were removed from storage and analysed following 53 and 161 days. Residues of dinotefuran, UF and DN did not show any indication of degradation under freezer conditions for at least 161 days. The maximum storage interval for field-harvested samples was 112 days (2–112 days).

Table 66 Dinotefuran and metabolites residues on spinach from supervised trials in the USA

Spinach country, year (variety)	Application				PHI Days	Residues, mg/kg			
	Form	kg ai/ha	water, L/ha	No.		Dinotefuran	UF	DN	Total
GAP, USA (Leafy vegetables)	SG	0.37 (soil) 0.15 (foliar)		[a] [b]	21 7				
Hummel, 2002: 41409A006									
USA, 2001 Porterville/CA (Polka)	SG	0.15 (foliar)	284–286	2	7	0.59, 0.64 mean 0.62	0.07, 0.07	0.17, 0.11	mean 0.89
USA, 2001 San Ardo/CA (Bolero)	SG	0.15 (foliar)	276–280	2	7	0.48, 0.48 mean 0.48	0.05, 0.06	0.08, 0.10	mean 0.68
USA, 2001 Germansville /PA (Tyee)	SG	0.15 (foliar)	262–267	2	7	1.2, 1.2 mean 1.2	1.0, 0.75	0.18, 0.16	mean 2.6

Spinach country, year (variety)	Application				PHI Days	Residues, mg/kg			
	Form	kg ai/ha	water, L/ha	No.		Dinotefuran	UF	DN	Total
USA, 2001 Tasley/VA (Unipack 151)	SG	0.15 (foliar)	221–233	2	1	0.81, 0.91 mean 0.86	0.10, 0.12	0.17, 0.10	mean 1.2
					3	0.59, 0.89 mean 0.74	0.05, 0.05	0.14, 0.15	mean 1.0
					5	0.37, 0.50 mean 0.44	0.03, 0.04	0.10, 0.05	mean 0.60
					7	0.43, 0.43 mean 0.43	0.06, 0.06	0.08, 0.10	mean 0.63
					10	0.34, 0.35 mean 0.35	0.03, 0.03	0.09, 0.13	mean 0.53
					14	0.08, 0.14 mean 0.11	< 0.01, 0.01	0.02, 0.07	mean 0.19
USA, 2001 East Bernard /TX (Bloomsdale Long Standing)	SG	0.15 (foliar)	235–245	2	7	0.49, 0.62 mean 0.56	0.15, 0.24	0.23, 0.41	mean 1.2
USA, 2001 Safford/AZ (Bolero)	SG	0.15 (foliar)	282–283	2	7	2.9, 3.6 mean 3.3	0.50, 0.45	0.38, 0.29	mean 4.4
Hummel, 2003: 43409A015									
USA, 2002 East Bernard /TX (Bloomsdale Long Standing)	SG	0.30 (soil)	111–331	2	21	0.59, 0.71 mean 0.65	< 0.01, < 0.01	0.055, 0.055	mean 0.73
	SG	0.15 (foliar)	298–305	2	7	1.8, 2.2 mean 2.0	0.030, 0.034	0.19, 0.20	mean 2.3
	SL	0.15 (foliar)	297–306	2	7	1.3, 1.4 mean 1.3	0.015, 0.020	0.11, 0.13	mean 1.5

GAP information: Do not combine foliar applications with soil applications, or vice versa. Only use one application method.

[a] Do not apply more than a total of 0.60 kg ai/ha per season

[b] Do not apply more than a total of 0.30 kg ai/ha per season

Watercress

Three field trials were conducted for this study during the 2006 growing season in the USA (Dorschner, 2009: IR-4 09514). At all trials, the treatment consisted of two foliar applications of dinotefuran 200 g/kg SG formulation at a rate of approximately 0.20 kg ai/ha each for a total of approximately 0.40 kg ai/ha. The applications were made 7 days apart. Watercress leaves and stems were collected one day after the final application. At both Hawaii trials an additional treatment was made in a separate plot. The test substance was applied at the same rate, with the same retreatment interval and PHI as the treatment, but a commercially available adjuvant (a non-ionic surfactant) was added to the tank mix.

The maximum storage interval for field-treated samples in this study was 607 days. Storage stability testing was performed after frozen storage for 608 days. Storage stability samples were fortified with dinotefuran and DN at 0.10 mg/kg and UF at 0.08 mg/kg after the receipt of the samples.

Table 67 Dinotefuran and metabolites residues on watercress from supervised trials in USA

Watercress country, year (variety)	Application				PHI Days	Residues, mg/kg			
	Form	kg ai/ha	water, L/ha	No.		Dinotefuran	UF	DN	Total
GAP, USA	SG	0.10-0.20		[a]	1				

Watercress country, year (variety)	Application				PHI Days	Residues, mg/kg			
	Form	kg ai/ha	water, L/ha	No.		Dinotefuran	UF	DN	Total
USA, 2006 Fellsmere/FL (B&W #1)	SG	0.20	271–281	2	1	3.3, 3.5 mean 3.4	0.051, 0.056 mean 0.054	0.23, 0.24 mean 0.23	mean 3.8
USA, 2006 Waipahu/HI (Florida)	SG	0.20	935–963	2	1	1.2, 1.3 mean 1.2	0.036, 0.033 mean 0.035	0.21, 0.24 mean 0.22	mean 1.5
		0.20 with adjuvant	945–954	2	1	1.4, 1.8 mean 1.6	0.034, 0.043 mean 0.039	0.24, 0.28 mean 0.26	mean 2.0
USA, 2006 Aiea/HI (Florida)	SG	0.20	468	2	1	1.8, 2.0 mean 1.9	0.093, 0.092 mean 0.092	0.29, 0.31 mean 0.30	mean 2.4
		0.20 with adjuvant	468–477	2	1	2.1, 2.1 mean 2.1	0.12, 0.13 mean 0.13	0.48, 0.53 mean 0.51	mean 2.9

[a] Do not apply more than a total of 0.40 kg ai/ha per season

Root and tuber vegetables

Potatoes

The trials were conducted at 10 locations in the major regions of commercial potato production in the USA (Hummel, 2003: 43408A016). Four applications of the test substance (dinotefuran 200 g/kg SG formulation) were made to each of the treated plots which included one initial soil-directed application at a rate of 0.37 kg ai/ha, and three foliar applications at a rate of 0.074 kg ai/ha to obtain the maximum season rate of 0.59 kg ai/ha. In general, there were 14 day intervals between each foliar application; the pre-harvest interval (PHI) was **7** days. Samples were taken at 1, 3, 7, 10, 14 and 21 days after the final application in one residue decline trial. The maximum storage interval for field-harvested samples was 106 days.

Residue trials to determine the magnitude of residues of dinotefuran in potatoes, following multiple applications of dinotefuran 200 g/kg SG formulation, were conducted during 2002 to 2003. Seven residue trials were established in typical potato growing areas in the USA, using commercially viable varieties of potatoes (Stearns, 2003: V-02-24871). The test substance was applied first at planting, using in-furrow equipment or backpack sprayers, applying approximately 0.37 kg ai/ha directly into the seedbed followed by three foliar treatments using backpack sprayers, at approximately 0.074 kg ai/ha, timed at 14 day intervals beginning 35 days before harvest.

Samples from this study were extracted for analysis within 134 days of sampling, and were maintained under frozen storage conditions (approximately -20 °C) from sampling until analysis. Storage stability studies have demonstrated that dinotefuran, UF and DN are all stable in/on potato tubers with quantitative recovery at 139 days when stored under frozen conditions.

Table 68 Dinotefuran and metabolites residues on potato from supervised trials in USA

Potato country, year (variety)	Application				PHI Days	Residues, mg/kg			
	Form	kg ai/ha	water, L/ha	No.		Dinotefuran	UF	DN	Total
GAP, USA (Tuberous and Corm vegetables)	SG	0.38 (soil) 0.076 (foliar)		[a] [b]	[c] 7				
Hummel, 2003: 43408A016 [d]									
USA, 2002 Ephrata/WA (Russet Ranger)	SG	0.37 (soil) 0.074 (foliar)	25115 280–282	1+3	1	0.015	< 0.01	< 0.01	0.035
					3	< 0.01	< 0.01	< 0.01	< 0.03
					7	0.017, 0.025 mean 0.021	< 0.01, < 0.01	< 0.01, < 0.01	mean 0.041

Potato country, year (variety)	Application				PHI Days	Residues, mg/kg			
	Form	kg ai/ha	water, L/ha	No.		Dinotefuran	UF	DN	Total
					10	< 0.01	< 0.01	< 0.01	< 0.03
					14	0.013	< 0.01	< 0.01	0.033
					21	< 0.01	< 0.01	< 0.01	< 0.03
	SL	0.074 (foliar)	280–282	3	7	< 0.01, < 0.01 mean < 0.01	< 0.01, < 0.01	< 0.01, < 0.01	mean < 0.03
USA, 2002 Aberdeen/ID (Russet Burbank)	SG	0.37 (soil) 0.074 (foliar)	122 195–202	1+3	7	0.018, 0.021 mean 0.019	< 0.01, < 0.01	< 0.01, < 0.01	mean 0.039
USA, 2002 Moses Lake/WA (Russet Burbank)	SG	0.37 (soil) 0.074 (foliar)	36143 206–207	1+3	7	< 0.01, < 0.01 mean < 0.01	< 0.01, < 0.01	< 0.01, < 0.01	mean < 0.03
USA, 2002 Moses Lake/WA (Russet Burbank)	SG	0.37 (soil) 0.074 (foliar)	36143 206–207	1+3	7	< 0.01, < 0.01 mean < 0.01	< 0.01, < 0.01	< 0.01, < 0.01	mean < 0.03
USA, 2002 Smithfield/UT (Red LaSoda)	SG	0.37 (soil) 0.074 (foliar)	233 234–235	1+3	7	< 0.01, < 0.01 mean < 0.01	< 0.01, < 0.01	< 0.01, < 0.01	mean < 0.03
USA, 2002 North Rose/NY (NY 79)	SG	0.37 (soil) 0.074 (foliar)	918 232–237	1+3	7	0.022, 0.025 mean 0.024	< 0.01, < 0.01	< 0.01, < 0.01	mean 0.044
	SL	0.074 (foliar)	228–238	3	7	< 0.01, < 0.01 mean < 0.01	< 0.01, < 0.01	< 0.01, < 0.01	mean < 0.03
USA, 2002 Suffolk/VA (Russet Norkotah)	SG	0.37 (soil) 0.074 (foliar)	252 245–282	1+3	7	0.017, 0.022 mean 0.020	< 0.01, < 0.01	< 0.01, < 0.01	mean 0.040
USA, 2002 Northwood/ND (Pontiac)	SG	0.37 (soil) 0.074 (foliar)	95 281–282	1+3	7	< 0.01, < 0.01 mean < 0.01	< 0.01, < 0.01	< 0.01, < 0.01	mean < 0.03
USA, 2002 Campbell/MN (Red Pontiac)	SG	0.37 (soil) 0.074 (foliar)	133 281–282	1+3	7	< 0.01, < 0.01 mean < 0.01	< 0.01, < 0.01	< 0.01, < 0.01	mean < 0.03
USA, 2002 Fergus Falls /MN (Red Norland)	SG	0.37 (soil) 0.074 (foliar)	132 280–282	1+3	7	< 0.01, < 0.01 mean < 0.01	< 0.01, < 0.01	< 0.01, < 0.01	mean < 0.03
Stearns, 2003: V-02-24871									
USA, 2002 North Rose/NY (NY-79)	SG	0.37 (soil) 0.074 (foliar)	76 183–186	1+3	7	0.02, 0.03 mean 0.03	< 0.01, < 0.01	< 0.01, < 0.01	mean 0.05
		0.75 (soil) 0.15 (foliar)	76 182–186	1+3	7	0.08, 0.09 mean 0.09	< 0.01, < 0.01	< 0.01, < 0.01	mean 0.11
USA, 2002 Arkansaw/WI (Russet Burbank)	SG	0.37 (soil) 0.074 (foliar)	76 138–142	1+3	3	< 0.01, < 0.01 mean < 0.01	< 0.01, < 0.01	< 0.01, < 0.01	mean < 0.03
					7	< 0.01, < 0.01 mean < 0.01	< 0.01, < 0.01	< 0.01, < 0.01	mean < 0.03
					10	< 0.01, < 0.01 mean < 0.01	< 0.01, < 0.01	< 0.01, < 0.01	mean < 0.03
					14	< 0.01, < 0.01	< 0.01, < 0.01	< 0.01, < 0.01	mean

Potato country, year (variety)	Application				PHI Days	Residues, mg/kg			
	Form	kg ai/ha	water, L/ha	No.		Dinotefuran	UF	DN	Total
					21	mean < 0.01 < 0.01, < 0.01 mean < 0.01	< 0.01, < 0.01	< 0.01, < 0.01	< 0.03 mean < 0.03
USA, 2002 LaSalle/CO (Snowdon)	SG	0.37 (soil) 0.074 (foliar)	85 139–142	1+3	7	< 0.01, < 0.01 mean < 0.01	< 0.01, < 0.01	< 0.01, < 0.01	mean < 0.03
USA, 2002 San Luis/CA (White C)	SG	0.37 (soil) 0.074 (foliar)	76 178–181	1+3	8	< 0.01, < 0.01 mean < 0.01	< 0.01, < 0.01	< 0.01, < 0.01	mean < 0.03
USA, 2002 Payette/ID (Russet Burbank)	SG	0.37 (soil) 0.074 (foliar)	92 186–190	1+3	7	< 0.01, < 0.01 mean < 0.01	< 0.01, < 0.01	< 0.01, < 0.01	mean < 0.03
USA, 2002 Tule Lake/CA (Russet Burbank)	SG	0.37 (soil) 0.074 (foliar)	96 139–140	1+3	8	< 0.01, < 0.01 mean < 0.01	< 0.01, < 0.01	< 0.01, < 0.01	mean < 0.03
USA, 2003 Oviedo/FL (Pontiac)	SG	0.37 (soil) 0.074 (foliar)	90 165–167	1+3	7	0.01, 0.02 mean 0.02	< 0.01, < 0.01	< 0.01, < 0.01	mean 0.04

GAP information: Do not combine foliar applications with soil applications, or vice versa. Only use one application method

[a] Do not apply more than a total of 0.38 kg ai/ha per season

[b] Do not apply more than a total of 0.23 kg ai/ha per season

[c] at pre-plant.

[d] Application: Each first treatment was made as an early-season soil application (e.g., in furrow at planting, soil directed side-dress, etc.). Applications 2, 3 and 4 were foliar sprays using ground application equipment.

Stalk and stem vegetables

Celery

The trials were conducted at six locations in the major regions of commercial celery production in the USA (Hummel, 2002: 41409A006). Two applications of the test substance (dinotefuran 200 g/kg SG formulation) were made to each of the treated plots at a rate of 0.15 kg ai/ha in a calibrated volume of 221–307 L/ha to obtain the maximum labelled per season rate of 0.30 kg ai/ha. There were 14 day intervals between each application and seven days between the last application and harvest (7 day PHI) for all trials except one, which had a 6 day PHI. A residue decline trial was sampled at 1, 4, 6, 10 and 14 days after the final application.

Samples were stored upon receipt at the analytical laboratory for varying lengths of time prior to analysis. However, all treated samples were analysed for dinotefuran within 188 days, for DN within 250 days, and for UF within 248 days after collection. Up to 12 months all mean recoveries for dinotefuran in the stored lettuce samples were found to be ≥ 70% (RCC study 827537). For DN and UF, up to 12 months all recoveries in the stored lettuce samples were found to be > 90% of the value on day 0 (RCC study 834388).

Table 69 Dinotefuran and metabolites residues on celery from supervised trials in the USA

Celery country, year (variety)	Application				PHI Days	Residues, mg/kg			
	Form	kg ai/ha	water, L/ha	No.		Dinotefuran	UF	DN	Total
GAP, USA (Leafy vegetables) [c]	SG	0.37 (soil) 0.15 (foliar)		[a] [b]	21 7				
USA, 2001 Porterville/CA	SG	0.15 (foliar)	282–305	2	1	0.29, 0.72 mean 0.51	0.03, 0.05	0.04, 0.07	mean 0.64

Celery country, year (variety)	Application				PHI Days	Residues, mg/kg			
	Form	kg ai/ha	water, L/ha	No.		Dinotefuran	UF	DN	Total
(Conquistor)					4	0.27, 0.34 mean 0.31	0.11, 0.14	0.11, 0.22	mean 0.70
					6	0.27, 0.29 mean 0.28	0.16, 0.08	0.23, 0.12	mean 0.67
					10	0.11, 0.17 mean 0.14	0.09, 0.08	0.18, 0.16	mean 0.48
					14	0.11, 0.12 mean 0.12	0.09, 0.11	0.20, 0.26	mean 0.55
USA, 2001 Porterville/CA (Conquistor)	SG	0.15 (foliar)	281–307	2	7	0.17, 0.19 mean 0.18	0.11, 0.10	0.20, 0.20	mean 0.58
USA, 2001 Kings City/CA (G-12)	SG	0.15 (foliar)	282–284	2	7	0.06, 0.06 mean 0.06	< 0.01, < 0.01	< 0.01, < 0.01	mean 0.08
USA, 2001 Camarillo/CA (Proprietary variety)	SG	0.15 (foliar)	283–284	2	7	0.10, 0.37 mean 0.24	0.02, 0.04	0.06, 0.06	mean 0.36
USA, 2001 Delavan/WI (Summer Pascal)	SG	0.15 (foliar)	262–267	2	7	< 0.01, 0.18 mean 0.10	0.03, 0.02	0.03, 0.06	mean 0.20
USA, 2001 Oviedo/FL (Utah)	SG	0.15 (foliar)	276–282	2	7	0.21, 0.23 mean 0.22	0.07, 0.05	0.17, 0.15	mean 0.51

GAP information: Do not combine foliar applications with soil applications, or vice versa. Only use one application method.

[a] Do not apply more than a total of 0.60 kg ai/ha per season

[b] Do not apply more than a total of 0.30 kg ai/ha per season

[c] Celery is classified into the crop group for leafy vegetables in the USA.

Cereal grains

Rice

The trials were conducted at 12 locations in the USA (Hummel, 2011: 43406A059). The test substance, formulated as a dinotefuran 200 g/kg SG, was administered in two late-season foliar applications to one treated plot at each trial location. Sampling for residues decline study was performed at two trial locations. Three of the trials included an extra treated plot that also received early-season applications to estimate the relative residue contribution of early-season applications compared to late-season applications.

The maximum intervals for frozen storage, defined as the interval between sampling date and extraction date, were 144 days for rice grain. Stability of residues was assessed for grain and straw in a companion study. Residues of dinotefuran, UF and DN were shown to be stable in rice grain for storage periods of 434 days.

Table 70 Dinotefuran and metabolites residues on rice grain from supervised trials in the USA

Rice grain country, year (variety)	Application				PHI Days	Residues, mg/kg			
	Form	kg ai/ha	water, L/ha	No.		Dinotefuran	UF	DN	Total
GAP, USA	SG	0.11–0.15		2	7				
USA, 2010	SG	0.15	141	2	7	2.8, 2.9	0.37, 0.40	0.31, 0.33	

Rice grain country, year (variety)	Application				PHI Days	Residues, mg/kg			
	Form	kg ai/ha	water, L/ha	No.		Dinotefuran	UF	DN	Total
Fisk/MO (CL-131)						mean 2.9			mean 3.8
	SG	0.30 0.15	142 140–142	1+2	7	3.0, 3.0 mean 3.0	0.40, 0.40	0.36, 0.34	mean 4.0
USA, 2010 Dexter/MO (CL-151)	SG	0.15	141	2	8	2.3, 2.5 mean 2.4	0.33, 0.36	0.34, 0.37	mean 3.3
USA, 2010 Proctor/AR (CL-151)	SG	0.15	125–129	2	7	2.3, 2.5 mean 2.4	0.58, 0.68	0.73, 0.93	mean 4.3
	SG	0.30 0.15	129	1+1	7	3.0, 4.2 mean 3.6	0.77, 1.1	0.97, 1.7	mean 6.5
USA/AR, 2010 Proctor/AR (Wells)	SG	0.15	125–129	2	5 7 9 11 13 15	3.8 2.8, 3.0 mean 2.9 2.9 2.3 2.8 2.5	0.57 0.54, 0.48 0.57 0.52 0.68 0.76	0.61 0.58, 0.53 0.51 0.50 0.68 0.78	5.3 mean 4.3 4.4 3.6 4.6 4.5
USA, 2010 Greenville/MS (CL-131)	SG	0.15	121–133	2	7	1.8, 2.0 mean 1.9	0.31, 0.34	0.30, 0.34	mean 2.7
USA, 2010 Boyle/MS (CL-151)	SG	0.15	129–130	2	7	1.9, 2.0 mean 1.9	0.37, 0.43	0.36, 0.37	mean 2.9
USA, 2010 Washington/LA (Cheniere)	SG	0.15	133–150	2	7	1.3, 1.4 mean 1.4	0.18, 0.20	0.19, 0.19	mean 1.9
USA, 2010 Washington/LA (Cocodrie)	SG	0.15	133–150	2	7	1.1, 1.6 mean 1.3	0.15, 0.21	0.16, 0.23	mean 1.8
USA, 2010 Fisk/MO (CLXL 745)	SG	0.15	141–143	2	7	1.8, 1.8 mean 1.8	0.25, 0.26	0.21, 0.21	mean 2.4
USA, 2010 Bernard/TX (Presidio)	SG	0.042 0.22	172 181	1+2	7	3.7, 3.8 mean 3.7	0.59, 0.59	0.51, 0.49	mean 5.1
	SG	0.089 0.044 0.22	180 177 176–181	1+1+2	7	3.9, 4.1 mean 4.0	0.58, 0.63	0.47, 0.49	mean 5.4
USA, 2010 Butte City/CA (M205)	SG	0.15	140	2	5 7 9 11 13 15	6.3 1.9, 2.1 mean 2.0 3.7 3.0 3.3 4.0	1.4 0.50, 0.52 0.88 0.74 0.93 0.97	2.7 0.69, 0.62 2.0 2.1 2.1 2.2	12 mean 3.5 7.4 6.7 7.2 8.1
USA, 2010 Durham/CA (M205)	SG	0.15	140	2	7	2.5, 2.5 mean 2.5	0.74, 0.66	0.76, 0.77	mean 4.4

Oilseeds

Cotton seed

The trials were conducted at 13 locations in the USA (Hattermann, 2002: 43411B004). Dinotefuran was applied to cotton as 200 g/kg SG formulation. Two applications were made per trial with a 14 day interval between applications at a rate of 0.15 kg ai/ha per application which equates to a total rate of 0.30 kg ai/ha. Applications were made with a tractor mounted boom powered by compressed air or CO_2, or with a CO_2 or N_2 powered backpack sprayer. Cotton seeds were collected from five trials with a spindle picker and from four trials with a stripper machine. The remaining four trials were harvested by hand. Samples were taken at 14 days after the last application for all trials except for one trial harvested at 15 days and another trial harvested at 16 days after the last application. Samples were ginned in the field for each trial to produce fuzzy seed.

Samples were stored upon receipt at the analytical laboratory for varying lengths of time prior to analysis. All treated samples were analysed for dinotefuran and UF within 188days and for DN within 148 days after collection. Storage stability studies have demonstrated that dinotefuran, UF and DN are all stable in cotton seeds with quantitative recovery at 12 months when stored under frozen conditions.

Table 71 Dinotefuran and metabolites residues on cotton seed from supervised trials in the USA

Cotton seed country, year (variety)	Application				PHI Days	Residues, mg/kg			
	Form	kg ai/ha	water, L/ha	No.		Dinotefuran	UF	DN	Total
GAP, USA	SG	0.05–0.15		[a]	14				
USA, 2001 Chula/GA (DP 458 B/RR)	SG	0.15	92–94	2	14	< 0.05, 0.05 mean 0.05	< 0.05, < 0.05	< 0.05, < 0.05	mean 0.15
USA, 2001 Montezuma/GA (Suregrow)	SG	0.15	126–127	2	14	< 0.05, 0.05 mean 0.05	< 0.05, < 0.05	< 0.05, < 0.05	mean 0.15
USA, 2001 Newport/AR (PM 1218 BG/RR)	SG	0.15	94	2	14	0.07, 0.07 mean 0.07	< 0.05, < 0.05	< 0.05, < 0.05	mean 0.17
USA, 2001 Washington/LA (Suregrow 125 B/R)	SG	0.15	94–104	2	2	0.11, 0.14 mean 0.13	< 0.05, < 0.05	< 0.05, < 0.05	mean 0.23
					3	0.08, 0.10 mean 0.09	< 0.05, < 0.05	< 0.05, < 0.05	mean 0.19
					4	0.09, 0.09 mean 0.09	< 0.05, < 0.05	< 0.05, < 0.05	mean 0.19
					5	0.08, 0.11 mean 0.10	< 0.05, < 0.05	< 0.05, < 0.05	mean 0.20
					7	0.10, 0.11 mean 0.11	< 0.05, < 0.05	< 0.05, < 0.05	mean 0.21
					10	0.06, 0.07 mean 0.07	< 0.05, < 0.05	< 0.05, < 0.05	mean 0.17
					14	0.06, 0.06 mean 0.06	< 0.05, < 0.05	< 0.05, < 0.05	mean 0.16
					21	0.05, 0.08 mean 0.07	< 0.05, < 0.05	< 0.05, < 0.05	mean 0.17
					28	0.06, 0.08 mean 0.07	< 0.05, < 0.05	< 0.05, < 0.05	mean 0.17

Cotton seed country, year (variety)	Application				PHI Days	Residues, mg/kg			
	Form	kg ai/ha	water, L/ha	No.		Dinotefuran	UF	DN	Total
					35	0.07, 0.07 mean 0.07	< 0.05, < 0.05	< 0.05, < 0.05	mean 0.17
USA, 2001 Washington/LA (Suregrow 125 B/R)	SG	0.15	94–97	2	14	0.09, 0.10 mean 0.10	< 0.05, < 0.05	< 0.05, < 0.05	mean 0.20
USA, 2001 East Bernard /TX (ST 4892 BR)	SG	0.15	91–94	2	14	< 0.05, 0.05 mean 0.05	< 0.05, < 0.05	< 0.05, < 0.05	mean 0.15
USA, 2001 Littlefield/TX (PM 2326 BG/RR)	SG	0.15	94	2	14	< 0.05, < 0.05 mean < 0.05	< 0.05, < 0.05	< 0.05, < 0.05	mean < 0.15
USA, 2001 Levelland/TX (PM 2326 BG1RR)	SG	0.15	94	2	14	< 0.05, < 0.05 mean < 0.05	< 0.05, < 0.05	< 0.05, < 0.05	mean < 0.15
USA, 2001 Colony/OK (PM 2280 BG/RR)	SG	0.15	119	2	15	0.07, 0.07 mean 0.07	< 0.05, < 0.05	< 0.05, < 0.05	mean 0.17
USA, 2001 Dill City/OK (PM 2326 BG/RR)	SG	0.15	121–122	2	14	0.14, 0.17 mean 0.16	0.06, 0.07	0.06, 0.06	mean 0.33
USA, 2001 Porterville/CA (DP 6207 Acala)	SG	0.15	137–141		1	< 0.05, < 0.05 mean < 0.05	< 0.05, < 0.05	< 0.05, < 0.05	mean < 0.15
					2	< 0.05, < 0.05 mean < 0.05	< 0.05, < 0.05	< 0.05, < 0.05	mean < 0.15
					3	< 0.05, < 0.05 mean < 0.05	< 0.05, < 0.05	< 0.05, < 0.05	mean < 0.15
					5	< 0.05, < 0.05 mean < 0.05	< 0.05, < 0.05	< 0.05, < 0.05	mean < 0.15
					7	< 0.05, < 0.05 mean < 0.05	< 0.05, < 0.05	< 0.05, < 0.05	mean < 0.15
					10	< 0.05, < 0.05 mean < 0.05	< 0.05, < 0.05	< 0.05, < 0.05	mean < 0.15
					14	< 0.05, < 0.05 mean < 0.05	< 0.05, < 0.05	< 0.05, < 0.05	mean < 0.15
					21	< 0.05, < 0.05 mean < 0.05	< 0.05, < 0.05	< 0.05, < 0.05	mean < 0.15
					28	< 0.05, < 0.05 mean < 0.05	< 0.05, < 0.05	< 0.05, < 0.05	mean < 0.15
					35	< 0.05, < 0.05 mean < 0.05	< 0.05, < 0.05	< 0.05, < 0.05	mean < 0.15
USA, 2001 Maricopa/AZ (DP 451 B/RR)	SG	0.15	91–94	2	14	< 0.05, < 0.05 mean < 0.05	< 0.05, < 0.05	< 0.05, < 0.05	mean < 0.15
USA, 2001 Stanfield/AZ (BR 458)	SG	0.15	94	2	16	< 0.05, < 0.05 mean < 0.05	< 0.05, < 0.05	< 0.05, < 0.05	mean < 0.15

[a] Do not apply more than a total of 0.30 kg ai/ha per season

Straw, fodder and forage of cereals

Rice straw

The trials are described under rice (Hummel, 2011: 43406A059).

Table 72 Dinotefuran and metabolites residues on rice straw from supervised trials in the USA

Rice grain country, year (variety)	Application				PHI Days	Residues, mg/kg			
	Form	kg ai/ha	water, L/ha	No.		Dinotefuran	UF	DN	Total
GAP, USA	SG	0.11–0.15		2	7				
USA, 2010 Fisk/MO (CL-131)	SG	0.15	141	2	7	1.2, 1.2 mean 1.2	0.12, 0.10	0.21, 0.18	mean 1.6
	SG	0.30 0.15	142 140–142	1+2	7	1.0, 1.2 mean 1.1	0.13, 0.15	0.26, 0.29	mean 1.6
USA, 2010 Dexter/MO (CL-151)	SG	0.15	141	2	8	0.46, 0.76 mean 0.61	0.08, 0.10	0.29, 0.25	mean 1.1
USA, 2010 Proctor/AR (CL-151)	SG	0.15	125–129	2	7	1.0, 1.3 mean 1.1	0.14, 0.19	0.14, 0.19	mean 1.5
	SG	0.30 0.15	129	1+1	7	0.63, 1.1 mean 0.85	0.10, 0.14	0.13, 0.16	mean 1.2
USA, 2010 Proctor/AR (Wells)	SG	0.15	125–129	2	5 7 9 11 13 15	1.4 1.4, 1.5 mean 1.4 0.93 0.94 0.68 0.52	0.14 0.15, 0.17 0.12 0.16 0.11 0.11	0.14 0.11, 0.14 0.10 0.12 0.10 0.03	1.8 mean 1.8 1.2 1.3 0.95 0.70
USA, 2010 Greenvile/MS (CL-131)	SG	0.15	121–133	2	7	0.24, 1.5 mean 0.87	0.03, 0.14	0.04, 0.08	mean 1.1
USA, 2010 Boyle/MS (CL-151)	SG	0.15	129–130	2	7	0.70, 1.1 mean 0.90	0.08, 0.18	0.04, 0.19	mean 1.2
USA, 2010 Washington/LA (Cheniere)	SG	0.15	133–150	2	7	0.71, 0.92 mean 0.82	0.08, 0.10	0.26, 0.27	mean 1.3
USA, 2010 Washington/LA (Cocodrie)	SG	0.15	133–150	2	7	0.55, 0.71 mean 0.63	0.07, 0.07	0.17, 0.20	mean 0.97
USA, 2010 Fisk/Mo (CLXL 745)	SG	0.15	141–143	2	7	1.1, 1.5 mean 1.3	0.15, 0.17	0.13, 0.20	mean 1.7
USA, 2010 Bernard/TX (Presidio)	SG	0.042 0.22	172 181	1+2	7	2.8, 3.5 mean 3.1	0.44, 0.49	0.98, 0.71	mean 4.8
	SG	0.089 0.044 0.22	180 177 176–181	1+1+2	7	1.9, 2.6 mean 2.2	0.34, 0.34	0.85, 0.83	mean 3.7
USA, 2010 Butte City/CA (M205)	SG	0.15	140	2	5 7 9	2.6 1.3, 2.7 mean 2.0 1.5	0.27 0.22, 0.44 0.21	0.49 0.50, 0.72 0.81	3.6 mean 3.2 2.8

Rice grain country, year (variety)	Application				PHI Days	Residues, mg/kg			
	Form	kg ai/ha	water, L/ha	No.		Dinotefuran	UF	DN	Total
					11	3.8	0.23	0.13	4.3
					13	1.7	0.17	0.44	2.5
					15	1.4	0.15	0.57	2.3
USA, 2010 Durham/CA (M205)	SG	0.15	140	2	7	2.6, 2.6 mean 2.6	0.34, 0.35	0.48, 0.56	mean 3.7

Cotton gin trash

The trials are described under cotton seed (Hattermann, 2002: 43411B004). Gin trash was collected in seven of the trials.

Table 73 Dinotefuran and metabolites residues on cotton gin trash from supervised trials in the USA

Cotton gin trash country, year (variety)	Application				PHI Days	Residues, mg/kg			
	Form	kg ai/ha	water, L/ha	No.		Dinotefuran	UF	DN	Total
GAP, USA	SG	0.05-0.15		[a]	14				
USA, 2001 Newport/AR (PM 1218 BG/RR)	SG	0.15	94	2	14	1.4, 1.6 mean 1.5	0.39, 0.43	1.0, 1.0	mean 3.3
USA, 2001 Littlefield/TX (PM 2326 BG/RR)	SG	0.15	94	2	14	1.9, 2.4 mean 2.2	1.3, 1.7	0.56, 0.64	mean 4.9
USA , 2001 Levelland/TX (PM 2326 BG1RR)	SG	0.15	94	2	14	1.9, 2.0 mean 1.9	0.93, 1.1	0.44, 0.47	mean 3.8
USA, 2001 Colony/OK (PM 2280 BG/RR)	SG	0.15	119	2	15	0.77, 0.96 mean 0.87	0.18, 0.26	0.22, 0.24	mean 1.5
USA, 2001 Dill City/OK (PM 2326 BG/RR)	SG	0.15	121–122	2	14	1.9, 2.2 mean 2.1	0.96, 1.2	1.9, 1.3	mean 5.6
USA, 2001 Maricopa/AZ (DP 451 B/RR)	SG	0.15	91–94	2	14	2.0, 2.6 mean 2.3	1.5, 1.8	1.7, 2.5	mean 7.1
USA, 2001 Stanfield/AZ (BR 458)	SG	0.15	94	2	16	0.91, 1.3 mean 1.1	0.78, 1.2	0.72, 0.72	mean 3.3

[a] Do not apply more than a total of 0.30 kg ai/ha per season

FATE OF RESIDUES IN STORAGE AND PROCESSING

In Processing

The Meeting received information on the fate of dinotefuran residues during the processing of grapes, tomatoes, potatoes, rice grains and cotton seeds. Processing factors have been calculated for dinotefuran residues in grapes, tomatoes, potatoes, rice grains and cotton seeds.

Grapes

Processing study for grape fruits was conducted at two locations in major regions of commercial grape production in the USA (Hattermann, 2003: 43403A019). One soil and two foliar applications of the test substance (dinotefuran 200 g/kg SG formulation) were made to each of the treated plots at a rate of 1.5 kg ai/ha (soil) and 0.74 kg ai/ha (foliar) to obtain 5× the maximum labelled per season rate (3.0 kg ai/ha). The soil application was made 29 days prior to harvest and the two foliar applications were made 15 days and 1 day prior to harvest, respectively.

Bulk RAC grape samples were shipped ambient to the processing facility, on the day of sampling and arrived the same day and the following day. The bulk material was processed after the processor removed RAC samples. The processed commodities as well as the RAC samples were shipped frozen to the analytical laboratory for analysis.

Grapes were hand de-stemmed and put on the trays (single layers of grapes). Raisins were obtained by stacking the trays on the dehydrator operated at 130–140% for approximately 48–96 hours (typical time was 72 hours) to achieve a moisture range between 15–18%.

Fresh grapes were crushed and de-stemmed in the crusher/de-stemmer. Wet mash was collected in an appropriate sized container and then loaded into one or more cloth stacks on the hydraulic press. The juice was collected in plastic bottles for storage.

Table 74 Dinotefuran and metabolites residues in processed commodities of grapes from supervised trials

country, year (variety)	Application			PHI Days	Commodity	Residues, mg/kg					
						Dinotefuran		UF	DN	Total	
	kg ai/ha	water, L/ha	no.			mg/kg	PF			mg/kg	PF
USA/NY, 2002 (Aurora)	1.5 (soil) 0.74 (foliar)	4459 L/plot 934–948	1 + 2	1	Fruit	1.3, 1.5 mean 1.4		0.11, 0.097	0.050, 0.053	mean 1.6	
					Processor	1.5, 1.8 mean 1.7		0.12, 0.16	0.070, 0.10	mean 2.0	
					Raisins	6.2, 6.2 mean 6.2	4.4	0.42, 0.083	0.16, 0.18	mean 6.7	4.2
					Juice	1.8, 2.0 mean 1.9	1.4	0.14, 0.13	0.064, 0.062	mean 2.2	1.4
USA/CA, 2002 (Thompson seedless)	1.5 (soil) 0.74 (foliar)	183 787–792	1 + 2	1	Fruit	0.48, 0.70 mean 0.59		0.012, 0.012	< 0.01, < 0.01	mean 0.62	
					Processor	0.38, 0.39 mean 0.39		< 0.01, < 0.01	0.012, 0.011	mean 0.42	
					Raisins	1.6, 2.0 mean 1.8	3.1	0.035, < 0.01	0.028, 0.021	mean 1.9	3.1
					Juice	0.55, 0.57 mean 0.56	0.95	0.011, 0.015	< 0.01, 0.012	mean 0.59	0.95

Tomatoes

Processing study for tomatoes was conducted at three locations in major regions of commercial tomato production in the USA (Hattermann, 2003: 43415A011). For two of the trials, two foliar applications were made and in one trial two soil applications were done. The application rate for both types of application was 1.0 kg ai/ha per application (2.0 kg ai/ha/year) which is 5× the maximum proposed label rate. These applications were made 7 days apart with the final application being 1 day prior to harvest (PHI) for the trials that received foliar applications. For the other trial the two soil applications were made 3 months apart with the final application made 21 days prior to harvest and the first application at planting.

Bulk RAC tomato samples were shipped ambient to the processing facility on the day of sampling and arrived on the same day and the following day. The co-operator collected RAC samples were shipped overnight (frozen) to the analytical laboratory for analysis. The bulk samples were processed after the processor took RAC samples from the bulk material. The processed commodities, as well as the RAC samples, were shipped frozen to the analytical laboratory for analysis.

The first processing step was to wash the tomatoes. The washed tomatoes were introduced into a grinder which discharged crushed tomatoes into a surge tank. The crush was pumped into the hot break system which had been started on water to obtain the required outlet temperature. After the cruch was heated, the hot crush was processed through a finisher to remove peel and seeds and produce juice. Concentration of juice to puree was done using a vacuum evaporator until the natural tomato soluble solids (NTSS) were within the range of 8–16%. Puree was condensed to paste using a vacuum kettle evaporator. The target NTSS for paste was 24–35%.

Table 75 Dinotefuran and metabolites residues in processed commodities of tomatoes from supervised trials

country, year (variety)	Application			PHI Days	Commodity	Residues, mg/kg					
	kg ai/ha	water, L/ha	no.			Dinotefuran		UF	DN	Total	
						mg/kg	PF			mg/kg	PF
USA/NY, 2002 (Glamor)	1.0 (foliar)	271–281	2	1	Fruit	0.44, 0.50 mean 0.47		0.019, 0.025	0.014, 0.022	mean 0.52	
					Processor	0.29, 0.58 mean 0.44		0.012, 0.028	< 0.01, 0.017	mean 0.48	
					Paste	1.8, 2.5 mean 2.1	4.4	0.10, 0.13	0.074, 0.081	mean 2.4	4.6
					Puree	0.70, 0.85 mean 0.77	1.6	0.044, 0.049	0.029, 0.035	mean 0.87	1.6
USA/CA, 2002 (3155)	1.0 (soil)	157–851	2	21	Fruit	0.054, 0.067 mean 0.061		< 0.01, < 0.01 < 0.01	< 0.01, < 0.01 < 0.01, < 0.01	mean 0.081	
					Processor	0.082, 0.095 mean 0.089		< 0.01, < 0.01 < 0.01	< 0.01, < 0.01 < 0.01, < 0.01	mean 0.11	
					Paste	0.33, 0.47 mean 0.40	6.6	< 0.01, < 0.01 < 0.01	< 0.01, < 0.01	mean 0.42	5.2
					Puree	0.14, 0.15 mean 0.15	2.5	< 0.01, < 0.01 < 0.01		mean 0.17	2.1
USA/FL, 2002 (Florida 91)	1.0 (foliar)	327–337	2	1	Fruit	0.68, 1.3 mean 0.98		< 0.01, < 0.01 < 0.01	< 0.01, 0.015	mean 1.0	
					Processor	0.52, 0.76 mean 0.64		< 0.01, < 0.01 < 0.01	< 0.01, < 0.01 0.027, 0.034	mean 0.66	
					Paste	3.0, 3.4 mean 3.2	3.3	0.037, 0.041	< 0.01, 0.013	mean 3.3	3.3
					Puree	0.97, 1.2 mean 1.1	1.1	0.011, 0.015		mean 1.1	1.1

Potatoes

Processing study was carried out at two locations in major regions of commercial potato production in the USA (Hattermann, 2003: 43408A017). One soil and three foliar applications of the test substance (dinotefuran 200 g/kg SG formulation) were made to each of the treated plots at a target rate of 1.9 kg ai/ha (soil) and 0.37 kg ai/ha (foliar) to obtain 5× the maximum labelled per season rate (3.0 kg ai/ha). The soil application was made at planting with subsequent foliar applications made at

14 (±2) day intervals with the final application done 7 days prior to harvest. An exception was seen in the North Dakota trial in that there were 18 days between the last two foliar applications due to unfavourable weather conditions.

Bulk RAC potato samples were shipped ambient to the processing facility on the day of sampling and arrived the following day for both trials. The RAC samples collected by the co-operator were shipped overnight (frozen) to the analytical laboratory for analysis. The bulk samples were processed after the processor took samples from the bulk RAC. The final processed commodities obtained from the bulk samples, as well as the RAC samples, were shipped frozen to the analytical laboratory for analysis.

Production of potato chips

Potato chips were produced by slicing peeled potatoes and then frying the slices in fresh vegetable oil. Frying was completed in a series of batches. After the chips were cooled, they were weighed.

Production of potato granules

Potato granules were produced by dicing the peeled potatoes. The diced potatoes were divided into two batches, one for producing seed granules and one for producing the final granules. The dices were cooked in boiling water until soft. The dices for seed granules were mashed until smooth, spread on dryer sheets, dried, and ground. The remaining cooked dices for the final granules were mashed until smooth. The seed granules were blended in until the mash had a grainy appearance. This mixture, after 30 minutes of conditioning, was then spread on dryer sheets, dried, and ground. The potato granules from each sample were collected in separate plastic bags.

Table 76 Dinotefuran and metabolites residues in processed commodities of potatoes from supervised trials

country, year (variety)	Application			PHI Days	Commodity	Residues, mg/kg					
	kg ai/ha	water, L/ha	no.			Dinotefuran		UF	DN	Total	
						mg/kg	PF			mg/kg	PF
USA/ND, 2002 (NorValley)	1.9 (soil) 0.37 (foliar)	94 281–290	1 + 3	7	Tuber	0.054, 0.059 mean 0.056		< 0.01, < 0.01	< 0.01, < 0.01	mean 0.076	
					Processor	0.052, 0.062 mean 0.057		< 0.01, < 0.01	< 0.01, < 0.01	mean 0.077	
					Granules	0.19, 0.22 mean 0.21	3.8	< 0.01, 0.011	< 0.01, < 0.01	mean 0.23	3.0
					Chips	0.14, 0.15 mean 0.14	2.5	< 0.01, < 0.01	< 0.01, < 0.01	mean 0.16	2.1
					Wet peel	0.018, 0.028 mean 0.023	0.41	< 0.01, < 0.01	< 0.01, < 0.01	mean 0.043	0.57
USA/ID, 2002 (Russet Burbank)	1.9 (soil) 0.37 (foliar)	181 187–191	1 + 3	7	Tuber	0.026, 0.029 mean 0.028		< 0.01, < 0.01	< 0.01, < 0.01	mean 0.048	
					Processor	0.025, 0.028 mean 0.027		< 0.01, < 0.01	< 0.01, < 0.01	mean 0.047	
					Granules	0.082, 0.089 mean 0.086	3.1	0.011, 0.011	< 0.01, < 0.01	mean 0.11	2.3
					Chips	0.047, 0.056 mean 0.051	1.8	< 0.01, < 0.01	< 0.01, < 0.01	mean 0.071	1.5
					Wet peel	< 0.01, < 0.01 mean < 0.01	< 0.4	< 0.01, < 0.01	< 0.01, < 0.01	mean < 0.03	< 0.6

Rice grains

Processing study was carried out at two locations in major regions of rice production in the USA (Hummel, 2011: 43406A061). Two applications of the test substance (dinotefuran 200 g/kg SG formulation) were made at a target rate of 0.75 kg ai/ha, which is 5× the maximum label rate. The pre-harvest interval (PHI) was 7 days for both trials.

Following collection and processing, RAC and processed samples were shipped frozen to the analytical laboratory and maintained in frozen storage until analysis.

Table 77 Dinotefuran and metabolites residues in processed commodities of rice grains from supervised trials

country, year (variety)	Application			PHI Days	Commodity	Residues, mg/kg					
	kg ai/ha	water, L/ha	no.			Dinotefuran		UF	DN	Total	
						mg/kg	PF			mg/kg	PF
USA/MO, 2010 (CL-171–AR)	0.75	140	2	7	Grain	13, 13, 18 mean 15		1.7, 1.8, 2.3	0.99, 0.97, 1.3	mean 20	
					Processor	15, 17 mean 16		2.0, 2.2	1.0, 1.2	mean 20	
					Hulls	55, 63 mean 59	3.9	7.3, 8.3	4.2, 4.8	mean 75	3.8
					Bran	5.6, 8.0 mean 6.8	0.45	0.73, 1.0	0.27, 0.50	mean 8.4	0.42
					Polished rice	0.22, 0.24 mean 0.23	0.02	0.051, 0.060	< 0.01, < 0.01	mean 0.31	0.02
USA/TX, 2010 (Presidio)	1.1	182	2	7	Grain	8.1, 10, 10 mean 9.4		1.3, 1.5, 1.5	1.2, 1.3, 1.4	mean 13	
					Processor	8.1, 12 mean 10		1.0, 1.8	0.96, 1.5	mean 13	
					Hulls	39, 62 mean 51	5.4	5.3, 9.6	5.1, 8.7	mean 70	5.4
					Bran	6.8, 10 mean 8.4	0.89	0.81, 1.3	0.49, 0.65	mean 11	0.85
					Polished rice	0.33, 0.58 mean 0.46	0.05	0.077, 0.13	< 0.01, < 0.01	mean 0.60	0.05

Cotton seed

Processing study was carried out in the USA (Hattermann, 2002: 43411B005). The test substance (dinotefuran 200 g/kg SG formulation) was applied to cotton twice at near the targeted application rate of 0.75 kg ai/ha for each application using a CO_2 tractor mounted sprayer in a calibrated volume of 94–131 L/ha. Samples were collected 14 days after the final application.

All RAC samples (co-operator and processor collected) were frozen immediately after collection and shipped frozen to the analytical laboratory for analysis. Bulk seed cotton samples were collected and immediately shipped to the processor. Prior to processing, the samples were removed from frozen storage and ginned. Processing was done according to a procedure that simulated commercial practices and the processed samples were frozen and shipped to the analytical laboratory for analysis.

Maximum storage intervals for RAC samples were 130 days and 107 days for processed samples. Dinotefuran, UF and DN were found to be stable in all matrices (RAC as well as PC) for 12 months in the storage stability studies.

Table 78 Dinotefuran and metabolites residues in processed commodities of cotton seeds from supervised trials

country, year (variety)	Application			PHI Days	Commodity	Residues, mg/kg					
	kg ai/ha	water, L/ha	no.			Dinotefuran		UF	DN	Total	
						mg/kg	PF			mg/kg	PF
USA/CA, 2001 (GTO Maxxa)	0.75	127–129	2	14	Seeds	0.24, 0.41 mean 0.33		0.10, 0.16 mean 0.13	0.06, 0.08 mean 0.07	mean 0.59	
					Processor	0.31, 0.74 mean 0.53		0.14, 0.32 mean 0.23	0.08, 0.15 mean 0.12	mean 0.98	
					Meal	< 0.05, 0.06 mean 0.06	0.18	< 0.05, < 0.05	< 0.05, < 0.05	mean 0.16	0.27
					Hulls	0.06, 0.07 mean 0.07	0.21	< 0.05, < 0.05	< 0.05, < 0.05	mean 0.17	0.29
					Refined oil	< 0.01, < 0.01 mean < 0.01	< 0.03	< 0.01, < 0.01	< 0.01, < 0.01	mean < 0.03	< 0.05
USA/AR, 2001 (Stoneville 474)	0.75	94	2	14	Seeds	0.08, 0.10 mean 0.09		< 0.05, < 0.05	0.22, < 0.05 mean 0.14	mean 0.32	
					Processor	0.09, 0.10 mean 0.10		< 0.05, < 0.05	< 0.05, < 0.05	mean 0.20	
					Meal	< 0.05, 0.05 mean 0.05	0.56	< 0.05, < 0.05	< 0.05, < 0.05	mean 0.15	0.47
					Hulls	0.12, 0.13 mean 0.13	1.4	< 0.05, < 0.05	< 0.05, < 0.05	mean 0.23	0.72
					Refined oil	< 0.01, < 0.01 mean < 0.01	< 0.11	< 0.01, < 0.01	< 0.01, < 0.01	mean < 0.03	< 0.09

RESIDUES IN ANIMAL COMMODITIES

Farm animal feeding studies

The Meeting received lactating dairy cow feeding studies.

Lactating dairy cow

The study was designed to qualify the residues of dinotefuran and its metabolites DN and UF found in milk and tissues of dairy cows following oral administration of dinotefuran, DN phosphate and UF for 29–30 days (Ross, 2003: MTU 261/024360). Groups of lactating dairy cows received dinotefuran, DN phosphate and UF incorporated twice daily into the concentrate feed in an aqueous formulation at levels equivalent to 100, 300 and 1000 mg total ai/ 550 kg/day for 29–30 days. These application rates equates to 5, 15 and 50 ppm total ai/kg dry feed. The test material was in the ratio 3:1:1 dinotefuran: UF: DN. An untreated group of cows was also included in the study to provide control data. Dose levels were based on a nominal feed intake of 20 kg (dry matter equivalent) per day for a cow of 550 kg bodyweight.

Animals were observed several times daily for any clinical signs of toxicity or ill health. Bodyweights were determined at intervals and concentrate food/hay consumption was monitored daily. Milk yields were recorded twice daily. Milk samples were taken from all cows daily throughout the test period and were submitted for assay or stored frozen as whole milk. Additional samples taken on two occasions were separated into cream and skimmed milk samples and were submitted for assay. At termination of the experimental period, all cows were sacrificed and examined macroscopically post mortem. Selected tissues were retained for determination of residue concentrations.

Samples were analysed using methodology developed and validated in the study MTU 250/024447 as described previously. Samples were extracted by shaking with an acidic acetonitrile/water mixture. Clean-up was by liquid-liquid partition and solid phase extraction using C_{18} cartridge. Quantitation was by HPLC using tandem mass spectrometric detection (HPLC-MS/MS). The analytical method for the determination of dinotefuran, DN and UF in bovine milk, cream and tissues (liver, kidney, muscle and fat) was validated over the range 0.01 to 1.0 mg/kg (mg/L for milk). The validation of methodology for the determination of dinotefuran, DN and UF in bovine milk, cream and tissues demonstrated that it can be accurately determined at a LOQ of 0.01 mg/kg.

No residues of dinotefuran, DN and UF above the LOQ were detected in any non-treated samples of any matrix.

UF was the predominant residue found, occurring in milk from all treated animals. Lower concentrations of dinotefuran occurred in the high dose group (10×) only with the exception of samples for 3× at day 2 p.m. and at day 14. With three exceptions 1× at day 1 a.m., 10× at day 2 a.m. and 10× at day 12, DN was not quantifiable in any whole milk samples.

The method for calculating total residues for animal products is illustrated below.

Dinotefuran	UF	Total
0.020	0.17	0.24 (0.020 + 0.17 × 1.3)
< 0.01	0.24	0.32 (0.01 + 0.24 × 1.3)
< 0.01	< 0.01	< 0.02 (0.01 + 0.01)

Table 79 Residues of dinotefuran and metabolites in whole milk (mg/L)

Day	Group 1 (control)			
	Dinotefuran	UF	DN	Total
–7	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)	< 0.02 (3)
–1	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)	< 0.02 (3)
1 AM	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)	< 0.02 (3)
1 PM	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)	< 0.02 (3)
2 AM	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)	< 0.02 (3)
2 PM	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)	< 0.02 (3)
4	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)	< 0.02 (3)
7	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)	< 0.02 (3)
10	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)	< 0.02 (3)
12	na	na	na	na
14	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)	< 0.02 (3)
18	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)	< 0.02 (3)
21	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)	< 0.02 (3)
23	na	na	na	na
25	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)	< 0.02 (3)
28	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)	< 0.02 (3)
29-31[c]	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)	< 0.02 (3)
	Group 2 (100 [a])			
	Dinotefuran	UF	DN	Total
–7	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)	< 0.02 (3)
–1	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)	< 0.02 (3)
1 AM	< 0.01 (3)	< 0.01 (3)	< 0.01, 0.028,	< 0.02 (3)
1 PM	< 0.01 (3)	0.011, < 0.01, 0.010	< 0.01	0.024, < 0.02, 0.023 mean 0.022
2 AM	< 0.01 (3)	0.012, 0.011, 0.012	< 0.01 (3)	0.026, 0.024, 0.026 mean 0.025
2 PM	< 0.01 (3)	0.019, 0.013, 0.015	< 0.01 (3)	0.035, 0.027, 0.030 mean 0.031
4	< 0.01 (3)	0.022, 0.017, 0.015	< 0.01 (3)	0.039, 0.032, 0.030 mean 0.034
7	< 0.01 (3)	0.017, 0.015., 0.021	< 0.01 (3)	0.032, 0.030, 0.037 mean 0.033
10	< 0.01 (3)	0.018, 0.015, 0.021	< 0.01 (3)	0.033, 0.030, 0.037 mean 0.033
12	na	na	< 0.01 (3)	na
14	< 0.01 (3)	0.019, 0.015, 0.018	na	0.035, 0.030, 0.033 mean 0.033
18	< 0.01 (3)	0.019, 0.016, 0.018	< 0.01 (3)	0.035, 0.031, 0.033 mean 0.033
21	< 0.01 (3)	0.018, 0.014, 0.019	< 0.01 (3)	0.033, 0.028, 0.035 mean 0.032
23	na	na	< 0.01 (3)	na

Day	Group 1 (control)			
	Dinotefuran	UF	DN	Total
25	< 0.01 (3)	0.019, 0.015, 0.021	na	0.035, 0.030, 0.037 mean 0.034
28	< 0.01 (3)	0.020, 0.015, 0.021	< 0.01 (3)	0.036, 0.030, 0.037 mean 0.034
29–31 [b]	< 0.01 (3)	0.012, 0.015, 0.021	< 0.01 (3) < 0.01 (3)	0.026, 0.030, 0.037 mean 0.031
Day	Group 3 (300 [a])			
	Dinotefuran	UF	DN	Total
–7	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)	< 0.02 (3)
–1	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)	< 0.02 (3)
1 AM	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)	< 0.02 (3)
1 PM	< 0.01 (3)	0.019, 0.032, 0.026	< 0.01 (3)	0.035, 0.052, 0.044 mean 0.044
2 AM	< 0.01 (3)	0.043, 0.036, 0.046	< 0.01 (3)	0.066, 0.057, 0.070 mean 0.064
2 PM	< 0.01, < 0.01, 0.011	0.057, 0.050, 0.044	< 0.01 (3)	0.084, 0.075, 0.068 mean 0.076
4	< 0.01 (3)	0.044, 0.042, 0.042	< 0.01 (3)	0.067, 0.063, 0.063 mean 0.064
7	< 0.01 (3)	0.040, 0.041., 0.055	< 0.01 (3)	0.062, 0.063, 0.082 mean 0.069
10	< 0.01 (3)	0.048, 0.051, 0.048	< 0.01 (3)	0.072, 0.076, 0.072 mean 0.073
12	na	na	na	na
14	< 0.01, 0.012, < 0.01	0.063, 0.057, 0.054	< 0.01 (3)	0.092, 0.086, 0.080 mean 0.086
18	< 0.01 (3)	0.045, 0.057, 0.062	< 0.01 (3)	0.069, 0.086, 0.091 mean 0.082
21	< 0.01 (3)	0.050, 0.064, 0.053	< 0.01 (3)	0.075, 0.093, 0.079 mean 0.082
23	na	na	na	na
25	< 0.01 (3)	0.063, 0.068, 0.062	< 0.01 (3)	0.092, 0.098, 0.091 mean 0.094
28	< 0.01 (3)	0.056, 0.056, 0.082	< 0.01 (3)	0.083, 0.083, 0.12 mean 0.095
29–31 [b]	< 0.01 (3)	0.063, 0.063, 0.069	< 0.01 (3)	0.092, 0.092, 0.10 mean 0.095
Day	Group 4 (1000 [a])			
	Dinotefuran	UF	DN	Total
–7	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)	< 0.02 (3)
–1	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)	< 0.02 (3)
1 AM	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)	< 0.02 (3)
1 PM	0.036, 0.039, 0.020	0.090, 0.097, 0.059	< 0.01 (3)	0.15, 0.17, 0.10 mean 0.14
2 AM	< 0.01 (3)	0.20, 0.15, 0.076	0.011, < 0.01,	0.27, 0.21, 0.11 mean 0.20
2 PM	0.022, 0.029, 0.015	0.15, 0.11, 0.097	< 0.01	0.22, 0.17, 0.14 mean 0.18
4	< 0.01 (3)	0.19, 0.21, 0.17	< 0.01 (3)	0.26, 0.28, 0.23 mean 0.26
7	< 0.01 (3)	0.24, 0.15., 0.11	< 0.01 (3)	0.32, 0.21, 0.15 mean 0.23
10	< 0.01 (3)	0.30, 0.23, 0.20	< 0.01 (3)	0.40, 0.31, 0.27 mean 0.33
12	< 0.01, 0.018, 0.015	0.29, 0.18, 0.13	< 0.01 (3)	0.39, 0.25, 0.18 mean 0.27
14	0.012, 0.017, 0.012	0.20, 0.16, 0.13	< 0.01, < 0.01,	0.27, 0.23, 0.18 mean 0.23
18	0.016, 0.017, 0.011	0.20, 0.16, 0.14	0.013	0.28, 0.23, 0.19 mean 0.23
21	0.011, 0.020, 0.012	0.25, 0.17, 0.15	< 0.01 (3)	0.34, 0.24, 0.21 mean 0.26
23	< 0.01 (3)	0.15, 0.18, 0.16	< 0.01 (3)	0.21, 0.24, 0.22 mean 0.22
25	< 0.01 (3)	0.31, 0.18, 0.23	< 0.01 (3)	0.41, 0.24, 0.31 mean 0.32
28	< 0.01 (3)	0.24, 0.17, 0.13	< 0.01 (3)	0.32, 0.23, 0.18 mean 0.24
29–31 [b]	< 0.01 (3)	0.23, 0.16, 0.11	< 0.01 (3) < 0.01 (3) < 0.01 (3)	0.31, 0.22, 0.15 mean 0.23

[a] mg total ai/550 kg/day

[b] sample from the last milking on the morning of sacrifice

na: not analysed

A similar distribution of residues was seen in the results of analysis of skimmed milk at day 14 and day 28, with measurable concentrations of UF for all treated animals. At day 14 residues of dinotefuran were detected in the high dose group (10×) only (with one exception at 3×). At day 28 a single residue of dinotefuran was found in one animal from the high dose group only. No quantifiable residues of DN were found in skimmed milk.

In cream, residues of UF occurred in the intermediate and high dose groups (3×, 10×) only. UF residues were not detected in the low dose group (1×). No quantifiable residues of dinotefuran and DN were found in any cream samples.

Table 80 Residues of dinotefuran and metabolites in skimmed milk and cream (mg/L)

Group	Day 14			Day 28		
	Dinotefuran	UF	DN	Dinotefuran	UF	DN
Skimmed milk						
1 Control	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)
2 100 [a]	< 0.01 (3)	0.027, 0.026, 0.022	< 0.01 (3)	< 0.01 (3)	0.018, 0.011, 0.017	< 0.01 (3)
3 300 [a]	< 0.01 (2), 0.015	0.11, 0.10, 0.073	< 0.01 (3)	< 0.01 (3)	0.033, 0.037, 0.038	< 0.01 (3)
4 1000 [a]	0.025, 0.033, 0.012	0.39, 0.24, 0.15	< 0.01 (3)	< 0.01 (2), 0.013	0.33, 0.15, 0.12	< 0.01 (3)
Cream						
1 Control	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)
2 100 [a]	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)
3 300 [a]	< 0.01 (3)	0.023, 0.019, 0.017	< 0.01 (3)	< 0.01 (3)	0.029, 0.016, 0.025	< 0.01 (3)
4 1000 [a]	< 0.01 (3)	0.071, 0.072, 0.060	< 0.01 (3)	< 0.01 (3)	0.091, 0.059, 0.036	< 0.01 (3)

[a] mg total ai/550 kg/day

In tissues, UF was again the predominant residue. Lower concentrations of DN were detected in one or more samples from animals in the high dose group only (10×) in the case of liver, kidney and muscle. No detectable residues of dinotefuran occurred in any tissue samples.

Table 81 Residues of dinotefuran and metabolites in tissues

Group	Residues, mg/kg			
	Dinotefuran	UF	DN	Total
Liver				
1 Control	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)	< 0.02 (3)
2 100[a]	< 0.01 (3)	< 0.01, < 0.01, 0.016	< 0.01 (3)	< 0.02, < 0.02, 0.031 mean 0.024
3 300 [a]	< 0.01 (3)	0.043, 0.033, 0.042	< 0.01 (3)	0.066, 0.053, 0.065 mean 0.061
4 1000 [a]	< 0.01 (3)	0.19, 0.12, 0.066	0.016, 0.023, 0.017	0.26, 0.17, 0.096 mean 0.18
Kidney				
1 Control	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)	< 0.02 (3)
2 100 [a]	< 0.01 (3)	0.011, < 0.01, 0.012	< 0.01 (3)	0.024, < 0.02, 0.026 mean 0.023
3 300 [a]	< 0.01 (3)	0.051, 0.046, 0.045	< 0.01 (3)	0.076, 0.070, 0.069 mean 0.072
4 1000 [a]	< 0.01 (3)	0.29, 0.13, 0.11	0.039, < 0.01, < 0.01	0.39, 0.18, 0.15 mean 0.24
Muscle				
1 Control	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)	< 0.02 (3)
2 100 [a]	< 0.01 (3)	0.012, 0.011, 0.013	< 0.01 (3)	0.026, 0.024, 0.027 mean 0.026
3 300 [a]	< 0.01 (3)	0.033, 0.039, 0.040	< 0.01 (3)	0.053, 0.060, 0.062 mean 0.058
4 1000 [a]	< 0.01 (3)	0.19, 0.11, 0.080	0.022, 0.023, 0.023	0.26, 0.15, 0.11 mean 0.17
Subcutaneous fat				
1 Control	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)	< 0.02 (3)

Group	Residues, mg/kg			
	Dinotefuran	UF	DN	Total
2 100 [a]	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)	< 0.02 (3) mean < 0.02
3 300 [a]	< 0.01 (3)	0.028, 0.042, < 0.01	< 0.01 (3)	0.046, 0.065, < 0.02 mean 0.044
4 1000 [a]	< 0.01 (3)	0.13, 0.053, 0.026	< 0.01 (3)	0.18, 0.079, 0.044 mean 0.10
Peritoneal fat				
1 Control	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)	< 0.02 (3)
2 100 [a]	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)	< 0.02 (3) mean < 0.02
3 300 [a]	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)	< 0.02 (3) mean < 0.02
4 1000 [a]	< 0.01 (3)	0.052, 0.028, < 0.01	< 0.01 (3)	0.078, 0.046, < 0.02 mean 0.048

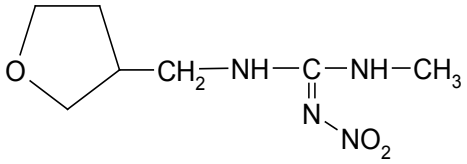
[a] mg total ai/550 kg/day

APPRAISAL

Residue and analytical aspects of dinotefuran were considered for the first time by the present Meeting. The residue evaluation was scheduled for the 2012 JMPR by the Forty-third Session of the CCPR (ALINORM 11/34/24)

Dinotefuran is an insecticide used for the control a range of sucking insects, such as whiteflies, plant bugs, leafhoppers and mealybugs, in vegetables, fruit, paddy rice and turf. The formulated products can be applied to foliage, soil, nursery boxes and to paddy water by spray, drench, broadcast and 'pricking-in-hole' treatment. The Meeting received information on identity, animal and plant metabolism, environmental fate in soil, rotational crops, analytical methods, storage stability, use patterns, supervised trials, farm animal feeding studies and fate of residues in processing.

(*RS*)-1-methyl-2-nitro-3-(tetrahydro-3-furylmethyl) guanidine



Dinotefuran is a 1:1 mixture of the enantiomers.

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

UF	1-methyl-3-(tetrahydro-3-furylmethyl) urea
DN	1-methyl-3-(tetrahydro-3- furylmethyl) guanidium dihydrogen
446-DO	1-[4-hydroxy-2-(hydroxymethyl) butyl]-3-methyl-2-nitroguanidine
FNG	2-nitro-1-(tetrahydro-3-furylmethyl) guanidine
PHP	6-hydroxy-5-(2-hydroxyethyl)-1-methyl-1, 3-diazinane-2-ylidene-N-nitroamine
MNG	1-methyl-2-nitroguanidine
NG	Nitroguanidine
MG	1-methylguanidine
BCDN	3-(methylamino)-9-oxa-2-aza-4-azoniabicyclo [4.3.0] non-3-ene hydrogen
UF-DO	1-[4-hydroxy-2-(hydroxymethyl) butyl]-3-methylurea

DN-OH 1-(2-hydroxytetrahydro-3-furylmethyl)-3-methylguanidine (DN-2-OH)
or 1-(3-hydroxytetrahydro-3-furylmethyl)-3-methylguanidine (DN-3-OH)

Animal metabolism

The Meeting received animal metabolism studies with dinotefuran in rats, lactating goats and laying hens. The metabolism and distribution of dinotefuran in animals were investigated using the [^{14}C-furanyl] and [^{14}C-guanidine]-labelled dinotefuran.

Metabolism in rats was summarized and evaluated by the WHO panel of the JMPR in 2012.

Lactating goats were dosed with ^{14}C-furanyl and ^{14}C-guanidine dinotefuran at a dose equivalent to approximately 10 ppm in the diet once daily for 5 consecutive days. Gelatin capsules containing ^{14}C-furanyl and ^{14}C-guanidine dinotefuran were administered orally via a balling gun. The majority of the dose was rapidly excreted in urine and faeces. Radioactive residues in the faeces, cage wash and urine accounted for 81.9% of the total administered dose.

Total ^{14}C residues in the milk accounted for 0.3% of the administered dose. Tissues contained approximately 1.1% of the administered dose. The remaining 16.6% of the administered radioactivity resided in the gastrointestinal tract and its contents.

The total radioactive residue (TRR) levels in milk reached a steady state of approximately 0.045 mg eq/kg by Day 2 of dosing. The residue levels detected in milk, liver, kidney, fat, muscle, blood and heart were 0.012–0.27 mg eq/kg and accounted for 0.01–0.73% of the administered dose.

Dinotefuran was the major component in milk (40.1% TRR, 0.018 mg/kg) with a number of minor metabolites (PHP, 446-DO, UF and FNG) detected but all were < 10% TRR and < 0.005 mg eq/kg. Bound residues were accounted for only 0.001 mg eq/kg in the post extraction solids (PES).

Dinotefuran was present as the major residue in muscle (0.018 mg/kg, 41.3% TRR), fat (0.002 mg/kg, 20.0% TRR), liver (0.017 mg/kg, 12.1% TRR) and kidney (0.035 mg/kg, 12.7% TRR). UF was also a major metabolite in muscle (0.006 mg eq/kg, 14.6% TRR). FNG was the major metabolite detected in the kidney (0.055 mg eq/kg, 20.1% TRR). No other individual compounds accounted for more than 10% of TRR in the tissues.

Laying hens were orally dosed with ^{14}C-furanyl and ^{14}C-guanidine dinotefuran at a dose equivalent to 10 ppm in the feed for 5 consecutive days. The majority of the dose was rapidly eliminated in the excreta. Radioactive residues in the excreta and cage wash accounted for 88.9% of the total administered dose.

Total ^{14}C residues in the eggs accounted for 0.07% (0.02% in the yolk and 0.05% in the white) of the administered dose. Tissues contained approximately 0.23% of the administered dose. The gastrointestinal tract and its contents accounted for 1.31% of the administered radioactivity residues. The residue levels detected in egg white, egg yolk, liver, fat, muscle and blood were 0.007–0.13 mg eq/kg and accounted for 0.01–0.12% of the administered dose.

Dinotefuran was the major residue component at 57.0% TRR (0.013 mg/kg) in egg white and 44.2% TRR (0.0071 mg/kg) in egg yolk. FNG was only metabolite in egg white (0.0030 mg eq/kg, 13.1% TRR) and egg yolk (0.0013 mg eq/kg, 8.0% TRR). All other components were detected at < 0.003 mg eq/kg in egg white and egg yolk. Bound residues were accounted for only 0.0012–0.0028 mg eq/kg in the post-extraction solids (PES).

Dinotefuran was detected at 9.1% TRR (0.0049 mg/kg) in muscle, 10.8% TRR (0.0012 mg/kg) in fat and 9.3% TRR (0.011 mg/kg) in liver. Minor metabolites FNG, UF and DN were identified at 2.2–7.4% TRR (0.0006–0.0080 mg eq/kg) in muscle, fat and liver (DN was not detected in fat). All other components were also detected at < 0.01 mg eq/kg in those tissues.

In animal metabolism studies, dinotefuran was metabolized to several compounds. Dinotefuran was the most important component in milk and muscle for lactating goat, and in eggs for laying hens.

Plant metabolism

The Meeting received plant metabolism studies performed on apples, lettuce, rice and oilseed rape with dinotefuran ^{14}C-labeled in two positions (^{14}C-furanyl and ^{14}C-guanidine).

In an apple metabolism study under field conditions, apple trees were treated with a foliar spray using a 20% SG formulation at a nominal rate of 200 g ai/ha (1× rate) on one tree and at an exaggerated rate treatment at 2000 g ai/ha (10× rate) on the second tree to generate metabolites for identification. The trees were treated 21 days before mature fruit harvest.

The overall residue levels (TRR) in the harvested apples were 0.15 mg/kg for fruits from the apple tree treated at the nominal application rate (1×) and 1.9 mg/kg in apples from exaggerated rate (10×) treatment. Dinotefuran was one of the major ^{14}C residues present and accounted for approximately 29–33% (0.044–0.63 mg/kg) of the total radioactivity in the harvested apples. UF was identified as the major metabolite, accounted for 0.031 mg/kg, 20.0% TRR at 1× rate and 0.40 mg/kg, 20.9% TRR at 10× rate. DN was also identified at a concentration ranging from 0.016 mg/kg, 10.4% TRR at 1× rate and 0.13 mg/kg, 6.9% TRR at 10× rate. PHP was also present at > 10% TRR (0.021 mg/kg, 13.5% TRR from 1×; 0.25 mg/kg, 13.2% TRR from 10×). NG, MNG, 446-DO, BCDN, UF-DO and FNG were identified in the apple fruit as minor metabolites (< 5% TRR).

In a lettuce metabolism study, under greenhouse conditions was carried out to generate metabolites for identification. The test substance was applied as a foliar spray using a 20% SG formulation at a nominal rate equivalent to 150 g ai/ha and at an exaggerated rate equivalent to 1500 g ai/ha. Lettuce plants were treated 14 days before mature harvest (approximately 8 weeks from seeding).

Residue levels in the mature lettuce were 1.8 and 11 mg/kg for the 150 and 1500 g ai/ha applications, respectively. The extracted radioactivity from the mature lettuce accounted for 97.6 and 98.0% TRR for the 150 and 1500 g ai/ha applications, respectively. Dinotefuran was the major residue in the mature lettuce, accounted for 1.1 mg/kg (61.6% TRR) and 6.9 mg/kg (64.7% TRR) for the 150 and 1500 g ai/ha applications, respectively. PHP, 446-DO, UF, DN-OH, BCDN, DN, NG and MNG were detected as metabolites in the mature lettuce, but none of these metabolites were present at greater than 5% TRR level. Among these metabolites, relatively higher amounts of PHP, UF and DN were detected as compared to the other minor metabolites, approaching 4–5% of the TRR level.

In a potato metabolism study under field conditions, the application of test item was performed by foliar spray at rates equivalent to 100 g ai/ha, 200 g ai/ha (six plants each), or 1000 g ai/ha (exaggerated dose; one plant) at the BBCH stage 50–59 (just before flowering). One potato plant at each of the two lower application rates was harvested 54 days after treatment. The remaining five plants per dosage as well as the plant of exaggerated dose were harvested at maturity (75 days after treatment).

The TRR in peel and pulp (peeled potato tubers) was determined. The TRR in the whole potato was very low for the 100 and 200 g ai/ha treatment rates, even at the first harvest interval. The majority of the radioactive residue was extracted with acetonitrile/water followed by water (94.5% to 97.7% of TRR for all samples). In the first harvest, dinotefuran accounted for 13.1% TRR (0.004 mg/kg) at 100 g ai/ha and 8.5% (0.003 mg/kg) at 200 g ai/ha application rate. One very polar fraction (M3) was determined with 61.7% TRR (0.019 mg/kg) and 57.1% TRR (0.021 mg/kg) at application rates of 100 and 200 g ai/ha, respectively. This fraction could be characterised to consist of traces of NG and at least six further unknown fractions each being less than 10% of the TRR. MNG was found at 7.6% TRR (0.002 mg/kg) and 9.6% TRR (0.003 mg/kg) at both application rates. Four other metabolites characterized as UF, PHP, 446-DO and FNG were all less than 7% TRR. The metabolic pattern of the mature potatoes was qualitatively similar to that of the first harvest, but the concentrations of dinotefuran and metabolites were even lower.

In a rice metabolism study under greenhouse conditions, simulating rice-paddy application of dinotefuran were performed to quantify total ^{14}C levels in rice plant tissue and grain and identify major components of the residue and their distribution in the plant.

There were two application times (5 and 20 days after bolting, DAB) and two application methods (soil and foliar spray application). Extracts of whole rice grain, brown rice, polished rice, chaff, bran, straw, root and soil were analysed to determine the nature of metabolites and their relative distribution in the samples. The results were similar for both application times (5 and 20 DAB).

Residue levels in whole grain from the soil applications were 0.35–0.40 mg/kg. Residues were greatly reduced when the chaff was removed from the whole grain and further reduced upon polishing of the brown rice to produce polished rice. The residue levels were 0.052–0.055 mg/kg in brown rice, and 0.033–0.039 mg/kg in polished rice. Dinotefuran was the major residue in whole rice grain and brown rice from the soil applications. The level of dinotefuran residues ranged from 0.23 mg/kg (66.0% TRR) to 0.24 mg/kg (60.5% TRR) in the whole grain, and 0.014 mg/kg (26.2% TRR) to 0.015 mg/kg (26.3% TRR) in the brown rice. After polishing of the brown rice, to produce polished rice, the residues of dinotefuran decreased to 0.008–0.010 mg/kg (21.2–29.9% TRR). PHP, 446-DO, UF, DN-OH, BCDN and DN were detected as the minor metabolites in whole grain and brown rice (< 10% TRR). PHP, 446-DO, UF and DN were also detected in polished rice at the level of less than 8% TRR.

The TRR in rice straw were 1.3–1.8 mg/kg for the soil applications. Dinotefuran was the major residue in straw, accounting for 0.70–0.97 mg/kg (51.6–53.0% TRR). UF and DN were observed as the major metabolites in straw. UF accounted for 0.18–0.22 mg/kg (13.4–11.8% TRR), while DN accounted for 0.089–0.091 mg/kg (6.6–5.0% TRR). PHP, 446-DO, DN-OH and BCDN were detected as the minor metabolites (< 5% TRR).

The TRR in the whole rice grain for the foliar spray applications accounted for 5.1–5.8 mg/kg. Residues were greatly reduced during processing to produce the brown rice and polished rice fractions, similar to what was observed for the soil application. The residue levels in brown rice were 0.34–0.61 mg/kg, while the residue levels in polished rice were 0.15–0.34 mg/kg. Dinotefuran was the major residue in whole rice grain and brown rice. The level of dinotefuran ranged from 2.1 mg/kg (35.9% TRR) to 2.7 mg/kg (52.7% TRR) in the whole grain, and 0.18 mg/kg (53.6% TRR) to 0.20 mg/kg (33.4% TRR) in the brown rice. The residues of dinotefuran were further reduced to 0.073 mg/kg (48.4% TRR) to 0.14 mg/kg (41.7% TRR) in the polished rice. UF were observed as the major metabolite, accounting for 0.70–1.0 mg/kg (13.7–17.2% TRR) in whole grain, 0.048–0.1 mg/kg (14.1–17.2% TRR) in brown rice, and 0.021–0.076 mg/kg (14.2–22.8% TRR) in polished rice. PHP, 446-DO, DN-OH, BCDN and DN were detected as the minor metabolites in whole grain, brown rice and polished rice (< 8% TRR).

The TRR in rice straw were 7.6–8.1 mg/kg for the foliar spray applications. Dinotefuran was the major residue in straw, accounting for 4.0–5.6 mg/kg (53.3–69.0% TRR). UF and DN were observed as the major metabolites in straw. UF accounted for 0.72–1.2 mg/kg (8.8–15.9% TRR), while DN accounted for 0.47–0.65 mg/kg (5.7–8.5% TRR). PHP, 446-DO, DN-OH and BCDN were detected as the metabolites in straw (< 5% TRR).

In an oilseed rape metabolism study under natural conditions, oilseed rape plants were treated at pre-flowering (growth stage 50–59) by foliar application with formulated ^{14}C-dinotefuran at doses of 100 g ai/ha (low dose), 200 g ai/ha (high dose) and 1000 g ai/ha (exaggerated dose, plant pot). Planting, cultivation and harvesting of the mature winter rape plants was carried out according to common agricultural practice.

The TRR in the whole rape plant was 0.21, 0.49 and 2.1 mg/kg for the 100, 200 and 1000 g ai/ha treatment rates, respectively. For seeds, the residue levels for the 100, 200 and 1000 g ai/ha doses were determined to be 0.055, 0.13 and 0.70 mg/kg, respectively. Accordingly, residue levels for foliage were determined to be 0.26, 0.65 and 2.4 mg/kg, respectively.

In seeds dinotefuran was found at levels of 0.006 mg/kg (14.8% TRR), 0.016 mg/kg (18.7% TRR) and 0.095 mg/kg (18.0% TRR) for doses 100, 200 and 1000 g ai/ha, respectively. MNG was

found as the most significant fraction, amounting to 0.005 mg/kg (12.4% TRR), 0.004 mg/kg (4.8% TRR) and 0.071 mg/kg (13.4% TRR) in seeds for the three application rates respectively. None of the other radioactive fractions exceeded 8.2% of TRR. UF, PHP, FNG, MG, DN and BCDN were found at low concentrations not exceeding 0.003 mg/kg for the low or high dosed plants with the exception of PHP amounting to 0.006 mg/kg.

In foliage dinotefuran was found at levels of 0.025 mg/kg (11.3% TRR), 0.094 mg/kg (16.9% TRR) and 0.22 mg/kg (10.6% TRR) for doses 100, 200 and 1000 g ai/ha, respectively. Radioactive fractions M3 (at least 8 compounds), MG and DN were found as significant metabolite fractions. M3 amounted to 0.12 mg/kg (53.4% TRR), 0.27 mg/kg (48.8% TRR) and 0.62 mg/kg (29.5% TRR) for the three application rates respectively. MG was found at 0.007 mg/kg (8.7% TRR), 0.010 mg/kg (4.9% TRR) and 0.088 mg/kg (11.5% TRR) for the three application rates respectively. DN was 0.037 mg/kg (13.2% TRR), 0.11 mg/kg (15.0% TRR) and 0.46 mg/kg (17.4% TRR) for the three application rates respectively. Furthermore detected in foliage at a dose of 1000 g ai/ha were UF, MNG (one of the compounds in M3) and BCDN amounting to 0.14 mg/kg (8.7% TRR), 0.14 mg/kg (6.5% TRR) and 0.043 mg/kg (2.7% TRR), respectively.

In the plant metabolism studies on apples, lettuce, potato, rice and oilseed rape, dinotefuran was the major component of the residues found in apples, lettuce, potato, rice (grain and straw) and oilseed rape (seed). UF and DN were significant components (> 10% TRR) in apples, rice (grain and straw) and foliage of oilseed rape. MNG was present at more than 10% TRR in seeds of oilseed rape but its concentration was less than 0.01 mg/kg at a normal rate. UF-DO and NG were only found in plants but the contributions were insignificant (< 5% TRR).

Environmental fate in soil

The Meeting received information on aerobic soil metabolism, soil photolysis and rotational crop study.

Dinotefuran degraded in soil under the aerobic conditions employed, principally via cleavage of tetrahydromethyl portion of the molecule to MNG and demethylation of MNG to NG. The DT_{50} was 10–52 days at 20 °C to 25 °C.

The photolysis study indicated that photolysis was not a significant degradation pathway for dinotefuran in soil incubated at 20 °C.

In confined rotational crop study, rotational crops (radish, lettuce, sorghum and wheat) were planted at 30 and 120 days after treatment. The test substance was applied as one broadcast spray application at a rate of 0.60 kg ai/ha in a volume of 1062 L/ha.

The TRR found in each of the plant fractions amounted to as much as 1.3 mg/kg in 30 day immature radish leaves to as little as 0.003 mg/kg in 120 day lettuce (mature and immature). The TRR was less than 0.01 mg/kg in all of the 120 day plant sample fractions except for the radish leaf samples where it was 0.035 mg/kg in the immature samples and 0.026 mg/kg in the mature samples.

For the 30 day after the application, in the immature radish samples, dinotefuran was present as the highest concentration of any individual components in the leaf and root samples (0.33 and 0.019 mg/kg, respectively). In the immature lettuce samples, BCDN and UF metabolites were found at the highest concentration (0.027 mg/kg for both). In the immature sorghum, the highest residue level was for dinotefuran followed by BCDN, DN, PHP, 446-DO, UF and MNG (0.20, 0.19, 0.14, 0.13, 0.080, 0.069 and 0.017 mg/kg, respectively).

In the mature radish samples (leaf and root), DN was present at the highest concentration (0.074 and 0.007 mg/kg, respectively). In the mature lettuce samples, PHP was found to be present at the highest concentration (0.083 mg/kg) followed by dinotefuran, BCDN and DN (0.064, 0.061 and 0.033 mg/kg, respectively). The mature sorghum samples analyses indicated extracted residues of 0.15, 0.12 and 0.034 mg/kg in forage, chaff and grain. The extracted residues consisted of BCDN and DN in forage and chaff as well as an unidentified component known as M16.

There were no individual components seen at concentrations above 0.006 mg/kg in any matrix in the 120 day samples.

Methods of analysis

The Meeting received description and validation data for analytical methods for residues of parent dinotefuran and its metabolites (DN and UF) in raw agricultural commodities, processed commodities, feed commodities and animal commodities. In most of the methods for determination of dinotefuran, UF and DN, homogenized samples were extracted with acetonitrile/water, and the extract was cleaned up with liquid –liquid partition followed by column chromatography using SPE cartridges. Residues were determined by HPLC with UV or MS/MS detection. The methods of analysis for a range of substrates were validated with LOQs of the 0.01 mg/kg for dinotefuran, UF and DN.

The multiresidue method with GC employing NPD or ECD detection was validated for dinotefuran in plant materials (non-fatty and fatty). LOQs were 0.01 mg/kg for dinotefuran.

Stability of residues in stored analytical samples

The Meeting received information on the freezer storage stability of dinotefuran and its metabolites (DN and UF) in plant (apple, peach, grape, cranberry, bulb onion, green onion, broccoli, melon, tomato, lettuce, watercress, potato, rice grain and cotton seed), their processed (rice bran, cotton meal and refined oil) commodities and animal products.

Storage stability results indicate that dinotefuran and its metabolites (DN and UF) residues were stable for at least 4 days in milk and eggs, for at least 2 months in bovine tissues, for at least 3 months in rice bran, for at least 4 months in grape and broccoli, for at least 5 months in melon, for at least 9 months in cranberry, for at least 12 months in apple, tomato, lettuce, potato and cotton (seeds, meal and oil), for at least 14 months in rice grain, for at least 20 months in watercress, for at least 22 months in green onion, for at least 24 months in bulb onion, and for at least 26 months in peach.

The periods of storage stability studies cover the sample storage intervals of residue trials.

Definition of the residue

In the lactating goat metabolism study, TRRs in kidney (0.27 mg/kg) and liver (0.14 mg/kg) were higher than those in milk (0.04–0.05 mg/kg), heart (0.05 mg/kg), muscle (0.04 mg/kg) and fat (0.01 mg/kg). Dinotefuran is the major component of the residue in muscle (41% TRR), milk (40% TRR), fat (20% TRR), kidney (13% TRR) and liver (12% TRR). FNG is the major component of the residue in kidney (20% TRR) but less than 7% TRR in all other tissues and milk.

In the lactating cow feeding study, the mixture of dinotefuran, UF and DN (3:1:1) was administered. Livestock are expected to be exposed to a mixture of dinotefuran, UF and DN at approximately 3:1:1 ratio from animal feedstuffs such as rice straw or cotton gin trash. UF was the predominant residue in tissues and milk. No detectable residues of dinotefuran occurred in any tissue samples and low concentrations of dinotefuran occurred in milk at the highest dose group (10×) only. Low concentrations of DN (0.02–0.04 mg/kg) were detected in liver, kidney and muscle from animals in the highest dose group only (10×). DN was occasionally found at very low concentrations (0.01 mg/kg) in whole milk. The concentrations of UF in tissues and milk are at least 10 times higher than those of dinotefuran at a highest dose. The concentrations of UF in tissues are approximately 10 times higher than those of DN at a highest dose.

In the laying hen metabolism study, TRRs were the highest for liver (0.13 mg/kg), followed by muscle (0.05 mg/kg), eggs (0.01–0.02 mg/kg for egg white, < 0.002–0.02 mg/kg for egg yolk) and fat (0.01 mg/kg). Dinotefuran is the major residue component in eggs (57% TRR for egg white, 44% TRR for egg yolk). FNG is present in egg white (13% TRR) but its concentration is very low (0.003 mg/kg). In other tissues (muscle, liver and fat), dinotefuran is detected at 9.1–11% TRR. However, all components were detected at ≤0.01 mg/kg (< 10% TRR) in all tissues and eggs.

The analytical methods for animal products submitted to the Meeting can quantify dinotefuran, UF and DN individually using the same analytical method.

The Meeting decided that parent dinotefuran and metabolite UF are suitable analytes for enforcement purposes and dietary risk assessment in animal commodities.

The octanol/water coefficient (log Pow) of dinotefuran is -0.6 at 25 °C (pH 7). In the lactating goat and laying hen metabolism study, dinotefuran residues in muscle were at least 4 times higher than those in fat. In the lactating cow feeding study, dinotefuran residues in skimmed milk were at least twice higher than those in cream. The Meeting considered the residue of dinotefuran is not fat soluble.

Parent dinotefuran was a major component (11–66% TRR) in apple, lettuce, potato, rice grains, rape seeds and foliage. UF, DN and MNG were detected as major metabolites in some matrices. UF was present in apple (21% TRR), rice grains (17% TRR for the spray application) and rice straw (16% TRR for the spray application, 13% TRR for the soil application,). DN was present in apple (10% TRR) and rape foliage (17% TRR). MNG was found in rape seeds (12%) but at very low concentration at a normal rate (< 0.01 mg/kg). No other radioactive components in the extracts from plant matrices were individually present at more than 10% TRR.

The results of the trials indicated that UF and DN residues were generally less than 10% of parent dinotefuran residue in food commodity matrices. These findings agree with the information obtained from the metabolism studies. The Meeting assumed the toxicity of UF and DN was comparable to that of dinotefuran.

The Meeting decided that parent dinotefuran is a suitable analyte for enforcement purposes and dinotefuran, UF and DN are suitable analytes for dietary risk assessment in plant commodities.

The Meeting recommended the following residue definition:

For plants:

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

Definition of the residue (for estimation of dietary intake): *Sum of dinotefuran, 1-methyl-3-(tetrahydro-3-furylmethyl) urea (UF) and 1-methyl-3-(tetrahydro-3-furylmethyl) guanidium dihydrogen (DN) expressed as dinotefuran*

For animal:

Definition of the residue (for compliance with the MRL and for estimation of dietary intake): *sum of Dinotefuran and 1-methyl-3-(tetrahydro-3-furylmethyl) urea (UF) expressed as dinotefuran*

The residue is not fat soluble

Residue of supervised residue trials on crops

The Meeting received supervised trial data for the foliar application and soil application (irrigation) of dinotefuran on peaches, grapes, cranberries, bulb onions, green onions, broccoli, cauliflowers, cabbages, cucumbers, melons, summer squashes, zucchinis, peppers, tomatoes, lettuces, spinach, watercress, potatoes, celery, rice and cotton. Residue trial data was made available from Japan and the USA.

Labels were available from Japan, Korea and the USA describing the registered uses of dinotefuran.

The OECD calculator was used as a tool to assist in the estimation of maximum residue levels from the selected residue data set obtained from the supervised residue trials. As a first step, the Meeting reviewed the trial conditions and other relevant factors related to each data set to arrive at a best estimate of the maximum residue level using expert judgement. Then the OECD calculator was employed. If the statistical calculation spreadsheet suggested a different value, a brief explanation of the deviation was supplied.

The residue concentrations are reported for dinotefuran, UF and DN. Total residues for estimation of STMRs are calculated by summing up the concentrations of dinotefuran, UF and DN.

In case that the residues of dinotefuran were found at high levels, UF and DN were also detected and the residue levels of both metabolites depend on the commodity.

Since the residue values were expressed as mg of the analyte/kg sample, UF and DN need to be converted into dinotefuran equivalent. The conversion factors are 1.3 (202.21/158.20=1.28) for UF and 1.3 (202.21/157.22=1.29) for DN. Residues of < LOQ for both analytes are not converted.

The method for calculation of the total residues for plant commodities is illustrated below.

Dinotefuran	UF	DN	Total
< 0.01	< 0.01	< 0.01	< 0.03 (0.01 + 0.01 + 0.01)
0.051	< 0.01	< 0.01	0.071 (0.051 + 0.01 + 0.01)
0.056	0.011	< 0.01	0.080 (0.056 + 0.011 × 1.3 + 0.01)
0.14	0.016	0.010	0.17 (0.14 + 0.016 × 1.3 + 0.010 × 1.3)

Stone fruits

Peach

Data were available from supervised trials on peaches in the USA.

The US GAP on peach and nectarine is for a soil application at a maximum rate of 0.30 kg ai/ha with a PHI of 21 days and a foliar application at a maximum rate of 0.20 kg ai/ha with a PHI of 3 days. The maximum seasonal rate is 0.30 kg ai/ha for soil application and 0.30 kg ai/ha for foliar application. The seasonal rate for foliar application of the trials was slightly higher (33%) than the US GAP. The Meeting agreed to use the residue data for foliar application because the contribution of the first foliar spray to the final residue was less than 20% based on the decline study.

Dinotefuran residues in peaches from trials in the USA approximating GAP were (n = 7): 0.09, 0.21 (2), 0.24, 0.31, 0.35 and 0.47 mg/kg.

Total residues in peaches were (n = 7): 0.11, 0.23, 0.25, 0.28, 0.35, 0.37 and 0.57 mg/kg.

Based on the trials for peaches in the USA, the Meeting estimated a maximum residue level, an STMR value and an HR value for dinotefuran in peach of 0.8, 0.28 and 0.57 mg/kg respectively.

The Meeting agreed to extrapolate these recommendations to nectarine.

Berries and other small fruits

Grape

Data were available from supervised trials on grapes in the USA.

The US GAP on Berry and Small fruit (Small fruit vine climbing, except fuzzy kiwifruit) is for a soil application at a maximum rate of 0.38 kg ai/ha with a PHI of 28 days and two foliar applications at a maximum rate of 0.15 kg ai/ha (at a maximum seasonal rate of 0.30 kg ai/ha for foliar application) with a PHI of 1 day.

Dinotefuran residues in grapes from trials in the USA matching GAP were (n = 13): 0.087, 0.10, 0.11, 0.12, 0.16, 0.17, 0.20, 0.22, 0.27 (2), 0.40, 0.52 and 0.55 mg/kg.

Total residues in grapes were (n = 13): 0.11, 0.12, 0.13, 0.14, 0.18, 0.19, 0.22, 0.24, 0.29 (2), 0.47, 0.57 and 0.67 mg/kg.

Based on the trials for grapes in the USA, the Meeting estimated a maximum residue level, an STMR value and an HR value for dinotefuran in grape of 0.9, 0.22 and 0.67 mg/kg respectively.

Cranberry

Data were available from supervised trials on cranberries in the USA.

The GAP on Berry and Small fruit (Low growing berry subgroup, except strawberry) of the USA is a foliar application at a maximum rate of 0.20 kg ai/ha (at a maximum seasonal rate of 0.40 kg ai/ha) with a PHI of 7 days.

Dinotefuran residues in cranberries from trials in the USA matching GAP were (n = 4): 0.01, 0.04, 0.05 and 0.06 mg/kg.

Total residues in cranberries were (n = 4): 0.03, 0.06, 0.07 and 0.10 mg/kg.

Based on the trials for cranberries in the USA, the Meeting estimated a maximum residue level, an STMR value and an HR value for dinotefuran in cranberry of 0.15, 0.065 and 0.10 mg/kg respectively.

Bulb vegetables

Onion, Bulb

Data were available from supervised trials on bulb onions in the USA.

The GAP on Onion, bulb and green of the USA is a soil application at a maximum rate of 0.30 kg ai/ha at planting and a foliar application at a maximum rate of 0.20 kg ai/ha with a PHI of 1 day. The maximum seasonal rate is 0.30 kg ai/ha for each application. The maximum seasonal rate is 0.43 kg ai/ha regardless of application method. The seasonal rate for foliar application of the trials was slightly higher (33%) than the US GAP. The Meeting accepted the residue data because it would accommodate the residue from potential combination from soil and foliar applications.

Dinotefuran residues in bulb onions from trials in the USA approximating GAP were (n = 8): < 0.01 (2), 0.01, 0.02 (3), 0.04 and 0.06 mg/kg.

Total residues in bulb onions were (n = 8): < 0.03 (2), 0.03, 0.04 (3), 0.06 and 0.09 mg/kg.

Based on the trials for bulb onions in the USA, the Meeting estimated a maximum residue level, an STMR value and an HR value for dinotefuran in onion, bulb of 0.1, 0.04 and 0.09 mg/kg respectively.

Onion, green

Data were available from supervised trials on green onions in the USA.

The GAP on Onion, bulb and green of the USA is a soil application at a maximum rate of 0.30 kg ai/ha at planting and a foliar application at a maximum rate of 0.20 kg ai/ha with a PHI of 1 day. The maximum seasonal rates are 0.30 kg ai/ha for each application. The residues from the trial for the soil application are insignificant, compared those for the foliar application. The Meeting decided to estimate a maximum residue level, an STMR and an HR based on the residue data for the foliar application.

Dinotefuran residues in green onions from trials in the USA matching GAP were (n = 5): 0.086, 0.22, 0.52, 1.3 and 1.9 mg/kg.

Total residues in green onions were (n = 5): 0.12, 0.59, 0.91, 1.5 and 2.3 mg/kg.

Based on the trials for green onions in the USA, the Meeting estimated a maximum residue level, an STMR value and an HR value for dinotefuran in spring onion of 4, 0.91 and 2.3 mg/kg respectively.

Brassica (cole or cabbage) vegetables, Head cabbages, Flowerhead cabbages

Broccoli and Cauliflower

Data were available from supervised trials on broccoli and cauliflowers in the USA.

The GAP of the USA for Head and stem brassica is for a soil application at a maximum rate of 0.37 kg ai/ha with a PHI of 21 days (at a maximum seasonal rate of 0.60 kg ai/ha for soil application) or a foliar application at a maximum rate of 0.20 kg ai/ha (at a maximum seasonal rate of 0.30 kg ai/ha for foliar application) with a PHI of 1 day. Only one application method can be used.

Dinotefuran residues in broccoli and cauliflowers from trials for the foliar application in the USA, matching GAP were (n = 6): 0.49 and 1.0 (2) mg/kg for broccoli, and 0.086, 0.20 and 0.36 mg/kg for cauliflowers. Dinotefuran residues in broccoli from trials for the soil application in the USA matching GAP were (n = 2): < 0.01 and 0.059 mg/kg.

Total residues in broccoli and cauliflowers for the foliar application were (n = 6): 0.56, 1.0 and 1.1 mg/kg for broccoli, and 0.11, 0.22 and 0.41 mg/kg for cauliflowers. Total residues in broccoli for the soil application were (n = 2): < 0.03 and 0.079 mg/kg.

Cabbage, Head

Data were available from supervised trials on head cabbages in the USA.

The GAP on Head and stem brassica of the USA is a soil application at a maximum rate of 0.37 kg ai/ha with a PHI of 21 days (at a maximum seasonal rate of 0.60 kg ai/ha for soil application) or a foliar application at a maximum rate of 0.20 kg ai/ha (at a maximum seasonal rate of 0.30 kg ai/ha for foliar application) with a PHI of 1 day. Only one application method can be used.

Dinotefuran residues in head cabbages from trials for the foliar application in the USA matching GAP were (n = 6): 0.013, 0.036, 0.22, 0.25, 0.78 and 0.85 mg/kg. Dinotefuran residue in cabbages from trials for the soil application in the USA matching GAP was 0.17 mg/kg.

Total residues in head cabbages for the foliar application were (n = 6): 0.033, 0.056, 0.28, 0.38, 0.90 and 1.1 mg/kg. Total residue in head cabbages for the soil application was 0.34 mg/kg.

The GAP is the same for broccoli, cauliflower and head cabbage. The Meeting considered that the residues from trials with the foliar application on broccoli/cauliflowers and head cabbages were similar. The Meeting agreed to explore a group maximum residue level for brassica (cole or cabbage) vegetables, head cabbages, flowerhead cabbages.

Since the residue populations from trials on broccoli/cauliflowers and head cabbages were not significantly different (Mann-Whitney U-test), the Meeting agreed that they could be combined. The residues of dinotefuran in those brassica vegetables for the foliar application were (n = 12): 0.013, 0.036, 0.086, 0.20, 0.22, 0.25, 0.36, 0.49, 0.78, 0.85 and 1.0 (2) mg/kg. Total residues for the foliar application were (n = 12): 0.033, 0.056, 0.11, 0.22, 0.28, 0.38, 0.41, 0.56, 0.90, 1.0 and 1.1 (2) mg/kg

Based on the trials for broccoli/cauliflowers and head cabbages in the USA, the Meeting estimated a maximum residue level, an STMR value and an HR value for dinotefuran in brassica (cole or cabbage) vegetables, head cabbages, flowerhead cabbages of 2, 0.40 and 1.1 mg/kg respectively.

Fruiting vegetables, Cucurbits

Cucumber

Data were available from supervised trials on cucumbers in the USA.

The GAP on Cucurbits of the USA is a soil application at a maximum rate of 0.37 kg ai/ha with a PHI of 21 days (at a maximum seasonal rate of 0.60 kg ai/ha for soil application) or a foliar application at a maximum rate of 0.20 kg ai/ha (at a maximum seasonal rate of 0.30 kg ai/ha for foliar application) with a PHI of 1 day. Only one application method can be used.

Dinotefuran residues in cucumbers from trials for the foliar application in the USA matching GAP were (n = 7): 0.13, 0.14, 0.17, 0.18 (2), 0.20 and 0.21 mg/kg. Dinotefuran residue in cucumbers from trials for the soil application in the USA matching GAP was 0.053 mg/kg.

Total residues in cucumbers for the foliar application were (n = 7): 0.18, 0.26 (3), 0.28 (2) and 0.33 mg/kg. Total residue in cucumbers for the soil application was 0.073 mg/kg.

Melon

Data were available from supervised trials on melons in the USA.

The GAP on Cucurbits of the USA is a soil application at a maximum rate of 0.37 kg ai/ha with a PHI of 21 days (at a maximum seasonal rate of 0.60 kg ai/ha for soil application) or a foliar application at a maximum rate of 0.20 kg ai/ha (at a maximum seasonal rate of 0.30 kg ai/ha for foliar application) with a PHI of 1 day. Only one application method can be used.

Dinotefuran residues in melons from trials for the foliar application in the USA matching GAP were (n = 6): 0.042, 0.054, 0.082, 0.15, 0.18 and 0.20 mg/kg. Dinotefuran residue in melons from trials for the soil application in the USA matching GAP was 0.040 mg/kg.

Total residues in melons for the foliar application were (n = 6): 0.073, 0.11, 0.16, 0.23, 0.24 and 0.32 mg/kg. Total residue in melons for the soil application was 0.060 mg/kg.

Summer squash and Zucchini

Data were available from supervised trials on summer squashes and zucchinis in the USA.

The GAP on Cucurbits of the USA is a soil application at a maximum rate of 0.37 kg ai/ha with a PHI of 21 days (at a maximum seasonal rate of 0.60 kg ai/ha for soil application) or a foliar application at a maximum rate of 0.20 kg ai/ha (at a maximum seasonal rate of 0.30 kg ai/ha for foliar application) with a PHI of 1 day. Only one application method can be used.

Dinotefuran residues in summer squashes and zucchini from trials for the foliar application in the USA matching GAP were (n = 5): 0.092, 0.15 and 0.18 mg/kg for summer squashes, and 0.10 and 0.15 mg/kg for zucchini. Dinotefuran residues in summer squashes from trials for the soil application in the USA matching GAP were (n = 2): 0.041 and 0.087 mg/kg.

Total residues in summer squashes and zucchini for the foliar application were (n = 5): 0.19, 0.30 and 0.32 mg/kg for summer squashes, and 0.14 and 0.22 mg/kg for zucchini. Total residues in summer squashes for the soil application were (n = 2): 0.065 and 0.11 mg/kg.

The GAP is the same for cucumber, melon, summer squash and zucchini. The Meeting considered that the residues from trials with the foliar application on cucumber, melon, summer squash and zucchini were similar. The Meeting agreed to propose a group maximum residue level for fruiting vegetables, cucurbits.

Since the residue populations from trials on cucumbers, melons, summer squashes and zucchinis for foliar application were not significantly different (Kruskal-Wallis H-test), the Meeting agreed that they could be combined. The residues of dinotefuran in those cucurbits for the foliar application were (n = 18): 0.042, 0.054, 0.082, 0.092, 0.10, 0.13, 0.14, 0.15 (3), 0.17, 0.18 (4), 0.20 (2) and 0.21 mg/kg. Total residues for the foliar application were (n = 18): 0.073, 0.11, 0.14, 0.16, 0.18, 0.19, 0.22, 0.23, 0.24, 0.26 (3), 0.28 (2), 0.30, 0.32 (2) and 0.33 mg/kg.

Based on the trials for cucumbers, melons, summer squashes and zucchinis in the USA, the Meeting estimated a maximum residue level, an STMR value and an HR value for dinotefuran in fruiting vegetables, cucurbits of 0.5, 0.25 and 0.33 mg/kg respectively.

Fruiting vegetables, other than Cucurbits

Peppers

Data were available from supervised trials on sweet peppers and chili peppers in Japan and the USA.

In Japan, dinotefuran is registered for use on sweet pepper and chili pepper at two foliar applications of 0.0067–0.010 kg ai/hL with a PHI of 1 day. Residues in green peppers from trials matching GAP of Japan were (n = 2): 0.43 and 1.2 mg/kg. However, the trials for green peppers matching GAP of Japan were insufficient to estimate a maximum residue level for the commodity.

The GAP on Fruiting vegetables of the USA is a soil application at a maximum rate of 0.37 kg ai/ha with a PHI of 21 days (at a maximum seasonal rate of 0.60 kg ai/ha for soil application) or a foliar application at a maximum rate of 0.20 kg ai/ha (at a maximum seasonal rate of 0.30 kg ai/ha for foliar application) with a PHI of 1 day. Only one application method can be used.

Dinotefuran residues in sweet peppers and chili peppers from trials for the foliar application in the USA matching GAP were (n = 8): 0.030, 0.042, 0.14 (2), 0.25 and 0.27 mg/kg for sweet peppers, and 0.23 and 0.41 mg/kg for chili peppers. Dinotefuran residues in sweet peppers from trials for the soil application in the USA matching GAP were (n = 2): 0.024 and 0.094 mg/kg.

Total residues in sweet peppers and chili peppers for the foliar application were (n = 8): 0.050, 0.062, 0.17, 0.18, 0.29 and 0.50 mg/kg for sweet peppers, and 0.26 and 0.55 mg/kg for chili peppers. Total residues in sweet peppers for the soil application were (n = 2): 0.044 and 0.11 mg/kg.

Tomato

Data were available from supervised trials on tomatoes in Japan and the USA.

In Japan, dinotefuran is registered for use on tomato at an irrigation treatment to nursery box of 0.20 kg ai/hL at planting and two foliar applications of 0.0067–0.010 kg ai/hL with a PHI of 1 day. Residues in tomatoes from trials matching GAP of Japan were (n = 2): 0.094 and 0.35 mg/kg. However, the trials for tomatoes matching GAP of Japan were insufficient to estimate a maximum residue level for the commodity.

The GAP on Fruiting vegetables (except varieties of tomato which are less than 2 inches in size) of the USA is a soil application at a maximum rate of 0.37 kg ai/ha with a PHI of 21 days (at a maximum seasonal rate of 0.60 kg ai/ha for soil application) or a foliar application at a maximum rate of 0.20 kg ai/ha (at a maximum seasonal rate of 0.30 kg ai/ha for foliar application) with a PHI of 1 day. Only one application method can be used.

Dinotefuran residues in tomatoes from trials for the foliar application in the USA matching GAP were (n = 15): 0.039, 0.051, 0.056, 0.060, 0.069 (2), 0.071, 0.084, 0.097, 0.13 (2), 0.14, 0.15, 0.16 (2) mg/kg. Dinotefuran residues in tomatoes from trials for the soil application in the USA matching GAP were (n = 2): 0.015 and 0.045 mg/kg.

Total residues in tomatoes for the foliar application were (n = 15): 0.059, 0.071, 0.080 (2), 0.089, 0.091, 0.10 (2), 0.12, 0.15 (2), 0.18, 0.19 (2) and 0.20 mg/kg. Total residues in tomatoes for the soil application were (n = 2): 0.035 and 0.065 mg/kg.

The GAP is the same for peppers and tomato and the residues of these commodities are similar. The Meeting agreed to propose a group maximum residue level for fruiting vegetables, other than cucurbits except sweet corn and mushrooms.

Since the residue populations from trials on peppers and tomatoes from foliar applications were not significantly different (Mann-Whitney U-test), the Meeting agreed that they could be combined. The residues of dinotefuran in those fruiting vegetables for the foliar application were (n = 23): 0.030, 0.039, 0.042, 0.051, 0.056, 0.060, 0.069 (2), 0.071, 0.084, 0.097, 0.13 (2), 0.14 (3), 0.15, 0.16 (2), 0.23, 0.25, 0.27 and 0.41 mg/kg. Total residues for the foliar application were (n = 23): 0.050, 0.059, 0.062, 0.071, 0.080 (2), 0.089, 0.091, 0.10 (2), 0.12, 0.15 (2), 0.17, 0.18 (2), 0.19 (2), 0.20, 0.26, 0.29, 0.50 and 0.55 mg/kg.

Based on the trials for peppers and tomatoes in the USA, the Meeting estimated a maximum residue level, an STMR value and an HR value for dinotefuran in fruiting vegetables, other than cucurbits except sweet corn and mushrooms of 0.5, 0.15 and 0.55 mg/kg respectively.

Leafy vegetables (including Brassica leafy vegetables)

Lettuce, Leaf

Data were available from supervised trials on leaf lettuce in the USA.

The GAP on Leafy vegetables from the USA is a soil application at a maximum rate of 0.37 kg ai/ha with a PHI of 21 days (at a maximum seasonal rate of 0.60 kg ai/ha for soil application) or a foliar application at a maximum rate of 0.15 kg ai/ha (at a maximum seasonal rate of 0.30 kg ai/ha for foliar application) with a PHI of 7 day. Only one application method can be used.

Dinotefuran residues in leaf lettuce from trials for the foliar application in the USA matching GAP were (n = 8): 0.15, 0.20, 0.21, 0.29, 0.32, 0.91, 1.1 and 2.4 mg/kg. Dinotefuran residues in leaf lettuce from trials for the soil application in the USA matching GAP were (n = 2): 0.016 and 0.11 mg/kg.

Total residues in leaf lettuce for the foliar application were (n = 8): 0.33, 0.63, 0.66, 0.73, 0.81, 1.7, 2.0 and 3.3 mg/kg. Total residues in leaf lettuce for the soil application were (n = 2): 0.039 and 0.20 mg/kg.

Lettuce, Head

Data were available from supervised trials on head lettuce in the USA.

The GAP on Leafy vegetables of the USA is a soil application at a maximum rate of 0.37 kg ai/ha with a PHI of 21 days (at a maximum seasonal rate of 0.60 kg ai/ha for soil application) or a foliar application at a maximum rate of 0.15 kg ai/ha (at a maximum seasonal rate of 0.30 kg ai/ha for foliar application) with a PHI of 7 day. Only one application method can be used.

Dinotefuran residues in head lettuce from trials for the foliar application in the USA matching GAP were (n = 7): 0.08, 0.12, 0.16 (2), 0.18, 0.19 and 0.53 mg/kg. Dinotefuran residue in head lettuce from trials for the soil application in the USA matching GAP was 0.016 mg/kg.

Total residues in head lettuce for the foliar application were (n = 7): 0.25, 0.29 (2), 0.46, 0.49, 0.83 and 1.4 mg/kg. Total residue in head lettuce for the soil application was 0.049 mg/kg.

Spinach

Data were available from supervised trials on spinach in the USA.

The GAP on Leafy vegetables of the USA is a soil application at a maximum rate of 0.37 kg ai/ha with a PHI of 21 days (at a maximum seasonal rate of 0.60 kg ai/ha for soil application) or a foliar application at a maximum rate of 0.15 kg ai/ha (at a maximum seasonal rate of 0.30 kg ai/ha for foliar application) with a PHI of 7 day. Only one application method can be used.

Dinotefuran residues in spinach from trials for the foliar application in the USA matching GAP were (n = 7): 0.43, 0.48, 0.56, 0.62, 1.2, 2.0 and 3.3 mg/kg. Dinotefuran residue in spinach from trials for the soil application in the USA matching GAP was 0.65 mg/kg.

Total residues in spinach for the foliar application were (n = 7): 0.63, 0.68, 0.89, 1.2, 2.3, 2.6 and 4.4 mg/kg. Total residue in spinach for the soil application was 0.73 mg/kg.

The GAP is the same for leaf lettuce, head lettuce and spinach. The median residue in leaf lettuce (0.305 mg/kg), head lettuce (0.16 mg/kg) and spinach (0.62 mg/kg) are similar. The Meeting agreed to propose a group maximum residue level for leafy vegetables except watercress.

The Meeting recognized that the residue populations from trials on leaf lettuce, head lettuce and spinach were significantly different according to statistical test. The therefore Meeting decided to use the crop with the highest residue, i.e., spinach, to estimate a maximum residue level for leafy vegetables.

Based on the trials in spinach from the USA, the Meeting estimated a maximum residue level, an STMR value and an HR value for dinotefuran in leafy vegetables of 6, 1.2 and 4.4 mg/kg, respectively.

Watercress

Data were available from supervised trials on watercress in the USA.

The GAP on watercress of the USA is a foliar application at a maximum rate of 0.20 kg ai/ha (at a maximum seasonal rate of 0.40 kg ai/ha for foliar application) with a PHI of 1 day.

Dinotefuran residues in watercress from trials in the USA matching GAP were (n = 3): 1.6, 2.1 and 3.4 mg/kg.

Total residues in watercress were (n = 3): 2.0, 2.9 and 3.8 mg/kg.

Based on the trials for watercress in the USA, the Meeting estimated a maximum residue level, an STMR value and an HR value for dinotefuran in watercress of 7, 2.9 and 3.8 mg/kg respectively.

Potato

Data were available from supervised trials on potatoes in the USA.

The GAP on tuberous and corm vegetables from the USA is as a pre-plant soil application at a maximum rate of 0.38 kg ai/ha (at a maximum seasonal rate of 0.38 kg ai/ha for soil application) or a foliar application at a maximum rate of 0.076 kg ai/ha (at a maximum seasonal rate of 0.23 kg ai/ha for foliar application) with a PHI of 7 day. Only one application method can be used.

The trials on potatoes in the USA did not match the GAP. Consequently, the Meeting could not estimate a maximum residue level for dinotefuran in potatoes.

Celery

Data were available from supervised trials on celery from the USA.

The GAP from the USA is for a soil application at a maximum rate of 0.37 kg ai/ha with a PHI of 21 days (at a maximum seasonal rate of 0.60 kg ai/ha for soil application) or a foliar application at a maximum rate of 0.15 kg ai/ha (at a maximum seasonal rate of 0.30 kg ai/ha for foliar application) with a PHI of 7 day. Only one application method can be used.

Dinotefuran residues in celery from the foliar application matching US GAP were (n = 6): 0.06, 0.10, 0.18, 0.22, 0.24 and 0.28 mg/kg.

Total residues in celery following the foliar application were (n = 6): 0.08, 0.20, 0.36, 0.51, 0.58 and 0.67 mg/kg.

Based on the trials for celery in the USA, the Meeting estimated a maximum residue level, an STMR value and an HR value for dinotefuran in celery of 0.6, 0.435 and 0.67 mg/kg respectively.

Rice

Data were available from supervised trials on rice in the USA.

Trials from the USA on rice were reported for the foliar application of a SG formulation (GAP: two foliar applications of a maximum rate of 0.15 kg ai/ha, PHI of 7 days).

Dinotefuran residues in rice grains from trials in the USA matching GAP were (n = 9): 1.4, 1.8, 1.9 (2), 2.4, 2.5, 2.9 (2) and 4.0 mg/kg.

Total residues in rice grains were (n = 9): 1.9, 2.4, 2.7, 2.9, 3.3, 3.8, 4.4, 4.6 and 8.1 mg/kg.

Based on the trials for rice in the USA, the Meeting estimated a maximum residue level, an STMR value for dinotefuran in rice of 8 and 3.3 mg/kg respectively.

Cotton seed

Data were available from supervised trials on cotton in the USA.

Trials from the USA on cotton were reported for a foliar application of a SG formulation (GAP: a foliar application of a maximum rate of 0.15 kg ai/ha with a seasonal maximum rate of 0.30 kg ai/ha, PHI of 14 days).

Dinotefuran residues in cotton seeds from trials in the USA matching GAP were (n = 12): < 0.05 (5), 0.05 (3), 0.07 (2), 0.10 and 0.16mg/kg.

Total residues in cotton seeds were (n = 12): < 0.15 (5), 0.15 (3), 0.17 (2), 0.20 and 0.33 mg/kg.

Based on the trials for cotton in the USA, the Meeting estimated a maximum residue level and an STMR value for dinotefuran in cotton seeds of 0.2 and 0.15 mg/kg respectively.

Animal feedstuffs

Rice straw

Data were available from supervised trials on rice in the USA.

Trials from the USA on rice were reported for the foliar application of a SG formulation (GAP: two foliar applications of a maximum rate of 0.15 kg ai/ha, PHI of 7 days).

Dinotefuran residues in rice straw from trials in the USA matching GAP were (n = 9): 0.61, 0.82, 0.87, 0.90, 1.2, 1.3, 1.4, 2.6 and 3.8 mg/kg.

Total residues in rice straw were (n = 9): 1.1 (2), 1.2, 1.3, 1.6, 1.7, 1.8, 3.7 and 4.3 mg/kg.

Based on the residues in rice straw from trials in the USA, the Meeting estimated a maximum residue level, a median residue value and a highest residue value for dinotefuran in rice straw and fodder, dry of 6, 1.6 and 4.3 mg/kg respectively.

Cotton gin trash

Data were available from supervised residue trials on cotton in the USA.

Trials from the USA on cotton were reported for the foliar application of a SG formulation (GAP: a foliar application of a maximum rate of 0.15 kg ai/ha at a seasonal maximum rate of 0.30 kg ai/ha, PHI of 14 days).

Total residues in cotton gin trash from trials in the USA matching GAP were (n = 7): 1.5, 3.3 (2), 3.8, 4.9, 5.6 and 7.1 mg/kg.

Based on the trials for cotton in the USA, the Meeting estimated a median residue value and a highest residue value for dinotefuran in cotton gin trash of 3.8 and 7.1 mg/kg respectively.

Rotational crops

The US GAP shows that for all crops other than berry and small fruit (subgroup small fruit vine climbing except fuzzy kiwifruit and low growing berry except strawberry), cotton, head and stem brassica, leafy brassica greens (including turnip greens), cucurbits, fruiting vegetables, leafy vegetables, bulb onion, green onion, peach and nectarine, tuberous and corm vegetables, and watercress, a 120-day plant-back interval must be observed. The Meeting noted that residues were not expected on rotational crops.

Fate of residues during processing

The fate of dinotefuran residues has been examined in grapes, tomatoes, potatoes, rice grains and cotton seeds processing studies. Based on the results of processing studies conducted in the USA,

processing factors were calculated for grapes, tomatoes, potatoes, rice grains and cotton seeds. Estimated processing factors and the derived STMR-Ps are summarized in the Table below.

Processing factors, STMR-P and HR-P for food and feed

Raw agricultural commodity (RAC)	Processed commodity	Calculated processing factors*	PF (Mean or best estimate)	RAC STMR (mg/kg)	STMR-P (mg/kg)	RAC HR (mg/kg)	HR-P (mg/kg)
Grape	Juice	0.95, 1.4	1.2	0.22	0.264	0.67	
	Raisin	3.1, 4.2	3.7		0.814		2.479
Tomato	Puree	1.1, 1.6, 2.1	1.6	0.10	0.16		
	Paste	3.3, 4.6, 5.2	4.6		0.46		
Potato	Granules	3.0, 2.3	2.7				
	Chips	2.1, 1.5	1.9				
Rice	Polished rice	0.02, 0.05	0.04	3.3	0.132		
	Bran	0.42, 0.85	0.64		2.112		
	Hulls	3.8, 5.4	4.6		15.18		
Cotton	Meal	0.27, 0.47	0.37	0.15	0.0555		
	Hulls	0.29, 0.72	0.51		0.0765		
	Refined oil	< 0.05, < 0.09	< 0.07		0.0105		

***** Each value represents a separate study. The factor is the ratio of the residue in processed commodity divided by the residue in the RAC.

The Meeting estimated a maximum residue level of 3 mg/kg (0.9 × 3.7 = 3.33 mg/kg) for dried grape and 0.3 mg/kg (8 × 0.04 = 0.32 mg/kg) for polished rice.

On the basis of the STMR and HR for sweet peppers and default dehydration factor of 10, the Meeting estimated at an STMR value and an HR value for dried chili peppers of 1.75 and 5.0 mg/kg respectively. Based on the maximum residue level of fruiting vegetables, other than cucurbits, the Meeting recommended a maximum residue level of 5 mg/kg for chili peppers (dry).

Residue in animal commodities

Farm animal dietary burden

The Meeting estimated the dietary burden of dinotefuran in farm animals on the basis of the diets listed in Appendix IX of the FAO Manual 2009. Calculation from highest residue, STMR (some bulk commodities) and STMR-P values provides levels in feed suitable for estimating MRLs, while calculation from STMR and STMR-P values for feed is suitable for estimating STMR values for animal commodities. The percentage dry matter is taken as 100% when the highest residue levels and STMRs are already expressed in a dry weight basis.

Estimated maximum and mean dietary burdens of farm animals

Dietary burden calculations for beef cattle, dairy cattle, broilers and laying poultry are provided in Appendix IX of the FAO manual. The calculations were made according to the animal diets from US-Canada, EU, Australia and Japan in the Table (Appendix IX of the FAO manual).

	Livestock dietary burden, dinotefuran, ppm of dry matter diet							
	US-Canada		EU		Australia		Japan	
	Max	Mean	Max	Mean	Max	Mean	Max	Mean
Beef cattle	1.5	1.3	9.3	2.6	13	4.6	3.1	1.4
Dairy cattle	5.5	2.3	6.6	2.2	**15a**	**6.3bc**	1.4	0.68
Poultry–broiler	1.0	1.0	0.24	0.24	2.4	2.4	0.12	0.12
Poultry–layer	1.0	1.0	1.6	0.52	**2.4d**	**2.4e**	0.47	0.47

[a] - Highest maximum beef or dairy cattle dietary burden suitable for MRL estimates for mammalian meat and milk

[b] - Highest mean beef or dairy cattle dietary burden suitable for STMR estimates for mammalian meat

[c] - Highest mean dairy cattle dietary burden suitable for STMR estimates for milk

[d] – Highest maximum poultry dietary burden suitable for MRL estimates for poultry meat and eggs

[e] – Highest mean poultry dietary burden suitable for STMR estimates for poultry meat and eggs

Farm animal feeding studies

The Meeting received a lactating dairy cow feeding studies, which provided information on likely residues resulting in animal commodities and milk from dinotefuran residues in the animal diet.

A poultry feeding study was not submitted as the expected residues of dinotefuran in poultry feed were very low. A poultry metabolism study at a dose rate of 10 ppm dinotefuran in feed demonstrated that there was very low transfer to eggs and tissues with all residues of dinotefuran and metabolites less than 0.01 mg/kg.

Lactating dairy cows

Lactating dairy cows were dosed with the mixture of dinotefuran, UF and DN (3:1:1) for 29–30 days at the equivalent of 5, 15 and 50 ppm in the diet. Residues of dinotefuran were below the LOQ (0.01 mg/kg) in whole milk with some exceptions at all feeding level. UF was the predominant residue found in milk from all treated animals. No detectable residues of dinotefuran occurred in any tissue samples. UF was again the predominant residue in all tissues.

Animal commodities maximum residue levels

For MRL estimation, the residue in the animal commodities is dinotefuran and UF.

Residues in tissues and milk at the expected dietary burden for dairy cattle are shown in the Table below. The total residue of dinotefuran and UF in milk reached a plateau at Day 4. The mean estimated residue in milk was calculated using the residue values of Day 4 to the final day.

	Feed level (ppm) for milk residues	Residues (mg/kg) in milk [a]	Feed level (ppm) for tissue residues	Residues (mg/kg) in [a]			
				Muscle	Liver	Kidney	Fat
MRL beef or dairy cattle							
Feeding study	15	0.082	15	0.062	0.066	0.076	0.065
Dietary burden and residue estimate	15	0.082	15	0.062	0.066	0.076	0.065
STMR beef or dairy cattle							
Feeding study	5 15	0.033 0.082	5 15	0.026 0.058	0.024 0.061	0.023 0.072	< 0.02 0.044
Dietary burden and residue estimate	6.3	0.039	6.3	0.030	0.030	0.030	0.025

[a] Sum of dinotefuran and UF expressed as dinotefuran (using a molecular weight conversion factor of 1.3 for UF)

Based on the highest estimated residue in milk (0.082 mg/kg), the Meeting estimated a maximum residue level of 0.1 mg/kg in milk.

Based on the highest estimated residue in muscle (0.062 mg/kg), the Meeting estimated a maximum residue level of 0.1 mg/kg, an HR value of 0.062 mg/kg in mammalian meat.

Based on the highest estimated residue in kidney (0.076 mg/kg), the Meeting estimated a maximum residue level of 0.1 mg/kg, an HR value of 0.076 mg/kg in mammalian edible offal.

Based on the mean estimated residues in tissues and milk, the Meeting estimated STMR values of 0.039 mg/kg in milk, 0.030 mg/kg in meat and 0.030 mg/kg in edible offal.

The maximum dietary burden for broiler and layer poultry is 2.4 and is lower than the dose level in the laying hen metabolism study of 10 ppm. In the metabolism study, in which dinotefuran equivalent to 10 ppm in the diet was dosed to laying hens for 5 consecutive days, no residues of dinotefuran, UF and DN exceed 0.01 mg/kg were detected in tissues and egg yolk. Dinotefuran was only detected at 0.013 mg/kg in egg white.

The Meeting estimated a maximum residue level of 0.02 (*) mg/kg, an STMR value of 0 mg/kg and an HR value of 0 mg/kg in poultry meat, poultry edible offal and eggs.

RECOMMENDATIONS

On the basis of the data from supervised trials, the Meeting concluded that the residue levels listed below are suitable for estimating maximum residue limits and for IEDI and IESTI assessment.

Plant commodities:

Definition of the residue for plant commodities (for compliance with the MRL):

Dinotefuran

Definition of the residue for plant (for estimation of dietary intake): *Sum of Dinotefuran, 1-methyl-3-(tetrahydro-3-furylmethyl) urea (UF) and 1-methyl-3-(tetrahydro-3-furylmethyl) guanidium dihydrogen (DN) expressed as dinotefuran*

Animal commodities:

Definition of the residue for animal commodities (for compliance with the MRL and for estimation of dietary intake): *Sum of Dinotefuran and 1-methyl-3-(tetrahydro-3-furylmethyl) urea (UF) expressed as dinotefuran*

The residue is not fat soluble

Commodity		Recommended MRL, mg/kg	STMR or STMR-P, mg/kg	HR or HR-P, mg/kg
CCN	Name	New		
VB 0040	Brassica (cole or cabbage) vegetables, Head cabbages, Flowerhead cabbages	2	0.40	1.1
VS 0624	Celery	0.6	0.435	0.67
SO 0691	Cotton seed	0.2	0.15	
FB 0265	Cranberry	0.15	0.065	0.10
DF 0269	Dried grapes (= currants, Raisins and Sultanas)	3	0.81	2.479
MO 0105	Edible offal (Mammalian)	0.1	0.030	0.076
PE 0112	Eggs	0.02*	0	0
VC 0045	Fruiting vegetables, Cucurbits	0.5	0.25	0.33
VO 0050	Fruiting vegetables, other than Cucurbits except sweet corn and mushrooms	0.5	0.15	0.55
FB 0269	Grapes	0.9	0.22	0.67
VL 0053	Leafy vegetables except watercress	6	1.2	4.4
MM 0095	Meat (from mammals other than marine mammals)	0.1	0.030	0.062
ML 0106	Milks	0.1	0.039	
FS 0245	Nectarine	0.8	0.28	0.57
VA 0385	Onion, Bulb	0.1	0.04	0.09
FS 0247	Peach	0.8	0.28	0.57
HS 0444	Peppers Chili, dried	5	1.75	5.0
PO 0111	Poultry, Edible offal of	0.02*	0	0
PM 0110	Poultry meat	0.02*	0	0
GC 0649	Rice	8	3.3	
CM 1205	Rice, polished	0.3	0.132	
AS 0649	Rice straw and fodder, dry	6	1.6	4.3
VA 0389	Spring onion	4	0.91	2.3
VL 0473	Watercress	7	2.9	3.8

* at or about the LOQ.

Commodity	STMR or STMR-P, mg/kg	HR or HR-P, mg/kg
Name	New	
Cotton gin trash	3.8	7.1
Cotton hulls	0.0765	
Cotton meal	0.0555	
Cotton seed oil, edible	0.0105	
Grape juice	0.264	
Rice bran, unprocessed	2.112	
Rice hulls	15.18	
Tomato paste	0.46	
Tomato puree	0.16	

DIETARY RISK ASSESSMENT

Long-term intake

The International Estimated Daily Intakes (IEDIs) of dinotefuran were calculated for the 13 GEMS/Food cluster diets using STMRs/STMR-Ps estimated by the current Meeting (se Annex 3 of the 2012 JMPR Report). The ADI is 0–0.2 mg/kg bw and the calculated IEDIs were 0–3% of the maximum ADI (0.2 mg/kg bw). The Meeting concluded that the long-term intakes of residues of dinotefuran, resulting from the uses considered by current JMPR, are unlikely to present a public health concern.

Short-term intake

The International Estimated Short-Term Intakes (IESTI) of dinotefuran were calculated for food commodities and their processed commodities using HRs/HR-Ps or STMRs/STMR-Ps estimated by the current Meeting (see Annex 4 of the 2012 JMPR Report). The ARfD is 1 mg/kg bw and the calculated IESTIs were a maximum of 30% of the ARfD. The Meeting concluded that the short-term intake of residues of dinotefuran, when used in ways that have been considered by the JMPR, is unlikely to present a public health concern.

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