

ZOXAMIDE (227)

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EXPLANATION

Zoxamide, a benzamide fungicide, was identified as a priority new compound at the 38th Session of the CCPR (ALINORM 06/29/24) for evaluation by the 2007 JMPR. The Meeting received information on physical and chemical properties, animal and plant metabolism, environmental fate, analytical methods, storage stability, use patterns, supervised trials and processing.

IDENTITY

ISO common name: Zoxamide

Chemical name

IUPAC: (RS)-3,5-dichloro-N-(3-chloro-1-ethyl-1-methyl-2-oxopropyl)-p-toluamide

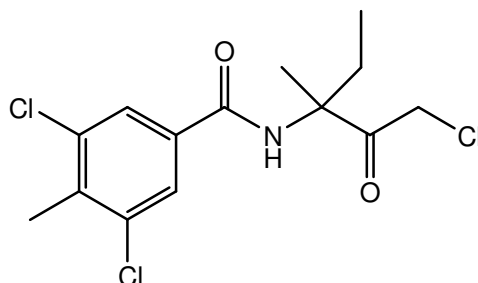
CAS: 3,5-dichloro-N-(3-chloro-1-ethyl-1-methyl-2-oxopropyl)-4-methylbenzamide

CAS Registry No.: 156052-68-5

CIPAC No.: 640

Synonyms: RH-117,281 and RH-7281

Structural formula:



Molecular formula: C₁₄H₁₆Cl₃NO₂

Molecular weight: 336.65

PHYSICAL AND CHEMICAL PROPERTIES**Pure active ingredient**

Property	Description or results	Reference
Appearance:	Lumpy white powder	(Ardern, 1998, DERBI 91601)
Odour:	Flour like odour	(Betterley, 1998, DERBI 91603)
Colour:	Munsell neutral scale of N9.5%R	
Vapor pressure:	<1.3 x 10 ⁻⁵ Pa at 25, 35 and 45 °C	(Kogovsek, 1996, DERBI 91517)
Melting point:	159.5 – 160.5 °C (98.8% purity)	(Ardern, 1998, DERBI 91601) (Betteley, 1998, DERBI 91602)
Relative density:	1.38 at 20 °C	

Property	Description or results	Reference
Octanol-water partition coefficient:	Log Kow 3.76 ± 0.04 at ambient temp.	(Reynolds, 1996, DERBI 91609)
Solubility at 20 °C:	Water: 0.681 ± 0.017 mg/L Ethyl acetate: 20.0 g/L Acetone: 55.7 g/L Xylene: 1.56 g/L n-Octanol: 6.49 g/L n-Heptane: 0.038 g/L 1,2-Dichloroethane: 12.5 g/L	(Reynolds, 1996, DERBI 91610) (Betteley, 1998, DERBI 91606)
Hydrolysis at 25 °C:	pH DT ₅₀ (days) 4 16 7 16 9 8	(Reynolds, 1998, DERBI 92157)
Photolysis:	In sterile buffer of pH 4 at 25 °C DT ₅₀ = 8 days	(Smalley, 1998, DERBI 92126)
Dissociation constant:	Not measurable by spectrophotometry, titration or conductivity Absorbance did not change in acidic, neutral, or basic conditions; Solubility was too low for titration; With decrease in concentration, no increase in reading was observed.	(Betteley, 1998, DERBI 91605)

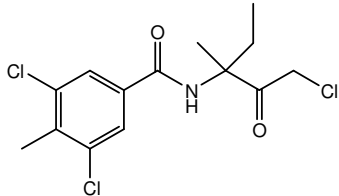
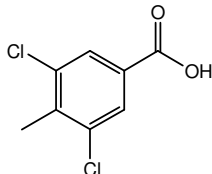
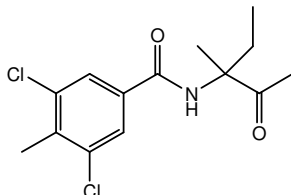
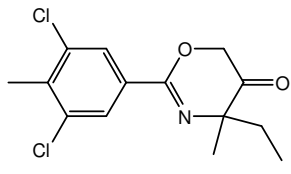
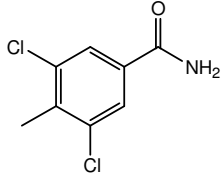
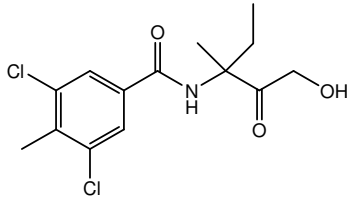
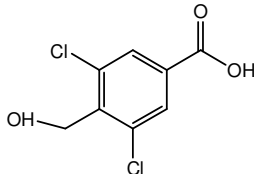
Technical material

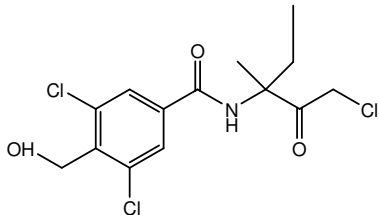
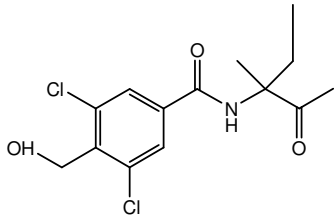
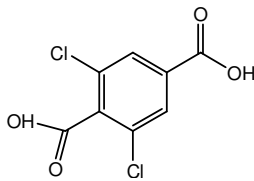
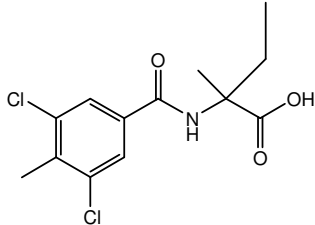
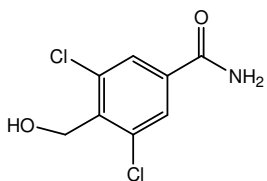
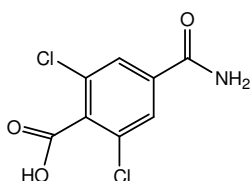
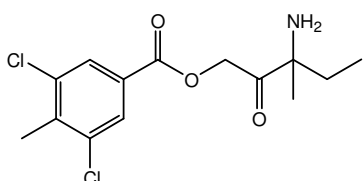
The technical material consists of a racemic compound containing one chiral centre. Both enantiomers are present in equal quantities.

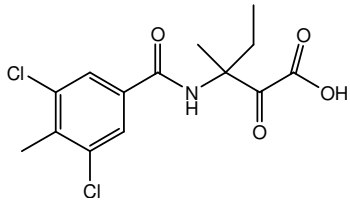
Property	Results	Reference
Purity:	Minimum 95%	(Roemmele, 2001, DERBI 104580)
Appearance:	Fine powder	(Roemmele, 1998, DERBI 92167)
Odour:	Liquorice-like	
Colour:	White Muncell neutral scale, N9.6 90%R	
Melting point:	159.5 – 161.0 °C	(Roemmele, 1998, DERBI 92167)
Stability:	Stable during storage in the following conditions: Elevated heat and pressure; and Elevated heat and pressure plus: 316L stainless steel (100 mesh size); carbon steel; Iron II; and Iron III.	(Roemmele, 1998, DERBI 92167)
Formulations:	Wettable powder (WP) and water dispersible granules (WG) in various concentrations alone or in combination with mancozeb or cymoxanil at various ratios. Labels from one country indicate combinations with iprovalicarb and famoxadone are registered.	

METABOLISM AND ENVIRONMENTAL FATE

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

Code number	Chemical name/description	Structure
Zoxamide RH-117281 RH-7281	3,5-dichloro- <i>N</i> -(3-chloro-1-ethyl-1-methyl-2-oxopropyl)-4-methylbenzamide	
RH-24549 RH-4549	3,5-dichloro-4-methylbenzoic acid	
RH-127450 RH-7450	3,5-dichloro- <i>N</i> -(1-ethyl-1-methyl-2-oxopropyl)-4-methylbenzamide	
RH-129151 RH-9151	2-(3,5-dichloro-4-methylphenyl)-4-ethyl-4-methyl-4H-1,3-oxazin-5(6H)-one	
RH-139432 RH-9432	3,5-dichloro-4-methylbenzamide	
RH-141288 RH-1288	3,5-dichloro- <i>N</i> -(3-hydroxy-1-ethyl-1-methyl-2-oxopropyl)-4-methylbenzamide	
RH-141452 RH-1452	3,5-dichloro-4-hydroxymethylbenzoic acid	

Code number	Chemical name/description	Structure
RH-141453 RH-1453 (present at abt 1% in the test material used; not a metabolite)	3,5-dichloro- <i>N</i> -(3-chloro-1-ethyl-1-methyl-2-oxopropyl)-4-hydroxymethylbenzamide	
RH-141454	3,5-dichloro- <i>N</i> -(1-ethyl-1-methyl-2-oxopropyl)-4-hydroxymethylbenzamide	
RH-141455 RH-1455	3,5-dichloro-1,4-benzenedicarboxylic acid 3,5-dichloro-terephthalic acid	
RH-141643	3,5-dichloro- <i>N</i> -(2-carboxy-1-ethyl-1-methyl)-4-methylbenzamide	
RH-149736	3,5-dichloro-4-hydroxymethylbenzamide	
RH-149737	4-carboxy-3,5-dichlorobenzamide	
RH-150721 RH-0721	(3-amino-3-methyl-2-oxo)pentyl-(3,5-dichloro-4-methyl)benzoate	

Code number	Chemical name/description	Structure
RH-163353	3,5-dichloro- <i>N</i> -(2-carboxy-1-ethyl-1-methyl-2-oxoethyl)-4-methylbenzamide	
M-1	3,5-Dichloro- <i>N</i> -(1-ethyl-1-methyl-2-oxopropyl)-benzamide-4-carboxylic acid	
M-2	Structurally isomeric glucuronic acid conjugate of 3,5-dichloro- <i>N</i> -(3-hydroxy-1-ethyl-1-methyl-2-oxopropyl)-4-hydroxymethylbenzamide	
M-3	Glucuronic acid conjugate of 3,5-dichloro- <i>N</i> -(2,3-dihydroxy-1-ethyl-1-methylpropyl)-4-hydroxymethylbenzamide	
M-4	Structurally isomeric glucuronic acid conjugate of 3,5-dichloro- <i>N</i> -(3-hydroxy-1-ethyl-1-methyl-2-oxopropyl)-4-hydroxymethylbenzamide	
M-5	Glucuronic acid conjugate of 4-hydroxymethyl-RH-141643	
M-6	Glucuronic acid conjugate of 4-hydroxymethyl-RH-141454	
M-7	Glucuronic acid conjugate of 4-hydroxymethyl-RH-141288	
M-8	Methyl sulfone metabolite related to RH-141453	
M-9	Methyl sulfoxide derivative of dechlorinated RH-117281	
M-10	Methyl sulfone metabolite related to RH-117281	
M-11	Unidentified compound	
M-12a, M-12b	Positional isomers of a dihydroxylated analogues of RH-127450	

Animal metabolism

The Meeting received information on the fate of orally-dosed zoxamide in a lactating goat. No information on metabolism of zoxamide in poultry was submitted.

Lactating goat

In a study by Robinson (1998; DERBI 92155), [U - ^{14}C -phenyl]zoxamide (isotopically diluted with [^{12}C] zoxamide and [^{13}C] zoxamide) was administered orally in gelatine capsules to a lactating goat once a day for 7 consecutive days. The test material was dosed at levels equivalent to a dietary concentration of 60.7 ppm. As a control, a second goat received placebo capsules administered orally.

Urine, faeces and milk samples were collected twice a day in the morning and afternoon. The urine and faeces samples were each pooled after sample collection. A cage rinse was collected at the end of each 24 h sampling period. Blood samples were taken from both animals on days 0 (control), 1, 3 and 7. Both goats were sacrificed approximately 23 h after the final dose. Omental fat, liver, kidney and muscle samples were removed at necropsy for further analysis.

Radioactivity of daily urine, daily cage rinse and final bile samples were analysed by direct liquid scintillation counting (LSC). Total radioactive residues (TRR) in milk samples, expressed as mg/kg equivalents of zoxamide, were determined by direct LSC. TRR in faeces, muscle (combination of leg and loin), liver, kidney and blood samples were determined by combusting samples to ^{14}C -carbon dioxide and counting by LSC. TRR in omental fat were determined by tissue solubilization.

The total dose recovered was 77.5%. Radioactivity analyses of urine and faeces samples from the treated goat showed values accounting for 37.1% and 36.1%, respectively, of the total administered dose. About 95% of the recovered radioactivity was found in urine and faeces. Individual tissues and cumulative milk samples on day 7 each amounted to <0.3% of the administered dose. A summary of the distribution of radioactivity and total terminal residues is presented in Table 1.

Table 1. Summary of the distribution of radioactivity in goat tissues and milk

Matrix	% administered dose	TRR (mg/kg)
Urine ^a	37.1%	-
Faeces ^a	36.1 %	-
Bile	0.10%	-
Milk ^a	0.27%	0.236 (day 4, pm) ^b
Liver	0.05%	0.450
Kidney	0.01 %	0.365
Leg Muscle	0.01 %	0.046
Loin Muscle	< 0.01 %	0.044
Omental Fat	0.02%	0.197
Blood	< 0.01 %	0.101 (at sacrifice)
Cage Rinse ^a	3.77%	-
Total	~ 77.5%	-

a - Values expressed as a cumulative percent of the total dose administered for 7 days

b - Highest value among samples taken during the 7 day period.

Milk samples taken on day 3 in the morning and day 4 in the afternoon were extracted with acetone and the extract partitioned between acetonitrile and hexane. Greater than 80% of the TRR went into the acetonitrile phase with the remaining residues distributed between the hexane fraction and unextractable residue fraction.

Muscle, liver and kidney samples were extracted with methanol/water and chloroform. Methanol extracted the majority of the radioactivity from liver and kidney, whereas most of the radioactive residues in muscle, which were organosoluble, resided in the chloroform extract. The chloroform extracts were concentrated and subjected to acetonitrile/hexane solvent partition, which resulted in a majority of radioactive residues present in the acetonitrile fraction. The majority of radioactivity in omental fat was extractable with hexane. The hexane extract was subjected to solvent partitioning with acetonitrile, resulting in removal of a majority of the radioactivity.

With the exception of liver, radioactivity remaining as unextractable residues was low for all samples, accounting for less than 10% of TRR (< 0.05 mg/kg). In liver samples, however, 11.8% of TRR (0.053 mg/kg) remained unextracted. Treatment of the liver unextractable fraction with protease enzyme released 5.1% of TRR (0.023 mg/kg).

Fractions containing significant TRR levels (> 10% of TRR, > 0.01 mg/kg) were analyzed for their metabolite profiles by reversed-phase HPLC (ODS 20 column with UV and/or radioactivity detection) and/or normal-phase TLC. Metabolites contributing a significant portion of the TRR in milk and tissues were identified by comparative chromatography and co-chromatography with the unlabeled reference standards using reversed-phase HPLC and normal-phase TLC and/or by liquid chromatography/ electrospray ionization mass spectrometry (LC/ESI-MS).

The total contribution of major metabolites in extractable fractions from milk and tissues is summarized in the tables below.

Table 2. Major metabolites in extractable fractions from milk and tissues

Metabolite	Milk – day 4		Fat		Liver		Kidney		Muscle	
	%TRR ^a	mg/kg ^b	%TRR ^a	mg/kg ^b	%TRR ^a	mg/kg ^b	%TRR ^a	mg/kg ^b	%TRR ^a	mg/kg ^b
RH-127450	20.24	0.048	65.18	0.129	2.01	0.009	0.85	0.003	15.13	0.007
RH-141288	11.88	0.028	15.75	0.031	1.72	0.008	3.95	0.014	12.64	0.006
RH-141454	18.00	0.043								
M-1, M-2, M-3, M-4					17.27 ^c	0.078	17.82 ³	0.065	2.96	0.001
M-5					14.53	0.065	13.61	0.050	5.03	0.002
M-5, M-6					16.72	0.075	20.11	0.074	8.34	0.004
M-7					23.17	0.104	10.54	0.039	5.20	0.002
M-8					1.55	0.007	4.99	0.018	12.26	0.006
M-9					1.54	0.007				
M-10					0.87	0.004			1.16	0.001

Metabolite	Milk – day 4		Fat		Liver		Kidney		Muscle	
	%TRR ^a	mg/kg ^b	%TRR ^a	mg/kg ^b	%TRR ^a	mg/kg ^b	%TRR ^a	mg/kg ^b	%TRR ^a	mg/kg ^b
M-11					1.97	0.009	0.64	0.002		
M-12a, M-12b	37.87	0.090					11.72	0.043	25.82	0.012
Others	2.54	0.006	4.07	0.008	4.36	0.020	4.20	0.015	3.49	0.002
Total	90.5	0.215	85.0	0.168	85.7	0.386	88.4	0.323	92.03	0.043

a - Percentage of total radioactive residues in milk or each tissue (see Table 2)

b - Parent compound equivalents

c - M-2 and M-4 are major components.

Zoxamide was extensively metabolized and readily eliminated following oral administration to a lactating goat. The efficient elimination processes resulted in negligible to modest retention of radioactive residues in milk and tissues with levels in milk up to 0.236 mg/kg (parent equivalents) on day 4. As the dose administered in the study, which is 60.7 ppm in the diet, was about 14 times the highest concentration found in any commodity after treatment of the respective crop in accordance with existing GAP, significant residue concentrations are unlikely to be detected in milk in practice. No parent was found in any tissue or milk.

The metabolism of zoxamide in the lactating goat was qualitatively similar to that of laboratory animals described in the toxicology section of the 2007 Report of the JMPR (see page 280).

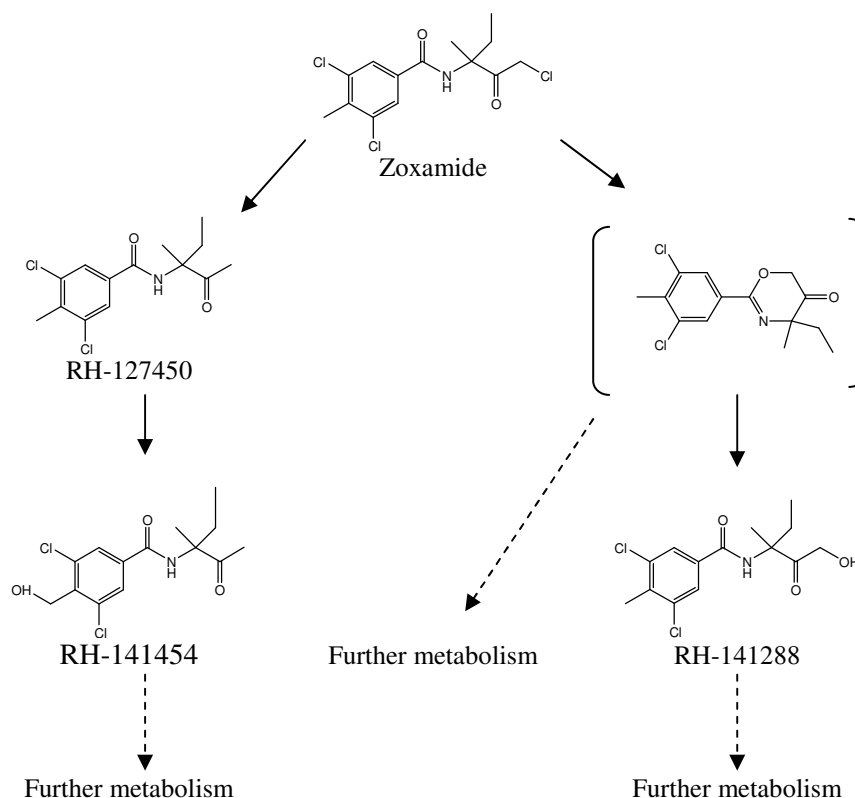


Figure 1. Proposed metabolic pathway of zoxamide in goat

Plant Metabolism

The Meeting received plant metabolism studies on grapes, cucumbers, tomato and potato.

Grapes

Reibach (1998;DERBI 91481) treated Concord grapes grown in Pennsylvania USA with [U-¹⁴C-phenyl]zoxamide (radiochemical purity 97.6%)(isotopically diluted with [¹²C] zoxamide and [¹³C] zoxamide) formulated as a 5% w/w active ingredient emulsifiable concentrate (EC). One grape vine was treated with three foliar applications, each at a rate of 1.87 kg ai/ha. Applications were made at about 30 day (\pm 4 day) intervals. A slight drizzle about two hours following the first application occurred but did not wash off the test material. Although the formulation caused transient and insignificant grape leaf injury, the yield of the grapes was not affected.

Aliquots of spray solutions collected at the time of each application were analysed by LSC for total radioactivity and by TLC for radiopurity. The results confirmed that the intended amount of test material was applied, and that the material was stable in spray solution.

The harvest of mature grapes occurred 1 day after the last spray. The samples were taken to the laboratory for analysis where they were frozen until sample preparation for analysis. Sample analyses were conducted 5 to 6 months after harvest. Grape samples were ground using a Waring food processor with dry ice. All grape samples from the control plot were processed before samples from the treated plot were processed. The samples were stored frozen until required for analysis

The total ¹⁴C activity was determined by combustion radio-assay. Based on the specific activity of the zoxamide used for treatment, the TRR for grapes at harvest was determined to be 0.74 mg/kg zoxamide equivalents.

Extraction of samples with methanol released 94.0 % of the TRR. The remaining solids were dried at room temperature and the remaining radioactivity was determined by combustion analysis resulting in 5.1 % of the TRR.

The methanol extract was concentrated to remove methanol and the concentrate was partitioned between water and ethyl acetate. The majority (72.1%) of the TRR was found in the ethyl acetate fraction (EA1). Following acidification, the aqueous fraction was again partitioned with ethyl acetate, this time removing only 2.2% of the TRR (EA2). The resulting aqueous fraction (AQ1) contained 16.2% of the TRR. The aqueous fraction was further fractionated by means of a C-18 solid phase extraction cartridge. This procedure resulted in an organic fraction which was eluted with methanol, containing 15.4% of the TRR, and a second aqueous fraction containing 0.4% of the TRR. The total recovery of each analytical procedure and the distribution of TRR in each fraction were determined via liquid scintillation counting or combustion.

The ethyl acetate fractions (EA1 and EA2), and the C-18 methanol fraction were compared to authentic standards of several potential metabolites using normal phase TLC by streaking or spotting the samples and standards about 1.5 cm from the bottom of the plate. Non radiolabelled standards were viewed under ultraviolet light. Radioactivity on the TLC plates was imaged using an SI optical imager. Radioactivity from the plates was quantified based on the % radiolabel in the peak of interest following background subtraction.

TLC in a non-polar solvent system consisting of hexane/ethyl acetate/acetic acid demonstrated that the EA1 fraction was composed primarily of parent zoxamide. There was significant activity remaining at the origin, demonstrating the formation of some polar metabolites.

The EA2 and methanol fractions showed little or no parent compound. In these two fractions the majority of the radiolabel remained at the origin (78.4 and 94.7% respectively). Due to the more polar nature of the origin material(s), chloroform/methanol/acetic acid or chloroform/methanol/methanoic acid were used as solvent systems to resolve these metabolites.

The ethyl acetate fraction EA1 was further analysed by two dimensional TLC. An aliquot of EA1 was mixed with standards of RH-129151, zoxamide, RH-127450, RH-139432, RH-24549, and RH-141288. The mixture spotted in the lower corner of a silica plate. The plate was developed in the

first dimension with hexane/ ethyl acetate / formic acid, and in the second dimension with chloroform / methanol / ammonium hydroxide. After drying, comparison of the UV visible spots from the standards with the radioactive image, confirmed that parent zoxamide was the major component and that RH-141288, RH-139432 and RH-129151 were also present together with several other metabolites at very low concentrations.

The EA1 fraction was found to contain parent compound as the only significant component (greater than 10%). Zoxamide accounted for 58.3% of the TRR. Further confirmation of parent compound was obtained by reversed phase HPLC analysis of the ethyl acetate fraction EA1 using a C-18 column, UV detection and an acidic mobile phase composed of acetonitrile/H₂O /phosphoric acid.

The fraction with the highest percentage of unidentified components was the MeOH fraction where 4.2% of the TRR remained at the origin. All other unknown degradation products were present at less than 5% of the TRR. The total accountability for the study is summarised in Table 3.

Table 3. Metabolites of zoxamide in grapes

Metabolite	% TRR	mg/kg ^a
Zoxamide	58.3	0.429
RH-129151	3.0	0.022
RH-139432	1.9	0.014
RH-141288	1.7	0.013
RH-149736	1.1	0.008
RH-149737	1.6	0.012
RH-150721	2.8	0.021
Unextracted radioactivity in remaining solids	5.1	0.036
Total unknown products in methanol fraction	15.3 ^b	0.113
Total unknown products in EA1 fraction	2.8	0.021
Total unknown products in EA2 fraction	1.1	0.008
C-18 aqueous fraction	0.4	0.003
Total accounted for	95.1	0.699

a - Expressed as parent equivalents

b - In the methanol fraction 4.2% of TRR remained at the origin. Three other bands were observed but not characterised further.

Cucumber

Cucumbers (variety Bush champion) grown in a sandy loam soil in North Carolina USA (Sharma, 1999; DERBI 91483) were treated with [U-¹⁴C-phenyl]zoxamide (radiochemical purity 94.8%). Three foliar broadcast applications were made at 7-day intervals at the rate of 1.3 kg ai/ha per application, to give a total seasonal use rate of 4.0 kg ai/ha. The crop samples were collected 1 day after the last application. Both cucumber fruit and foliage were collected at this sampling interval. Radioactive counting of the spray solutions indicated that 106% of the nominal dose was applied.

A combustion analysis of the crop samples collected showed that cucumber foliage contained an average residue of 108 mg/kg, while the average residue in the fruits was 1.53 mg/kg, greatly lower than that in foliage. The relative standard deviation of the residues in both fruit and foliage were 46% (n=8) and 129% (n=6), respectively. The residue from all crop samples was extracted with acetonitrile-water, which solubilised 100% of the total radioactivity. There were no volatile or unextractable residues in the cucumber fruit or foliage. In each case, the extracted residue was partitioned into ethyl acetate/water before analysis.

The residue in cucumbers was quantified by HPLC showing the parent compound to be the major component of the residue. Zoxamide accounted for 89% and 87% of the TRR in foliage and fruit, respectively. Much smaller amounts of metabolites were detected and none of these metabolites exceeded 5% of the TRR. Among them, RH-9151 and RH-7450 were found in the foliage and RH-0721 and RH-7450 were found in the fruit. In addition, trace quantities of RH-1288, RH-4549, and RH-1452 were also detected. Identification of zoxamide, RH-9151, RH-7450, RH-0721 and RH-4549 were obtained by LC/MS while the others were identified by HPLC and TLC.

The nature of the minor degradation products suggests that they are products of hydrolysis and photolysis for the most part, rather than true plant metabolites.

Table 4. Metabolites of zoxamide in cucumber fruit and foliage.

	Fruit %TRR	mg/kg	Foliage %TRR	mg/kg
TRR	100	1.53	100	108
%Extractable	100	1.53	100	108
<u>Metabolite</u>	%TRR	mg/kg	%TRR	mg/kg
Zoxamide ^a	86.7	0.327	92.2	99.6
RH-0721 ^a	4.80	0.073	0.19	0.209
RH-7450 ^a	1.96	0.030	1.51	1.627
RH-9432	1.16	0.018	0.93	1.007
RH-1288	0.41	0.006	0.43	0.467
RH-4549 ^a	0	0	0.28	0.301
RH-9151 ^a	0	0	1.64	1.77
Total	95.1	1.45	98.9	107

a - Identified by LC/MS and HPLC. All others were identified by HPLC and TLC only.

Tomato

Tomatoes (variety Celebrity) grown in a loamy sand soil in a greenhouse in North Carolina USA (Sharma, 1999; DERBI 91486) were treated with [U-¹⁴C-phenyl]zoxamide (radiochemical purity 99%). Three foliar broadcast applications of an EC formulation were made at 18 day intervals at the rate of 0.86 kg ai/ha per application, to give a total seasonal use rate of 2.6 kg ai/ha. The crop samples were collected 1 day after the last application. Both tomato fruit (green and red) and foliage were collected at this sampling interval.

Based on the combustion radioassay of homogenized tomato samples, the average total radioactive residue (TRR) was 0.29 mg/kg in the green tomato (n=15) and 0.497 mg/kg in the red tomato (n=19).

Samples of green and red tomato were extracted using acetonitrile and water to determine the nature of the residues present in these samples. Homogenized tomato samples were extracted once with 100% acetonitrile and twice more with acetonitrile/water (8:2). This procedure recovered 92 – 94% of the TRR with 6 – 8% remaining unextracted. The extracted residue was partitioned into ethyl acetate, butanol and water. The components soluble in ethyl acetate and butanol were analysed by HPLC and TLC which indicated the presence of parent compound as the largest component of the residue, and small amounts of a large number of highly degraded and/or polar metabolites. Only the parent compound exceeded 10% of the TRR in all samples. The aqueous fraction contained less than 4% of the TRR. The residues in both ethyl acetate and butanol were separated by HPLC and quantified by LSC in collected eluate fractions. All radioactivities injected to HPLC were recovered in the eluates.

Table 5. Metabolites of zoxamide in tomatoes

	Green tomato		Red tomato	
	dpm/g	mg/kg	dpm/g	mg/kg
TRR	13000±9300	0.290	22300±2820	0.497
Extraction	% TRR	mg/kg	% TRR	mg/kg
Residue Extracted (ACN-H2O)	94.04	0.273	91.95	0.457
Unextracted Residue	5.96	0.017	8.05	0.040
Partitioning				
Residue Extracted (ACN-H2O)	94.04	0.273	91.95	0.457
Volatile residue	0.0	0.0	0.0	0.000
Residue in ethyl acetate	75.20	0.218	68.13	0.339
Residue in butanol	15.15	0.044	21.57	0.107
Residue in aqueous	3.70	0.011	2.25	0.011
Quantitation (ethyl acetate fraction)				
Zoxamide	48.03	0.139	43.95	0.219

	Green tomato		Red tomato	
	dpm/g	mg/kg	dpm/g	mg/kg
TRR	13000±9300	0.290	22300±2820	0.497
Extraction	% TRR	mg/kg	% TRR	mg/kg
RH-1452 ^a	15.04	0.044	11.19	0.056
RH-1288 ^a	6.82	0.020	2.74	0.014
RH-4549	2.49	0.007	0.84	0.004
RH-7450	1.10	0.003	1.36	0.007
All other fractions	1.71	0.005	8.05	0.039
Quantitation (butanol fraction)				
Fr-3	1.15	0.003	5.61	0.028
Fr-4, [glucose conjugate-2 ^b]	3.63	0.011	10.01 ^d	0.050
Fr-5, [glucose conjugate-1 ^c]	2.86	0.008		
Fr-6, [AA-conjugate-3 ^e]	1.07	0.003	1.79	0.009
Fr-7	1.81	0.005	1.89	0.009
Fr-8	1.46	0.004	0.94	0.005
Fr-9	3.16	0.009	0.66	0.003

a - Material ID represents only a portion of the activity in these fractions.

b - glucose conjugate of RH-1452 accounted for a part of the residue in this fraction.

c - glucose conjugate-1 accounted for a part of the residue in this fraction.

d - for red tomato, fractions 4/5 are shown together as 10%TRR, and it contained both conjugates 1 and 2.

e - aspartic acid conjugate-3 accounted for a portion of this fraction.

Analysis of the collected eluates indicated that about 48% and 44% of the TRR in green and red tomato, respectively, was the parent compound. Much smaller amounts of metabolites were identified and none exceeded 3% of the TRR. RH-1452 and RH-1288 were identified in different fractions but their actual concentrations were not determined. Among the minor components, RH-4549, RH-1288 and RH-7450 were found in the ethyl acetate fractions of fruit. The minor metabolites were identified by HPLC and TLC. The major component, zoxamide, was identified by chromatographic and mass spectroscopic methods (GC/MS and LC/MS).

The results of the study show zoxamide to be the main residue of concern. The nature of the minor degradation products suggested that they were mainly hydrolysis and photolysis products for the most part. A small portion of zoxamide underwent photolytic dechlorination and then hydrolysis and/or oxidation in tomato. The carboxylic acids generated via extensive degradations (RH-4549 and 1452) generated polar conjugates with sugars or amino acids or both. Some highly degraded/oxidized small metabolites, none of which could be found in significant amounts, were also produced.

Potato

White potatoes (variety Kennebec) grown in North Carolina USA (Reibach, 1998; DERBI 91482) were treated with [U-¹⁴C-phenyl]zoxamide (radiochemical purity 94.8%). Three foliar broadcast applications were made at a rate of 0.9 kg ai/ha per application, to give a total seasonal use rate of 2.7 kg ai/ha. The first application was made at 39 days after planting. The second and the third applications were made at 21 and 17 day intervals respectively. ¹⁴C-zoxamide-treated plants were grown in elliptical galvanised steel tanks containing loamy sand soil.

The test substance was formulated as a 5% w/w active ingredient emulsifiable concentrate (EC) and applied as an aqueous suspension. There was transient and insignificant potato leaf injury caused by the EC formulation used but this did not affect the yield of the potatoes.

The harvest of mature potato tubers was at 14 days after the last spray. Diseased, damaged or immature tubers were discarded. Freshly dug tubers were washed lightly to remove any soil and allowed to dry. The tubers were diced into cubes, weighed and stored frozen until required for analysis.

Potato samples were prepared for analysis by cryogenic milling. The total radioactivity was determined by LSC following combustion. Based on the specific activity of the radiolabelled

zoxamide used for treatment, the total radioactive residues (TRR) at harvest were determined to be 0.178 mg/kg for the homogenised potato tubers. The final harvest potato samples were analyzed to determine the nature of the residues present. The potato samples were extracted with methanol and then methanol was removed by rotary evaporation. The residue was dissolved in ethyl acetate and partitioned with water. The aqueous fraction was acidified with 6N hydrochloric acid and partitioned with ethyl acetate. Following acidification, the aqueous fraction was further fractionated by sequential elution from a C-18 solid phase extraction cartridge with methanol and water.

The total recovery of each analytical procedure and the distribution of TRR in each fraction were determined via liquid scintillation counting or combustion radioassay. The results of this analysis are presented in the table below.

Table 6. Distribution of radioactivity in potato treated with zoxamide

	%TRR	mg/kg
Extraction		
Total methanol extract	72.4	0.129
Unextracted residue	31.1	0.055
Total recovered	103.5	0.184
Fractionation of methanol extract		
Total extract	72.4	0.129
Ethyl acetate fraction	47.9	0.085
Water fraction	22.7	0.040
Fractionation of water fraction with C18 column		
Eluted by methanol	14.3	0.025
Eluted by water	7.6	0.013

The post-extraction solids (PES) resulting from the initial methanol extraction were hydrolysed using amyloglucosidase. The hydrolysis procedure released an additional 25.1% of the TRR, resulting in second PES that contained only 6% of the TRR. The second PES was not fractionated further. Following acidification, the hydrolysed fraction was further fractionated by means of a C-18 solid phase extraction cartridge. This procedure resulted in methanol eluate containing 3.7% of the TRR, and an aqueous fraction containing 15.9% of the TRR.

The ethyl acetate fraction, the C-18 methanol fraction and the aqueous fraction were concentrated by rotary evaporation and compared to reference standards using normal phase TLC. The ethyl acetate fraction was first analyzed by TLC using chloroform/methanol/acetic acid followed by development in a second system containing butanol/methanol/water/acetic acid. Standards of parent zoxamide, RH-1452, RH-141288, RH-24549, RH-129432 and RH-1455 were co-spotted with the sample. The ethyl acetate fraction was further analysed by two-dimensional TLC using chloroform/methanol/acetic acid followed by butanol/methanol/water/acetic acid as solvent systems. No parent compound was found. RH-1452 and RH-1455 were identified as the major components of the residue. Further confirmation was obtained using reverse phase HPLC using a C-18 column, UV detection and acetonitrile, water and phosphoric acid as solvent.

RH-1452 and RH-1455 were isolated from the ethyl acetate fraction using preparative TLC (butanol / methanol / water / acetic acid as solvent). The identity was confirmed by comparison of the isolated samples with non-radiolabelled standards using TLC. Structures proposed for the two major isolated metabolites were confirmed by mass spectrometry following reaction with diazomethane to convert any carboxyl groups to methyl esters.

The aqueous fractions contained a metabolite that may have been RH-1455 as judged by the similar R_f value, but due the polar nature of the fraction, the results were not conclusive. Since this fraction contained only 7.6% of the TRR (≈ 0.01 mg/kg), further analysis was not carried out.

The methanol and aqueous fractions obtained after hydrolysis of the initial PES with amyloglucosidase were also analyzed by TLC. The results of these analyses suggest that the material retained by the C18 cartridge was additional RH-1452 and RH-1455 solubilised by the enzyme

treatment. Analysis of the aqueous fraction suggested formation of radiolabelled sugars, most probably glucose released from the hydrolysis of starch.

The overall results of the fractionation of the methanol extraction of residues from potato are summarised in the table below.

Table 7. Metabolites of zoxamide in the methanol extract of potato

Identity of fraction	%TRR	mg/kg ^a
TRR	100	0.173
RH-1455	39.0	0.069
RH-1452	20.9	0.037
Glucose or other sugars	15.9	0.028
Enzyme hydrolysis unknowns	1.7	0.003
Aqueous unknowns	5.2	0.009
Organic unknowns	6.5	0.012
Unextractable residue ^b	6.0	0.011
Total accounted for	95.2	0.169

a - mg/kg calculated as parent equivalents

b - Calculated by subtraction

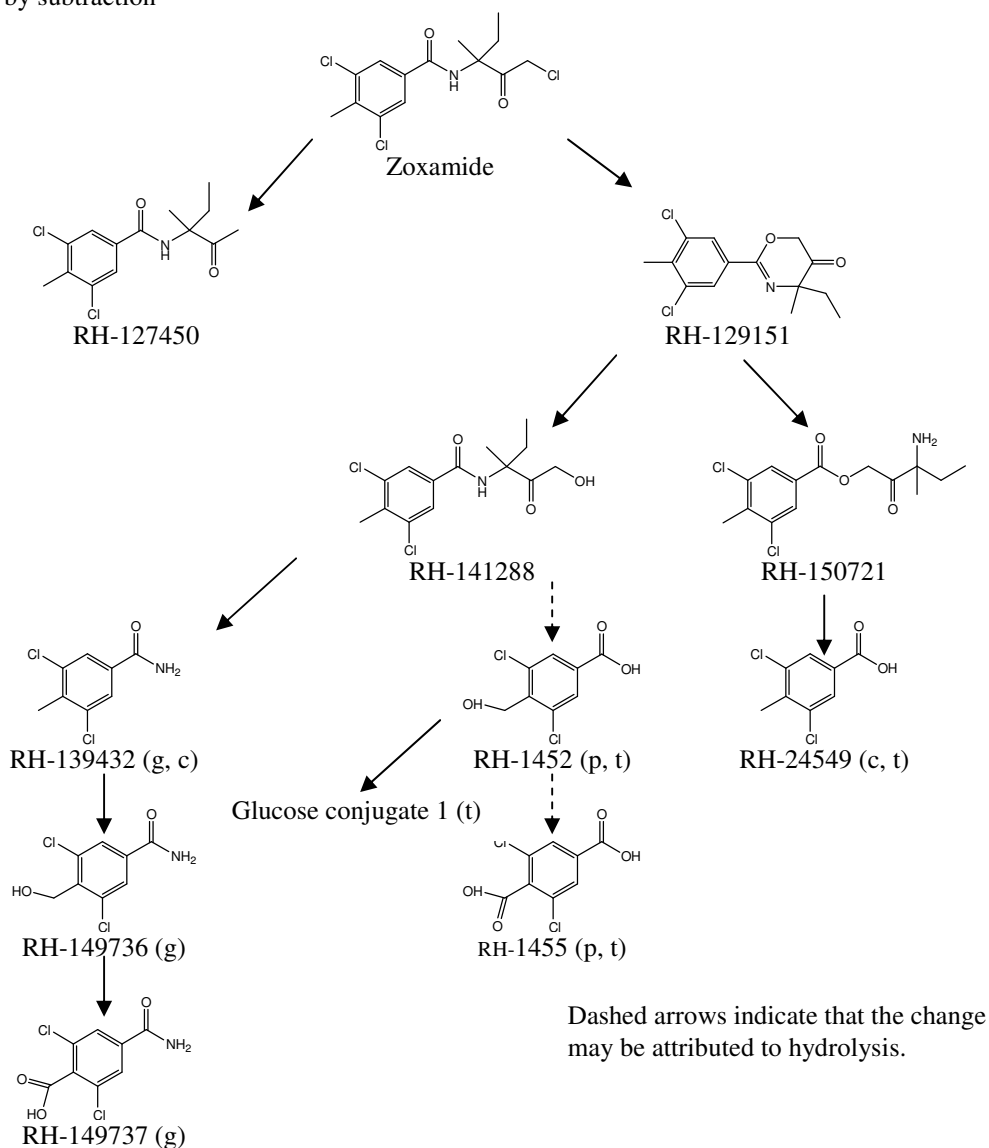


Figure 2. Proposed metabolic pathway of zoxamide in plants.

NB. c, g, p, t in parentheses mean that the compound was found only in cucumber, grape, potato or tomato, respectively

Environmental Fate in Soil

The Meeting received information on aerobic soil metabolism of zoxamide and its degradation products, a rotational crop study as well as numerous studies on photolysis in soil, absorption, desorption and mobility in soil and fate in water-sediment systems, water and air. Since zoxamide is intended for protection of potatoes and supervised trials were conducted with potatoes, studies on aerobic soil metabolism and rotational crops are relevant for the current review.

Aerobic soil metabolism

IHD Group Aerobic soil metabolism studies were conducted using [U-¹⁴C-phenyl]zoxamide applied to various soils which were then incubated under aerobic conditions. The studies are summarized below.

Under aerobic conditions, zoxamide applied to soil was rapidly degraded. After 120 – 122 days, 6 – 10% (dose rate, 1.5 mg/kg; 25 °C) or 0.6 – 1.0% (dose rate, 0.2 mg/kg; 20 °C) of applied zoxamide remained as the parent. Carbon dioxide was steadily evolved from all soils and accounted for 34 – 58% of the dose applied after 120 – 122 days. RH-127450, RH-129151, RH-24549, RH-139432 and RH-163353 were formed and then degraded during the study periods. Non-extractable radioactivity, 0.4 – 3.3% of the applied dose (3.3% in silt loam dosed at 1.5 mg/kg; for other soils tested 0.4 – 0.8%) on day 0, increased steadily to reach 24 – 38% of the applied dose on day 120 – 122. Several other degradates were observed at very low concentrations.

The half-life of zoxamide was calculated to be 10 days at 1.5 mg/kg dose rate (25 °C) and 2.0 – 4.2 days at 0.2 mg/kg dose rate (20 °C). When maintained at 10°C, the degradation of the parent was slower showing a half life of 7.7 days. None of RH-127450, RH-24549 and RH-163353 had a half-life longer than 13 days at 20 °C (applied dose of 0.2 mg/kg) however while when stored at 10 °C, the range was 28 – 89 days.

Soil: Loamy sand
Dose rate: 1.5 mg/kg
Duration: 122 days
Temperature: 25 ± 1°C
pH: 6.9
Half-life: 10 days

% zoxamide remaining at the end: 10%

Metabolites	Max % of dose	Day
RH-127450	5.71	14
RH-129151	1.60	3
RH-139432	1.11	3
RH-24549	7.42	14

Ref: Smalley, J., 1997; DERBI 92128
Moisture: 75% field moisture capacity
Organic carbon: 2.4%
¹⁴C accountability: 92-104%
% mineralization at the end: 34%
% unextractable at the end: 38%

Soil: Silt loam
Dose rate: 1.5 mg/kg
Duration: 122 days
Temperature: 25 ± 1°C
pH: 6.8
Half-life: 10 days

% zoxamide remaining at the end: 6.0%

Metabolites	Max % of dose	Day
RH-127450	2.94	3
RH-129151	0.58	3
RH-139432	0.47	3
RH-24549	6.06	3

Ref: Smalley, J., 1997; DERBI 92128
Moisture: 75% field moisture capacity
Organic carbon: 1.8%
¹⁴C accountability: 93-103%
% mineralization at the end: 48%
% unextractable at the end: 33%

Soil: Loam
 Dose rate: 0.2 mg/kg
 Duration: 120 days
 Temperature: 25 ± 1°C
 pH: 7.4
 Half-life: 2.0 days
 % zoxamide remaining at the end: 0.7%

Metabolites	Max % of dose	Day
RH-127450	11	3
RH-163353	13	3
RH-24549	12	3

Ref: Burgerer, A., 1998; DERBI 92179
 Moisture: 75% field moisture capacity
 Organic carbon: 2.3%
¹⁴C accountability: 94-100 %
 % mineralization at the end: 49%
 % unextractable at the end: 35%

Soil: Sandy loam
 Dose rate: 0.2 mg/kg
 Duration: 120 days
 Temperature: 20 ± 2°C
 pH: 7.4
 Half-life: 2.7 days
 % zoxamide remaining at the end: 1.0%

Metabolites	Max % of dose	Day
RH-127450	11	7
RH-163353	15	3
RH-24549	8.3	7

Ref: Burgerer, A., 1998; DERBI 92179
 Moisture: 50% max water-holding capacity
 Organic carbon: 1.2%
¹⁴C accountability: 95-102%
 % mineralization at the end: 58%
 % unextractable at the end: 29%

Soil: Sandy loam
 Dose rate: 0.2 mg/kg
 Duration: 120 days
 Temperature: 10 ± 1°C
 pH: 7.4
 Half-life: 7.7 days
 % zoxamide remaining at the end: 2.7%

Metabolites	Max % of dose	Day
RH-127450	9.4	14
RH-163353	12	14
RH-24549	13	7

Ref: Burgerer, A., 1998; DERBI 92179
 Moisture: 50% max water-holding capacity
 Organic carbon: 1.2%
¹⁴C accountability: 94-102%
 % mineralization at the end: 36%
 % unextractable at the end: 34%

Soil: Sandy loam
 Dose rate: 0.2 mg/kg
 Duration: 120 days
 Temperature: 20 ± 2°C
 pH: 7.4
 Half-life: 2.3 days
 % zoxamide remaining at the end: 0.6%

Metabolites	Max % of dose	Day
RH-127450	12	3
RH-163353	13	7
RH-24549	13	3

Ref: Burgerer, A., 1998; DERBI 92179
 Moisture: 100% field capacity
 Organic carbon: 1.2%
¹⁴C accountability: 92-101%
 % mineralization at the end: 56%
 % unextractable at the end: 28%

Soil: Clay loam
 Dose rate: 0.2 mg/kg
 Duration: 120 days
 Temperature: 20 ± 2°C
 pH: 8.1
 Half-life: 2.4 days
 % zoxamide remaining at the end: 0.7%

Metabolites	Max % of dose	Day
RH-127450	8.1	3
RH-163353	13	7
RH-24549	34	7

Ref: Burgerer, A., 1998; DERBI 92179
 Moisture: 75% field moisture capacity
 Organic carbon: 0.80%
¹⁴C accountability: 93-101%
 % mineralization at the end: 56%
 % unextractable at the end: 32%

Soil: Silt loam
 Dose rate: 0.2 mg/kg
 Duration: 120 days
 Temperature: 20 ± 2 °C
 pH: 5.0
 Half-life: 4.2 days
 % zoxamide remaining at the end: 2.5%

Ref: Burgerer, A., 1998; DERBI 92179
 Moisture: 75% field moisture capacity
 Organic carbon: 1.8%
 ^{14}C accountability: 91-101%
 % mineralization at the end: 43%
 % unextractable at the end: 24%

Metabolites	Max % of dose	Day
RH-127450	15	7
RH-163353	7.9	7
RH-24549	5.5	7

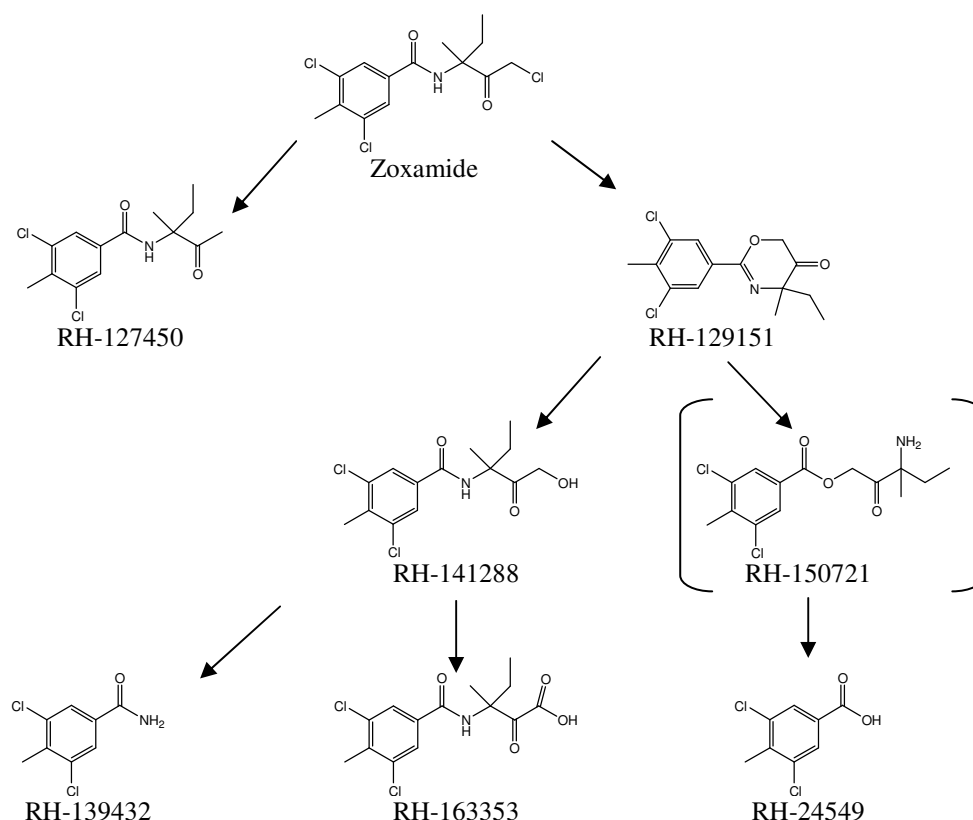


Figure 3. Proposed degradation pathway of zoxamide in soil

Residues in succeeding crops

In an outdoor confined rotation study conducted in North Carolina (Kim-Kang, 1998; DERBI 92158), mustard, radish, turnip, sorghum and soybean were planted at 30, 137, 210 and 365 days following the last of four applications of [^{14}C -phenyl]zoxamide (Radiopurity of test material for each treatment was > 96%). The active substance, formulated as an emulsifiable concentrate (EC), was applied to bare soil between mid April and early June (18 day intervals) at a rate of 0.5 kg ai/ha.

Crops were harvested at an intermediate stage and when mature. Crop and soil samples were processed within 1 month of harvest. Soil and plants samples were cryogenically milled and total radioactive residues (TRR) were determined by combustion analysis and LSC. Crop components (e.g., roots and tops, grain and straw) were analyzed separately. TRRs were very low for all samples at all plant back intervals. The results are shown in the following table.

Table 8. Total radioactive residues determined in rotational crops

Plantback Interval	Crop / Component	Radioactive residues (mg/kg parent equivalent)	
		Intermediate Harvest	Mature Harvest
30 days	Mustard	0.078	0.041
30 days	Radish root	0.033	0.023
30 days	Radish top	0.043	0.048
30 days	Sorghum stover	0.050	0.027
30 days	Sorghum grain	NA	0.026
30 days	Soybean hay	0.099	0.189
30 days	Soybean seed	NA	0.092
137 days	Mustard	0.020	< 0.01
137 days	Turnip root	0.023	0.042
137 days	Turnip top	0.011	< 0.01
137 days	Wheat grain	NA	< 0.01
137 days	Wheat straw	< 0.010	< 0.01
210 days	Mustard	0.042	0.030
210 days	Radish root	0.024	0.026
210 days	Radish top	0.038	0.031
210 days	Sorghum stover	0.017	< 0.01
210 days	Sorghum grain	NA	0.011
210 days	Soybean hay	0.034	0.024
210 days	Soybean seed	NA	0.020
365 days	Mustard	0.019	0.011
365 days	Radish root	0.014	0.011
365 days	Radish top	0.012	0.011
365 days	Sorghum stover	< 0.010	0.012
365 days	Sorghum grain	NA	0.014
365 days	Soybean hay	0.019	0.014
365 days	Soybean seed	NA	0.012

All crop samples containing residues more than 0.01 mg/kg, except for intermediate mustard leaf (137 DALA) and intermediate turnip root and top (137 DALA), were subjected to solvent extraction and analysis by HPLC, TLC and LC/MS. Homogenized crop samples were extracted with MeOH/H₂O/CHCl₃ and then with CHCl₃ and the extracts were combined. Following separation, the CHCl₃ fraction was first concentrated and partitioned with a mixture of CH₃CN and hexane to yield a CH₃CN-soluble fraction and a hexane-soluble fraction. Duplicate aliquots of each fraction were taken for LSC. The post-extraction solids (PES) were allowed to dry and were then subjected to combustion analysis.

In general, the amount of extractable residues was low in all the crop samples. Between 7% and 40% of the TRR was recovered in the polar MeOH/H₂O fractions for all the crops grown on treated soil. About 2 to 36% of the TRR was found in the organic extracts (CHCl₃, CH₃CN and hexane) of all the crops. The levels in these samples did not exceed 0.023 mg/kg. The values of extracts for all the crop samples showed a significant ratio of unextractable residues: generally 49% or greater, except for 365 DALA mature radish root which contained about 35% of the TRR as unextractable residues.

Analyses of metabolites in various fractions were performed using HPLC with radiometric detection. The overall distribution of metabolites in the extractable fractions showed metabolite A-II and RH-1452 as the major metabolites in most crop samples. However, only in samples of the 30 DALA soybean forage did levels exceed 0.01 mg/kg (levels were 0.016 and 0.023 mg/kg, respectively). Other metabolites were detected at low levels in some crops. Due to the low concentrations in all crops, the metabolites other than RH-1452 were not characterized.

Only intermediate 30 DALA radish root samples from the first planting contained Metabolite A-1 at a level greater than 0.01 mg/kg.

Among the PES fractions, those containing greater than 0.050 mg/kg of the TRR were subjected to a series of hydrolysis steps using enzymes, acid and base until the residue in the PES fraction became less than 0.050 mg/kg. The unextractable residue from 30 DALA soybean forage was subjected to enzyme hydrolysis using cellulase, which yielded 7.68% (0.008 mg/kg) of the TRR in the hydrolysate. The hydrolysate was not further analyzed due to the low residue level.

RESIDUE ANALYSIS

Analytical Methods

Analytical methods for determination of residues of zoxamide have been developed for a wide range of matrices including cucurbits, grapes, tomato, potato and relevant processed commodities. After an extraction specific to the matrix, and a reasonably standard cleanup, determination of zoxamide is made by gas chromatography generally using electron capture detection (GC/ECD) for quantitation and mass selective detection (GC/MSD) for confirmation. The methods presented for potato and its processed commodities determine zoxamide and the RH-1452 and RH-1455 metabolites. All other methods presented determine residues of only the parent compound.

Multi-Residue Methods

FDA Multi-Residue Method PAM, Vol. I, Appendix II (1/94)

The applicability of the multi-residue screen methods outlined in the FDA Pesticide Analytical Manual, Volume I, Appendix II (1/94) was assessed for zoxamide and two metabolites, RH-1455 and RH-1452 (Conrath, 1998; DERBI 91460). Using the multi-residue method, acceptable recoveries were generated for zoxamide but not for the metabolites.

Protocol A. Zoxamide and the two metabolites (RH-141452 and RH-141455) were tested to see if they were naturally fluorescent. None of the three produced a detectable emission response at an excitation wavelength of 216 nm, the absorbance maximum for all of them. Since natural fluorescence was not observed, Protocol A testing was not required.

Protocol B. Zoxamide is not an acid or a phenol, so testing it through Protocol B was not required. The metabolites, which are acids, were methylated and tested for recovery via florasil cleanup with GC/ECD detection resulting in recoveries > 86%. Recovery of the metabolites from potatoes was then tested using method 402 extraction E2 of the PAM manual with gel permeation chromatography and florasil cleanup. All recoveries of RH-1452 were < 20%. Recoveries of RH-1455 were 2% and 68% at 0.05 mg/kg and 2% at 0.50 mg/kg.

Protocol C. The gas chromatographic behavior of all three compounds was evaluated using multiple column types (DB-1, DB-17, DB-225 and DEGS) with electron capture detection (ECD), electrolytic conductivity detector (ELCD) in the halogen mode, or nitrogen-phosphorus detection (NPD). Zoxamide chromatographed within acceptable limits under Level I guidelines (180 and 200 °C) with all column/detector combinations. The chromatography of the methylated metabolites was also acceptable with all combinations tested. Additional work was done on determination of zoxamide exhibited sufficient response using ELCD such that the florasil cleanup was not necessary.

Protocol D. Testing (without florasil cleanup) for recovery of zoxamide from potatoes using ELCD resulted in an average recovery of 124% at 0.10 mg/kg 110% at 0.50 mg/kg.

Protocol E. Zoxamide was recoverable from the complete method in Protocol E with both the C1 and C2 cleanup with average recoveries of 100% and 107% respectively.

Protocol F. As potato was a non-fatty food, testing through Protocol F was not required.

Protocol G. Neither zoxamide nor the metabolites have an N-methylcarbamate structure and are not substituted urea compounds, so testing through Protocol G was not required.

Multi-Residue Enforcement Method DFG S19

The applicability of the multi-residue enforcement method DFG S19 (extended revision) as given in the Modular Multi Method L 00.00-34 of the Official Collection of Test Methods according to German §35 LMBG (Law of Food and Commodities), November 1999 was investigated using the following matrices: wheat grain (cereals and dry material), table grapes (watery material), whole oranges (acidic material) and rapeseed (oily and difficult matrix) (Class, 2001; DERBI 104700). For grain, grapes and oranges, samples were subjected to water/acetone extraction, partitioning into ethyl acetate/cyclohexane and clean up by gel permeation chromatography. For oilseed rape, extraction was with acetonitrile/acetone, followed by clean up with gel permeation chromatography. Determination was achieved using capillary gas chromatography with electron capture detection. Confirmation was achieved by mass spectrometric detection.

Acceptable recoveries were obtained at the LOQ of 0.01 mg/kg and at ten times the LOQ. No signals or peaks that could interfere with the determination of zoxamide were observed in untreated blank control samples of grain or grape. However, for orange and rapeseed, GC/ECD signals at a slightly shorter retention time interfered with the determination of zoxamide. For these matrices, background subtraction was required to determine zoxamide levels at or near the LOQ of the method.

Table 9. Summary of method validation of Multi-Residue Enforcement Method DFG S19

Matrix	Limit of quantitation	Recovery fortification level (mg/kg)	Recoveries (mean) %	Precision Repeatability RSD% (n)
Wheat grain	0.01 mg/kg	0.01	96	4 (5)
		0.1	91	5 (5)
White grapes	0.01 mg/kg	0.01	104	8 (5)
		5.0	80	4 (5)
Oranges, whole	0.01 mg/kg	0.01	93	13 (5)
		0.1	109	5 (5)
Rapeseed	0.01 mg/kg	0.01	96	5 (4)
		0.1	97	6 (5)

Analytical Methods Used in Supervised Trials and Processing Studies

Grapes and their processed products

Residues of zoxamide were determined in grapes using method TR 34-96-128 (Guo, 1996; DERBI 91475). This method was validated by an independent laboratory (Wais, 1999; DERBI 221739). The residues were extracted from the grapes using methanol/water (80/20 v/v) and blending for three minutes. The resulting slurry was filtered. The extract was added to a 0.1N NaCl/dichloromethane (2/1 v/v) mixture and partitioned into the organic phase. The organic layer was evaporated to dryness and reconstituted in hexane. Purification was carried out using carbon solid phase extraction (SPE) and alumina-B SPE. Following the SPE the eluate was evaporated and reconstituted in hexane and an aliquot was analyzed. Quantitation was performed by gas chromatography using electron capture detection (GC/ECD) and confirmation was done using mass selective detection (GC/MSD). The LOQ was 0.010 mg/kg.

Residues of zoxamide were determined in grape juice using method TR 34-97-136 (Kendi, 1998; DERBI 91467). The residues were extracted from the grape juice using a 10% NaCl/ethyl acetate (2/1 v/v) mixture and partitioned into the organic phase. The organic layer was then evaporated to dryness and reconstituted in hexane. Purification was carried out using alumina-B solid phase extraction (SPE). After the SPE eluate was evaporated and reconstituted in ethyl acetate, an aliquot was analyzed. Quantitation was performed by gas chromatography using electron capture detection (GC/ECD) and confirmation was done using mass selective detection (GC/MSD). The LOQ was 0.010 mg/kg.

Residues of zoxamide were determined in dried grapes using method TR 34-97-164 (Kendi, 1998; DERBI 91466). The dried grapes were prepared for analysis by first grinding them along with silica packing into a uniform powder. The powder was passed through a 14 mesh sieve and collected in a solid phase extraction tube containing only a frit at the bottom. The solid particulate that remained in the sieve was added on top of the powder. A second frit was then added to the top of the tube. The contents of the tube were then washed with hexane and the residues were eluted using 10/90 (v/v) ethyl acetate/hexane. The eluate was evaporated and reconstituted in hexane. Purification was carried out using Florasil solid phase extraction (SPE). The SPE eluate was evaporated to dryness and reconstituted in ethyl acetate and an aliquot was analyzed. Quantitation was performed by gas chromatography using electron capture detection (GC/ECD) and confirmation was done using mass selective detection (GC/MSD). The LOQ was 0.010 mg/kg.

Residues of zoxamide were determined in wine using method TR 34-98-148 (Kendi, 1998; DERBI 92251). This method was validated by an independent laboratory (Wais, 1999, DERBI 92292). The residues were extracted from wine by partitioning with ethyl acetate and 1% potassium bicarbonate. The organic layer was collected and concentrated to 9 – 10 mL and brought to a final volume of 10 mL using ethyl acetate. Quantitation was performed by gas chromatography using electron capture detection (GC/ECD) and confirmation was done using mass selective detection (GC/MSD). The LOQ was 0.010 mg/kg. The validation data for zoxamide are presented below.

Table 10. Procedural recovery of zoxamide from fortified grapes and processed products

Matrix Method	Fortification Level (mg/kg)	Recovery (%)		% RSD	N
		Range	Average		
Grapes TR 34-96-128	0.01	75 - 133	98	18	9
	0.02	84 - 97	89	6	4
	0.05	80 - 108	92	12	7
	0.1	83 - 116	103	17	3
	0.15	80 - 114	92	16	6
	0.01 - 0.15	75 - 133	95	15	29
Grape Juice TR 34-97-136	0.01	77 - 132	96	16	10
	0.02	61 - 105	89	18	6
	0.05	67 - 97	84	12	6
	0.1	60 - 107	89	19	6
	0.15	68 - 130	93	27	6
	0.01 - 0.15	60 - 132	91	18	34
Dried grapes TR 34-97-164	0.01	87 - 120	101	13	5
	0.05	75 - 93	86	8	6
	0.1	76 - 103	86	11	6
	0.5	63 - 96	77	16	6
	1.0	74 - 106	86	14	6
	0.01 - 1.0	63 - 120	87	15	29
Wine TR 34-98-148	0.01	91 - 125	107	12	8
	0.02	81 - 115	99	13	5
	0.05	91 - 114	101	7	7
	0.10	98 - 131	113	15	4
	0.15	70 - 105	94	14	7
	0.01 - 0.15	70 - 131	102	13	31

Cucurbits

Residues of zoxamide were determined in cucumbers, cantaloupe, zucchini and melons using method TR 34-99-43 (Guo, 1999; DERBI 92230). This method was validated by two independent laboratories (Wais, 1999; DERBI 224109 and Wasser, 2000; DERBI 224108). The matrices tested were cucumber, cantaloupe, melon and zucchini. The residues were extracted from these matrices using acetonitrile and blending for one minute. The extract was added to a 0.1N NaCl solution and was then partitioned into ethyl acetate. The organic layer was evaporated to dryness and dissolved in hexane. Further purification was achieved using carbon solid phase extraction (SPE) and alumina-B SPE. Following the SPE the eluate was evaporated and reconstituted in hexane and an aliquot was analyzed. Quantitation was performed by gas chromatography using electron capture detection

(GC/ECD) and confirmation was done using mass selective detection (GC/MSD). The LOQ was 0.010 mg/kg.

Table 11. Procedural recovery of zoxamide from fortified cucurbits, samples were analyzed by method TR 34-99-43

Matrix	Fortification Level (mg/kg)	Recovery (%)		% RSD	N
		Range	Average		
Cucumber	0.01	78 - 108	92	9	19
	0.02	70 - 98	80	12	9
	0.05	75 - 101	87	11	9
	0.1	73 - 104	83	9	15
	1.0	74 - 89	83	6	6
	0.01 - 1.0	70 - 108	86	11	58
Cantaloupe	0.01	73 - 99	89	13	4
	0.02	83 - 85	84	2	2
	0.05	85 - 94	89	8	2
	0.1	94 - 95	95	1	2
	0.01 - 0.1	73 - 99	89	9	10
	Zucchini	0.01	83 - 100	90	9
0.02		86 - 100	93	11	2
0.05		89 - 96	93	5	2
0.1		80 - 95	87	12	2
0.01 - 0.1		80 - 100	91	8	10
Melon (flesh)		0.01	72 - 102	79	17
	0.02	73 - 83	77	5	5
	0.05	73 - 80	77	4	5
	0.1	70 - 82	76	6	5
	0.01 - 0.1	70 - 102	77	9	20
	Melon (skin)	0.01	72 - 93	83	10
0.02		73 - 81	76	4	5
0.05		72 - 83	76	6	5
0.1		72 - 82	76	5	5
0.01 - 0.1		72 - 93	78	7	20
Melon (whole)		0.01	77 - 92	85	7
	0.1	81 - 99	90	9	4
	1.0	74 - 88	80	7	4
	0.01 - 1.0	74 - 99	85	9	12

Tomato and its processed products

Residues of zoxamide were determined in tomatoes using method TR 34-99-68 (Burdge, 1999; DERBI 92229). This method was validated by an independent laboratory (Wais, 2000; DERBI 138543). The residues were extracted from the tomatoes by blending with acetonitrile. The resulting slurry was then filtered. The extract was evaporated to approximately 10 mL. A liquid-liquid partition was performed using a 0.1 M sodium bicarbonate solution and ethyl acetate. The organic layer was collected, evaporated to dryness and reconstituted in hexane. Purification was carried out using carbon solid phase extraction (SPE) and alumina-B SPE. The SPE eluate was evaporated and reconstituted in hexane and an aliquot was analyzed. Quantitation was performed by gas chromatography using electron capture detection (GC/ECD) and confirmation was done using mass selective detection (GC/MSD). The LOQ was 0.010 mg/kg.

Residues of zoxamide were determined in tomato puree and paste using method TR 34-99-95 (Kurilla, 1999; DERBI 92234). The paste or puree was weighed into a mortar, ground along with Bondesil until homogeneous and dried under vacuum. The dried material was broken up and added to a solid phase extraction tube containing a frit at the bottom, aluminium oxide, basic powder and a second frit. The dried material was added on top of the second frit, a third frit was added to the top of the tube. The residue was then extracted using acetonitrile. The eluate was evaporated and reconstituted in hexane. Purification was carried out using carbon solid phase extraction (SPE) and alumina-B SPE. The SPE eluate was evaporated and reconstituted in hexane and an aliquot was analyzed. Quantitation was performed by gas chromatography using electron capture detection

(GC/ECD) and confirmation was done using mass selective detection (GC/MSD). The LOQ was 0.010 mg/kg.

Table 12. Procedural recovery of zoxamide from fortified tomato and processed products

Matrix Method	Fortification Level (mg/kg)	Recovery (%)		% RSD	N
		Range	Average		
Tomato TR 34-99-68	0.01	74 - 103	89	10	15
	0.02	86 - 125	102	15	5
	0.05	87 - 104	95	7	6
	0.1	71 - 97	81	11	10
	0.01 - 0.1	71 - 125	89	13	36
Tomato Puree TR 34-99-95	0.01	70 - 113	92	18	10
	0.02	83 - 110	94	13	5
	0.05	83 - 102	91	10	4
	0.1	85 - 104	96	8	4
	0.01 - 0.1	70 - 113	93	14	23
Tomato Paste TR 34-99-95	0.01	77 - 107	91	9	9
	0.02	82 - 98	91	7	5
	0.05	87 - 98	93	5	4
	0.1	87 - 94	91	4	3
	0.01 - 0.1	77 - 107	91	7	21

Potato and its processed product and by-product

Residues of zoxamide and its two acid metabolites, RH-1452 and RH-1455, were determined in potatoes using method TR 34-97-90 (Desai, 1998; DERBI 91463). The residues were extracted from the potatoes by blending with acetonitrile/2% potassium bicarbonate (80:20 v/v). The resulting slurry was filtered. The extract was evaporated to approximately 30 mL. A liquid-liquid partition was performed using a 2% potassium bicarbonate solution and ethyl acetate. The organic layer, containing zoxamide, was collected, evaporated to dryness and reconstituted in hexane. The resulting solution was purified using Florisil and alumina-B SPE. Following the SPE the eluate was evaporated and reconstituted in hexane and an aliquot was analyzed.

The aqueous layer from the liquid-liquid partition, containing the two metabolites, was acidified and partitioned into ethyl acetate to which sodium sulfate was added. The solution was then evaporated to dryness and reconstituted in ethyl acetate/methanol. Diazomethane was added to the solution to methylate the metabolites. Following methylation, the solution was evaporated to dryness and reconstituted in hexane. Further purification was achieved using florisil solid phase extraction (SPE). The SPE eluate was evaporated and reconstituted in hexane and an aliquot was analyzed.

For all analytes, quantitation was performed by gas chromatography using electron capture detection (GC/ECD) or mass selective detection (GC/MSD). The LOQ was 0.020 mg/kg.

The method was also radiovalidated (Desai, 1998; DERBI 92232). A sample from the ¹⁴C-metabolism study was reanalysed using this method. The results of the radiovalidation agreed with the results of the metabolism study (Reibach, 1998; DERBI 91482) thus supporting the accuracy of the method. The concentrations of zoxamide, RH-1452 and RH-1455 found in a sample from the metabolism study were 0.00, 0.037 and 0.069 mg/kg, respectively, and the amounts found in that sample using the analytical method were 0.00, 0.038, and 0.065 mg/kg.

Residues of zoxamide and its two acid metabolites, RH-1452 and RH-1455, were determined in potato chips and flakes using method TR 34-98-140 (Desai, 1998; DERBI 91465). The residues were extracted from the potato chips and flakes with acetonitrile/2% potassium bicarbonate (80:20 v/v) in an accelerated solvent extractor. A liquid-liquid partition was performed using a 2% potassium bicarbonate solution and ethyl acetate. The organic layer, containing zoxamide, was collected, evaporated to < 1 mL and dissolved in hexane. The resulting solution was purified using Florisil and alumina-B SPE. The SPE eluate was evaporated and reconstituted in cyclohexane and an aliquot was analyzed.

The aqueous layer from the liquid-liquid partition, containing the two metabolites, was acidified and partitioned into ethyl acetate to which sodium sulfate was added. The solution was then evaporated to < 1 mL and dissolved in dichloromethane. Diazomethane was then added to the solution to methylate the metabolites. Following methylation the solution was evaporated to near dryness and reconstituted in hexane. Purification was carried out using Florisil solid phase extraction (SPE). The SPE eluate was evaporated and reconstituted in toluene and an aliquot was analyzed.

For all analytes, quantitation was performed by gas chromatography using mass selective detection (GC/MSD). Confirmation was done using electron capture detection (GC/ECD). The LOQ was 0.020 mg/kg.

Residues of zoxamide and its two acid metabolites, RH-1452 and RH-1455, in potato peel waste were determined using method TR 34-00-34 (Guo, 2000; DERBI 92657). Residues were extracted by blending homogenized peel with acetonitrile/2% sodium bicarbonate (80:20 v/v). The extract was concentrated and the residues were separated via a liquid-liquid partition using acetonitrile/2% sodium bicarbonate (80:20 v/v) and ethyl acetate. The layers were separated and taken through additional clean-up steps. Zoxamide in the organic layer was passed through Florisil and alumina-B solid phase extraction. The metabolites in the aqueous phase were methylated and passed through a Florisil column. For all analytes, quantitation was performed by gas chromatography using electron capture detection (GC/ECD). The LOQ was 0.020 mg/kg.

Table 13. Procedural recovery of zoxamide, RH-1452 and RH-1455 from fortified potato, its processed products and by-product

Matrix Method Analyte	Fortification Level (mg/kg)	Recovery (%)		% RSD	N
		Range	Average		
Potato TR 34-97-90					
Zoxamide (GC/ECD)	0.02	65 - 101	79	20	6
	0.05	99 - 103	101	2	3
	0.10	59 - 96	73	28	3
	0.15	79 - 84	81	4	2
	0.02 - 0.15	59 - 103	83	20	14
Zoxamide (GC/MSD)	0.02	57 - 93	74	19	5
	0.05	74 - 100	87	21	2
	0.10	71 - 106	90	20	3
	0.15	97 - 106	102	6	2
	0.02 - 0.15	57 - 106	85	20	12
RH-1452 (GC/ECD)	0.02	57 - 93	81	16	6
	0.05	77 - 92	84	9	3
	0.10	72 - 79	75	5	3
	0.15	64 - 81	71	12	3
	0.02 - 0.15	57 - 93	79	13	15
RH-1452 (GC/MSD)	0.02	57 - 125	96	25	8
	0.05	91 - 117	102	11	4
	0.10	87 - 118	104	13	4
	0.15	64 - 81	71	12	3
	0.02 - 0.15	57 - 143	104	20	20
RH-1455 (GC/ECD)	0.02	45 - 96	68	33	5
	0.05	70 - 103	86	19	3
	0.10	70 - 101	85	18	3
	0.15	67 - 92	78	16	3
	0.02 - 0.15	45 - 103	78	23	14
RH-1455 (GC/MSD)	0.02	60 - 94	79	17	8
	0.05	71 - 113	88	20	4
	0.10	81 - 98	92	8	4
	0.15	67 - 92	78	16	3
	0.02 - 0.15	60 - 113	87	15	20

Matrix Method Analyte	Fortification Level (mg/kg)	Recovery (%)		% RSD	N
		Range	Average		
Potato chips TR 34-00-34					
Zoxamide	0.02	107 - 121	114	9	2
	0.05	81 - 81	81	-	1
	0.10	117 - 117	117	-	1
	0.20	80 - 80	80	-	1
	0.02-0.20	80 - 121	101	19	5
RH-1452	0.02	77 - 124	109	14	8
	0.04	86 - 93	89	6	2
	0.05	94 - 110	103	7	4
	0.10	84 - 114	102	13	5
	0.20	75 - 107	96	13	5
	0.40	103 - 103	103	-	1
0.02 - 0.40	75 - 124	102	13	25	
RH-1455	0.02	93 - 131	113	10	8
	0.04	79 - 84	81	4	2
	0.05	77 - 93	87	8	4
	0.10	76 - 106	87	13	5
	0.20	59 - 108	83	21	5
	0.40	78 - 78	78	-	1
	0.02 - 0.40	59 - 131	94	18	25
Potato flakes TR 34-00-34					
Zoxamide	0.02	108 - 126	117	11	2
	0.05	86 - 86	86	-	1
	0.10	88 - 88	88	-	1
	0.20	88 - 88	88	-	1
	0.02-0.20	86 - 126	99	18	5
RH-1452	0.02	94 - 115	103	7	8
	0.05	85 - 108	95	10	4
	0.10	87 - 118	103	15	4
	0.20	75 - 111	98	17	4
	0.02 - 0.20	75 - 118	100	11	20
RH-1455	0.02	81 - 124	97	18	8
	0.05	64 - 105	86	20	4
	0.10	70 - 107	88	18	4
	0.20	63 - 146	95	39	4
	0.02 - 0.20	63 - 146	93	22	20
Potato peel TR 34-00-34					
Zoxamide	0.02	77 - 131	95	20	10
	0.05	68 - 106	89	15	10
	0.10	73 - 99	89	12	5
	0.02-0.10	68 - 131	91	17	25
RH-1452	0.02	76 - 161	96	28	10
	0.05	61 - 116	90	20	10
	0.10	63 - 95	82	16	5
	0.02 - 0.10	61 - 161	91	23	25
RH-1455	0.02	53 - 135	97	30	10
	0.05	68 - 108	86	18	10
	0.10	56 - 103	79	21	5
	0.02 - 0.10	53 - 135	89	26	25

Grapes, Potatoes, and Tomatoes

Residues of zoxamide in grapes, potatoes and tomatoes were determined using method GRM 02.07.R1 (Pinheiro, 2002; DERBI 221152). Residues were extracted from the grape samples by homogenizing with methanol; and from tomato and potato samples with acetonitrile/2% potassium bicarbonate (80:20 v/v). An aliquot was partitioned with hexane and the organic phase was discarded. The remaining phase was then partitioned with toluene and cleaned using silica SPE. The eluate was

evaporated to dryness and reconstituted in ethyl acetate containing an internal standard. Analysis was performed using gas chromatography with mass selective detection (GC/MSD). The method LOQ was 0.01 mg/kg.

Table 14. Procedural recovery of zoxamide from fortified grape, potato and tomato, samples were analyzed using method GRM 02.07.R1

Matrix	Fortification Level (mg/kg)	Recovery (%)		% RSD	N
		Range	Average		
Grape	0.01	82 - 102	94	7	7
	3.0	87 - 114	98	15	3
	0.01 - 3.0	82 - 114	95	9	10
Potato	0.01	102 - 114	108	4	9
	0.10	79 - 87	83	5	3
	0.01 - 0.10	79 - 114	102	12	12
Tomato	0.01	73 - 91	83	8	7
	0.1	99 - 104	103	3	3
	0.2	96 - 102	99	3	3
	0.01 - 0.2	73 - 104	91	11	13

Enforcement Methods

Grapes, Grape Juice and Dried grapes

The methods TR 34-96-128, TR 34-97-136 and TR 34-97-164 were compiled and subjected to radiovalidation using grapes from the grape metabolism study (Reibach, 1998; DERBI 91481). This compilation was issued as the tolerance enforcement method (Burdge, 1998; DERBI 92154). The enforcement method was validated by an independent laboratory (Szuter, 1998; DERBI 92291). The results of the radiovalidation agreed with the results of the metabolism study thus supporting the accuracy of the method. The concentration of zoxamide found in a sample from the metabolism study was 0.429 mg/kg and the amount found in that sample using the enforcement method was 0.408 mg/kg (95%).

Table 15. Independent laboratory validation of enforcement method for grapes, grape juice and dried grapes

Matrix	Fortification Level (mg/kg)	Recovery (%)		% RSD	N
		Range	Average		
Grapes	0.01	91 - 119	105	19	2
	5.0	77 - 90	84	12	2
	0.01 - 5.0	77 - 119	94	19	4

Cucurbits

The method TR 34-99-43 was radiovalidated and issued as the tolerance enforcement method (Guo, 1999; DERBI 91461). The enforcement method was then validated by an independent laboratory (Tauber, 1999; DERBI 92655). The results of the radiovalidation agreed with the results of the relevant metabolism study (Sharma, 1999; DERBI 91483) thus supporting the accuracy of the method. For the combustion, extraction and quantitation steps the enforcement method showed 61,600 dpm/g, 106% and 1.28 mg/kg, respectively compared to 60,089 dpm/g, 119% and 1.33 mg/kg for the metabolism study.

Table 16. Independent laboratory validation of enforcement method for cucumber

Matrix	Fortification Level (mg/kg)	Recovery (%)		% RSD	N
		Range	Average		
Cucumber	0.01	83 - 84	83	1	2
	2.0	79 - 81	80	2	2
	0.01 - 2.0	79 - 84	82	3	4

Tomato, Tomato Puree and Tomato Paste

The methods TR 34-99-68 and TR 34-99-95 were compiled and subjected to radiovalidation using tomatoes from the metabolism study (Sharma, 1999; DERBI 91486). This compilation was issued as the tolerance enforcement method (Burdge, 1999; DERBI 91462). The enforcement method was validated by an independent laboratory (Bruns, 1999; DERBI 92656). The results of the radiovalidation agreed with the results of the metabolism study thus supporting the accuracy of the method. The concentration of zoxamide found in a sample from the metabolism study was 0.116 mg/kg and the amount found in that sample using the enforcement method was 0.092 mg/kg (79%).

Table 17. Independent laboratory validation of method TR 34-99-43 for tomato, tomato puree and tomato paste

Matrix	Fortification Level (mg/kg)	Recovery (%)		% RSD	N
		Range	Average		
Tomato	0.01	83-97	90	11	2
	2.0	71-79	75	8	2
	0.01 - 2.0	71-97	82	13	4

Potato and Potato Processed Fractions

The methods TR 34-97-90 and TR 34-00-34 were compiled and issued as the tolerance enforcement method TR34-98-142 (Meyer, 1998; DERBI 91480). The enforcement method was validated by an independent laboratory (Bruns, 1998; DERBI 92231).

Table 18. Independent laboratory validation of method TR 34-98-142 for potato and its processed products and by-product

Matrix Analyte	Fortification Level (mg/kg)	Recovery (%)		% RSD	N
		Range	Average		
Potato					
Zoxamide (GC/MSD)	0.02	73 - 87	80	13	2
	0.05	92 - 93	92	0	2
	0.02 - 0.05	73 - 93	86	11	4
Zoxamide (GC/ECD)	0.02	79 - 84	82	4	2
	0.05	79 - 111	95	24	2
	0.02 - 0.05	79 - 111	88	17	4
RH-1452 (GC/MSD)	0.02	72 - 113	93	31	2
	0.05	72 - 80	76	7	2
	0.02 - 0.05	72 - 113	84	23	4
RH-1452 (GC/ECD)	0.02	109 - 111	110	2	2
	0.05	80 - 83	81	3	2
	0.02 - 0.05	80 - 111	96	17	4
RH-1455 (GC/MSD)	0.02	71 - 104	88	27	2
	0.05	72 - 96	84	20	2
	0.02 - 0.05	71 - 104	86	20	4
RH-1455 (GC/ECD)	0.02	108 - 109	108	1	2
	0.05	86 - 91	89	4	2
	0.02 - 0.05	86 - 109	99	12	4

Stability of Pesticide Residues in Stored Analytical Samples

The Meeting received information on frozen storage stability of residues of zoxamide in grapes, grape juice, dried grapes, wine, cucumbers, tomatoes, tomato juice, tomato paste, tomato puree and potatoes.

Grapes, grape juice, dried grapes and wine

The stability of zoxamide in grapes stored frozen was evaluated by fortifying homogenized grapes in glass 20 mL bottles with approximately 0.1 – 0.6 mg/kg of zoxamide (Ross, 1998; DERBI 91487). The samples were capped and placed in frozen storage at approximately -20 °C. Day 0 fortifications were verified by dosing empty bottles with an aliquot of the zoxamide fortification solution and analysing. Samples were periodically removed from frozen storage and analyzed over a 17 month period. Residue concentrations were determined by GC/ECD analysis using method TR 34-96-128 (Guo, 1996; DERBI 91475). Each analysis set contained a control sample, two procedural recovery samples and the storage stability samples. A total of four procedural recoveries ranged from 83 – 99% with an average of 90% and a standard deviation of 7.5%. The results of the study showed zoxamide to be stable when stored frozen in grapes for up to 14 months.

Table 19. Storage stability of zoxamide in grapes fortified with zoxamide

Matrix	Storage day	Fortification mg/kg	% Remaining ^a	
			Results	Average
Grapes	0	1.0	94, 83, 85, 99	90
	166	0.098	86, 80, 85	84
	255	0.186	90, 75, 69	78
	341	0.576	111, 115, 107	111
	433	0.098	141, 99, 112	117

a - % remaining=(measured concentration)/(concentration of fortification)×100

The stability of zoxamide in grape juice stored frozen was evaluated by fortifying grape juice in glass 20 mL bottles with approximately 0.1 – 0.9 mg/kg of zoxamide (Reibach, 2001; DERBI 135577). The samples were capped and placed in frozen storage at approximately -20° C. Day 0 fortifications were verified by dosing empty bottles with an aliquot of the zoxamide fortification solution and analysing. Samples were periodically removed from frozen storage and analyzed over a 29 month period. Residue concentrations were determined by GC/ECD analysis using method TR 34-97-136 (Kendi, 1998; DERBI 91467) through 307 days of storage and TR 34-98-150 (Burdge, 1998; DERBI 92154) for analyses performed after that point. Each analysis set contained a control sample, two procedural recovery samples and the storage stability samples. A total of four procedural recoveries were tested using TR 34-97-136, they ranged from 93 – 109% with an average of 99% and a standard deviation of 7.1%. A total of eight concurrent recoveries were tested using TR 34-98-150, they ranged from 52 – 79% with an average of 68% and a standard deviation of 9.5%. The results of the study showed zoxamide to be relatively stable when stored frozen in grape juice for 29 months.

Table 20. Storage stability of zoxamide in grape juice fortified with zoxamide

Matrix	Storage days	Fortification mg/kg	% Remaining ^a	
			Results	Average
Grape Juice	0	1.0	93, 95, 100, 109	99
	32	0.942	70, 55, 62	62
	125	0.102	66, 86, (16.6)	76
	212	0.248	49, 46, 74	56
	307	0.187	72, 90, 68	77
	572	0.942	69	84
	580		99	
	665	0.102	60	61
	667		65	
	673		57	

Matrix	Storage days	Fortification mg/kg	% Remaining ^a	
			Results	Average
	756	0.248	94	75
	758		67	
	763		64	
	851	0.187	76	78
	853		65	
	858		81	

a - % remaining=(measured concentration)/(concentration of fortification)×100

The number in parentheses was not used in the calculation of the average.

The stability of zoxamide in dried grapes stored frozen was evaluated by fortifying homogenized raisins in glass 20 mL bottles with approximately 0.1 – 0.8 mg/kg of zoxamide (Reibach, 2001; DERBI 135576). The samples were capped and placed in frozen storage at approximately -20 °C. Day 0 fortifications were verified by dosing empty bottles with an aliquot of the zoxamide fortification solution and analysing. Samples were periodically removed from frozen storage and analyzed over a 26 month period. Residue concentrations were determined by GC/ECD analysis using methods TR 34-97-164 (Kendi, 1998; DERBI 91466) through 307 days of storage and TR 34-98-150 (Burdge, 1998; DERBI 92154) for analyses performed after that point. Each analysis set contained a control sample, at least two procedural recovery samples and the storage stability samples. A total of five procedural recoveries were analyzed using TR 34-97-164, they ranged from 73 – 90% with an average of 78% and a standard deviation of 6.9%. A total of four procedural recoveries were analyzed using TR 34-98-150, they ranged from 90 – 101% with an average of 95% and a standard deviation of 4.6%. The results of the study showed zoxamide to be relatively stable when stored frozen in raisins for 26 months.

Table 21. Storage stability of zoxamide in dried grapes fortified with zoxamide

Matrix	Storage day	Fortification mg/kg	% Remaining ^a	
			Results	Average
Dried grapes	0	1.0	75, 80, 75, 73, 90	79
	32	0.840	109, 108, 102	106
	125	0.109	70, 92, 92	85
	212	0.288	70, 75, 52	65
	307	0.230	42, 45, 63	50
	504	0.840	102	99
	510		96	
	597	0.109	86	86
	603		(270)	
	688	0.288	74	74
	694		74	
	783	0.230	64	71
	789		77	

a - % remaining=(measured concentration)/(concentration of fortification)*100

Number in parentheses was not used in the calculation of the averages.

The stability of zoxamide in wine stored frozen was evaluated by reanalysing wine samples from a vinification study (Mamouni, 1998; DERBI 92294) after about 8 months of frozen storage at approximately -20 °C. Radioactive residues of zoxamide were determined in wine samples at the end of the fermentation process and again at the end of the frozen storage period using HPLC with a ¹⁴C-detector. One white and one red wine sample were reanalyzed showing 100 and 104% remaining after storage. These results showed zoxamide to be stable when stored frozen in wine for at least 8 months.

Cucumbers

The stability of zoxamide in cucurbits stored frozen was evaluated by fortifying homogenized cucumbers in glass 20 mL bottles with approximately 1 mg/kg of zoxamide (Graves, 2001; DERBI 135579). The samples were capped and placed in frozen storage at approximately -20 °C. Day 0

fortifications were verified by dosing empty bottles with an aliquot of the zoxamide fortification solution and analysing. Samples were periodically removed from frozen storage and analyzed over a 28 month period. Residue concentrations were determined by GC/ECD analysis using method TR 34-99-43 (Guo, 1999; DERBI 92230). Each analysis set contained a control sample, two procedural recovery samples and three storage stability samples. A total of eleven procedural recoveries ranged from 80 – 97% with an average of 88% and a standard deviation of 5.3%. The results of the study showed zoxamide to be stable when stored frozen in cucumbers for at least 28 months.

Table 22. Storage stability of zoxamide in cucumber fortified with zoxamide

Matrix	Storage day	Fortification mg/kg	% Remaining ^a		
			Results	Average	
Cucumber	0	0.1	86, 80	83	
		0.5	97, 94, 92, 86	92	
		1.0	87, 92, 82, 84, 84	86	
	52 57 58	1.02		104	98
				96	
				94	
	127 132 133	1.00		97	95
				95	
				92	
	232 237 238	0.93		94	95
				96	
				94	
682 686 688	1.02		73	77	
			79		
			80		
	757 761 763	1.00	77	80	
			85		
			79		
	862 866 868	0.93		93	90
				93	
				84	

a - % remaining=(measured concentration)/(concentration of fortification)×100

Tomatoes, tomato juice, tomato paste and tomato puree

The stability of zoxamide in tomatoes stored frozen was evaluated by fortifying homogenized tomatoes in glass 30 mL vials with approximately 1 mg/kg of zoxamide (Hanauer, 2001; DERBI 135578). The samples were capped and placed in frozen storage at approximately -20 °C. Day 0 fortifications were verified by dosing empty vials with an aliquot of the zoxamide fortification solution and analysing. Samples were periodically removed from frozen storage and analyzed over a 27 month period. Residue concentrations were determined by GC/ECD analysis using method TR 34-99-68 (Brudge, 1999; DERBI 92229). Each analysis set contained a control sample, two procedural recovery samples and the storage stability samples. A total of nine procedural recoveries (excluding one outlier) ranged from 94 – 117% with an average of 101% and a standard deviation of 10.4%. The results of the study showed zoxamide to be stable when stored frozen in tomatoes for at least 27 months.

Table 23. Storage stability of zoxamide in tomatoes fortified with zoxamide

Matrix	Storage day	Fortification mg/kg	% Remaining ^a		
			Results	Average	
Tomatoes	0	0.05	94	94	
		0.1	94, 85, 110	96	
		0.5	117, 97	107	
		1	112, 96, 103	104	
	43 44 53	1.04		67	74
				67	
				88	

Matrix	Storage day	Fortification mg/kg	% Remaining ^a	
			Results	Average
Tomatoes	124	1.17	61	70
	125		65	
	134		84	
	230	1.00	67	84
	231		77	
	240		109	
	622	1.04	96	96
			96	
			97	
	810	1.00	95	100
			105	
			100	

a - % remaining=(measured concentration)/(concentration of fortification)×100

The stability of zoxamide in tomato juice stored frozen was evaluated by fortifying store bought tomato juice in glass 30 mL vials with approximately 1 mg/kg of zoxamide (Hanauer, 2001; DERBI 135640). The samples were capped and placed in frozen storage at approximately -20 °C. Day 0 fortifications were verified by dosing empty vials with an aliquot of the zoxamide fortification solution and analysing. Samples were periodically removed from frozen storage and analyzed over a 27 month period. Residue concentrations were determined by GC/ECD analysis using method TR 34-99-91 (Guo, 1999; DERBI 92228). Each analysis set contained a control sample, two procedural recovery samples and the storage stability samples. A total of six procedural recoveries ranged from 82 – 93% with an average of 90% and a standard deviation of 4.2%. The results of the study showed zoxamide to be stable when stored frozen in tomato juice for at least 27 months.

Table 24. Storage stability of zoxamide in tomato juice fortified with zoxamide

Matrix	Storage day	Fortification mg/kg	% Remaining ^a	
			Results	Average
Tomato juice	0	0.1	92	92
		0.5	92, 88	90
		1.0	93, 91, 82	89
	354	1.06	78	85
	356		87	
	370		82	
	370		91	
	629	1.05	77	77
	631		80	
	645		75	
	710	1.09	73	72
	712		62	
	726		81	
	816	1.01	90	90
	818		88	
	832		93	

a - % remaining=(measured concentration)/(concentration of fortification)×100

The stability of zoxamide in tomato paste stored frozen was evaluated by fortifying store bought tomato paste in plastic 30 mL vials with approximately 1 – 2 mg/kg of zoxamide (Reibach, 1999; DERBI 92672). The samples were capped and placed in frozen storage at < -10 °C. Day 0 fortifications were verified by dosing empty vials with an aliquot of the zoxamide fortification solution and analysing. Samples were periodically removed from frozen storage and analyzed over an 8 month period. Residue concentrations were determined by GC/ECD analysis using method TR 34-99-95 (Kurilla, 1999; DERBI 92234). Each analysis set contained a control sample, two procedural recovery samples and the storage stability samples. A total of six procedural recoveries ranged from 97 – 114% with an average of 107% and a standard deviation of 6.8%. The results of the study showed zoxamide to be stable when stored frozen in tomato paste for at least 8 months.

Table 25. Storage stability of zoxamide in tomato paste fortified with zoxamide

Matrix	Storage day	Fortification mg/kg	% Remaining ^a		
			Results	Average	
Tomato paste	0	0.5	111, 109, 112	111	
		1.0	97, 114, 101	104	
		1.06	92	99	
	43	44	50	104	75
				100	
				66	
	124	125	131	76	105
				83	
				102	
	230	231	237	110	103
				103	
				103	

a - % remaining=(measured concentration)/(concentration of fortification)×100

The stability of zoxamide in tomato puree stored frozen was evaluated by fortifying store bought tomato puree in plastic 30 mL vials with approximately 1 mg/kg of zoxamide (Reibach, 1999; DERBI 92645). The samples were capped and placed in frozen storage at approximately -20 °C. Day 0 fortifications were verified by dosing empty vials with an aliquot of the zoxamide fortification solution and analysing. Samples were periodically removed from frozen storage and analyzed over an 8 month period. Residue concentrations were determined by GC/ECD analysis using method TR 34-99-95 (Kurilla, 1999; DERBI 92234). Each analysis set contained a control sample, two procedural recovery samples and the storage stability samples. A total of six procedural recoveries ranged from 90 – 103% with an average of 97% and a standard deviation of 4.2%. The results of the study showed zoxamide to be stable when stored frozen in tomato puree for at least 8 months.

Table 26. Storage stability of zoxamide in tomato paste fortified with zoxamide

Matrix	Storage day	Fortification mg/kg	% Remaining ^a		
			Results	Average	
Tomato puree	0	0.2	103	103	
		0.5	97, 97	97	
		1.0	97, 90, 99	95	
	34	35	41	83	78
				78	
				75	
	115	116	122	93	87
				83	
				85	
	221	222	228	99	92
				82	
				94	

a - % remaining=(measured concentration)/(concentration of fortification)×100

Potatoes

The stability of zoxamide in potatoes stored frozen was evaluated by fortifying homogenized potatoes in glass 20 mL bottles with approximately 0.06 – 0.9 mg/kg of zoxamide (Ross, 1998; DERBI 91487; Reibach, 2000; DERBI 138407). The samples were capped and placed in frozen storage at approximately -20 °C. Day 0 fortifications were verified by dosing empty bottles with an aliquot of the zoxamide fortification solution and analysing. Samples were periodically removed from frozen storage and analyzed over a 24 month period. Residue concentrations were determined by GC/ECD analysis using method TR 34-97-90 (Desai, 1998; DERBI 91463). Each analysis set contained a control sample, two procedural recovery samples and the storage stability samples. A total of five procedural recoveries ranged from 82 – 103% with an average of 91% and a standard deviation of 7.8%. The results of the study showed zoxamide to be stable when stored frozen in potatoes for at least 24 months.

Table 27. Storage stability of zoxamide in potatoes fortified with zoxamide

Matrix	Storage day	Fortification mg/kg	% Remaining ^a	
			Results	Average
Potatoes	0	1.0	91, 88, 89, 82	88
		0.5	103	103
	56	0.857	105, 96, (206)	101
	149	0.058	101, 117, (159)	109
	239	0.187	128, 133, 108	123
	334	0.164	112, 103, (50)	108
	708	0.857	100, 83, (47)	92

a - % remaining=(measured concentration)/(concentration of fortification)*100

Numbers in parentheses were not used in the calculation of the averages.

The stability of metabolites RH-1452 and RH-1455 in potatoes was demonstrated by re-analysing samples from the metabolism study after 29 months of storage (Ross, 1998; DERBI 92233).

Table 28. Storage stability of RH-1452 and RH-1455 in potatoes

Metabolite	Metabolism Study, April, 1996 mg/kg parent equivalents	Radiovalidation GC/ECD Analysis, September, 1998 mg/kg parent equivalents
RH-1452	0.037	0.038 ± 0.013
RH-1455	0.069	0.065 ± 0.015

USE PATTERNS

The Meeting received labels in many countries in Europe, North America and Latin America and the Republic of Korea. It also received labels from other countries but they were not used in the review as they were either not related to the supervised trials provided or written in languages other than English, Spanish or French. Pests controlled are summarized in Table 29.

Information on registered formulations, applications methods and dosage rates of zoxamide for uses on the crops for which supervised trial data were provided is summarized in Table 30. Unless otherwise noted, each of the following GAPS are for field use and all applications are broadcast applications. On most of labels provided there are instructions about application intervals.

Table 29. Information on pests and diseases controlled by zoxamide

Crop	Pests/diseases controlled	Growth stage and season ^a
Grapes	Downy mildew of grapevine	55 to 81 (Summer)
Grapes, Table	Black knot of grapevine, Downy mildew of grapevine, Black rot of grapevine; Red fire disease of grapevine	07 to 89 (Spring-Summer)
Grapes, Wine	Black knot of grapevine, Downy mildew of grapevine, Black rot of grapevine; Red fire disease of grapevine	07 to 89 (Spring-Summer)
Vines	Downy mildew of grapevine	13 to 85 (Spring/Summer)
Cucumber	Downy mildew of cucurbits	14 to 89 (Summer)
Cucurbit	Downy mildew of cucurbits	14 to 89 (Summer)
Melons	Downy mildew of cucurbits	14 to 89 (Summer)
Pumpkin	Downy mildew of cucurbits	14 to 89 (Summer)
Squash	Downy mildew of cucurbits	14 to 89 (Summer)
Watermelon	Downy mildew of cucurbits	14 to 89 (Summer)
Tomato	Late blight of tomato; Early blight of tomato	13 to 89 (Summer)
Potato	Late blight of potato, Early blight of potato	12 to 89 (Spring-Summer-Autumn)

a - Growth stage listed using BBCH codes.

Table 30. Registered uses of zoxamide relevant to the present review

Crop	Country	Formulation		Application of zoxamide					PHI days
		Type	Conc. of zoxamide (conc. of other ingredient)	Method	Rate kg ai/ha	Volume L/ha	Spray conc. kg ai/hL	Number max	
Grapes									
Grapes	Austria	WG	83 g/kg (667 g/kg mancozeb)	Foliar		400-1000 [-1600]	0.015	4	56
Grapes	Brazil	WG	330 g/kg (330 g/kg cymoxanil)	Foliar		1000	0.010-0.012	NS ^a	7
Grapes	Brazil	WP	73 g/kg (727 g/kg mancozeb)	Foliar	0.10-0.13	600-2000		NS	7
Grapes	Canada	WG	84 g/kg zoxamide 667 g/kg mancozeb	Foliar	0.19			6	66
Grapes	Croatia	WG	83 g/kg (667 g/kg mancozeb)	Foliar	0.15-0.17			4	28
Grapes	Cyprus	WG	83 g/kg (667 g/kg mancozeb)	Foliar	0.13-0.17			1	28
Grapes	France	WG	62 g/kg (689 g/kg mancozeb)	Foliar			0.012	3	28
Grapes	France	WG	62 g/kg (689 g/kg mancozeb)	Foliar	0.12			3	28
Grapes	Germany	WG	83 g/kg (667 g/kg mancozeb)	Foliar	0.24 (base amt 0.06)	800-1600		4	56
Grapes	Hungary	WG	83 g/kg (667 g/kg mancozeb)	Foliar	0.14	800-1000		6	28
Grapes	Italy	WG	83 g/kg (667 g/kg mancozeb)	Foliar	0.14-0.17		0.014-0.017	5	28
Grapes	Luxembourg	WG	62 g/kg (689 g/kg mancozeb)	Foliar			0.012	3	28
Grapes	Luxembourg	WG	62 g/kg (689 g/kg mancozeb)	Foliar	0.12			3	28
Grapes, Wine	Moldova	WG	83 g/kg (667 g/kg mancozeb)	Foliar	0.15-0.17			3	30
Grapes, Table	Portugal	WG	83 g/kg (667 g/kg mancozeb)	Foliar		-1000 ^b	0.012-0.015	3	28
Grapes, Wine	Portugal	WG	83 g/kg (667 g/kg mancozeb)	Foliar		-1000 ^b	0.012-0.015	3	56
Grapes	Rep. Korea	WP	100 g/kg (120 g/kg cymoxanil)	Foliar		Spray enough	0.01	3	7
Grapes	Rep. Korea	WG	80 g/kg (90 g/kg famoxadone)	Foliar		Spray enough	0.008	2	14
Grapes	Romania	WG	88 g/kg (685 g/kg mancozeb)	Foliar	0.13	1000	0.013	3	28
Grapes	Slovenia	WG	83 g/kg (667 g/kg mancozeb)	Foliar	0.12-0.15	1000	0.012-0.015	4	28
Grapes	Spain	WG	83 g/kg (667 g/kg mancozeb)	Foliar	0.12-0.15			2	28
Grapes	Switzerland	WG	86 g/kg (660 g/kg mancozeb)	Foliar	0.12-0.19		0.015	3	NS
Grapes	United Kingdom	WG	83 g/kg (667 g/kg mancozeb)	Foliar		600-1600	0.015	6	56
Grapes	USA	WP	800 g/kg	Aerial	0.14-0.22	47		8	14
Grapes	USA	WP	800 g/kg	Foliar	0.14-0.22			8	14
Cucurbits									
Cucumber	Mexico	WG	330 g/kg (330 g-cymoxanil/kg)	Foliar	0.13-0.17	200-400		NS	3
Cucumber	Mexico	WG	83 g/kg (667 g-mancozeb/kg)	Foliar	0.12-0.17	200-400		6	5
Cucumber	Poland	WG	62 g/kg (689 g/kg mancozeb)	Foliar	0.12-0.15	700-800		3	4

Crop	Country	Formulation		Application of zoxamide					PHI days
		Type	Conc. of zoxamide (conc. of other ingredient)	Method	Rate kg ai/ha	Volume L/ha	Spray conc. kg ai/hL	Number max	
Cucumber	Romania	WG	88 g/kg (685 g/kg mancozeb)	Foliar	0.13	1000	0.013	3	3
Cucumber	Rep. Korea	WP	80 g/kg (600 g/kg mancozeb)	Foliar		Spray enough	0.008	3	3
Cucumber	Rep. Korea	WP	100 g/kg (120 g/kg cymoxanil)	Foliar		Spray enough	0.01	3	3
Cucumber	Rep. Korea	WG	80 g/kg (90 g/kg famoxadone)	Foliar		Spray enough	0.008	3	7
Cucumber	Rep. Korea	WP	100 g/kg (120 g/kg iprovalicarb)	Foliar		Spray enough	0.01	3	3
Cucurbit ^c	USA	WG	83 g/kg (667 g/kg mancozeb)	Aerial	0.14-0.19	19- (47- in CA)		8	5
Cucurbit ^c	USA	WG	83 g/kg (667 g/kg mancozeb)	Foliar	0.14-0.19			8	5
Melons	Honduras	WG	83 g/kg (667 g/kg mancozeb)	Foliar	0.17-0.21	200-400		NS	5
Melons	Mexico	WG	330 g/kg (330 g-cymoxanil/kg)	Foliar	0.13-0.17	200-400		NS	3
Melons	Mexico	WG	83 g/kg (667 g/kg mancozeb)	Foliar	0.12-0.17	200-400		6	5
Melon, Oriental Pickling	Rep. Korea	WG	80 g/kg (90 g/kg famoxadone)	Foliar		Spray enough	0.008	3	2
Melons	Rep. Korea	WG	80 g/kg (90 g/kg famoxadone)	Foliar		Spray enough	0.008	3	7
Pumpkin	Mexico	WG	83 g/kg (667 g/kg mancozeb)	Foliar	0.12-0.17	200-400		6	5
Squash	Mexico	WG	330 g/kg (330 g-cymoxanil/kg)	Foliar	0.13-0.17	200-400		NS	3
Squash	Mexico	WG	83 g/kg (667 g/kg mancozeb)	Foliar	0.12-0.17	200-400		6	5
Watermelon	Mexico	WG	330 g/kg (330 g-cymoxanil/kg)	Foliar	0.13-0.17	200-400		NS	3
Watermelon	Rep. Korea	WP	100 g/kg (120 g/kg cymoxanil)	Foliar		Spray enough	0.01	3	3
Tomato									
Tomato	Brazil	WG	330 g/kg (330 g/kg cymoxanil)	Foliar	0.10-0.13	650		NS	7
Tomato	Brazil	WP	73 g/kg (727 g/kg mancozeb)	Foliar	0.10-0.13	800-1200		NS	7
Tomato	Chile	WP	73 g/kg (727 g/kg mancozeb)	Foliar	0.11-0.15			NS	14
Tomato	Hungary	WG	83 g/kg (667 g/kg mancozeb)	Foliar	0.14	400-600		3	7 5 ^d
Tomato	Italy	WG	83 g/kg (667 g/kg mancozeb)	Foliar	0.14-0.17		0.014-0.017	5	3
Tomato	Mexico	WG	330 g/kg (330 g-cymoxanil/kg)	Foliar	0.13	200-400		NS	5
Tomato	Mexico	WG	83 g/kg (667 g/kg mancozeb)	Foliar	0.10-0.17	200-400		6	5
Tomato	Moldova	WG	83 g/kg (667 g/kg mancozeb)	Foliar	0.02-0.15			3	30
Tomato	Morocco	WG	83 g/kg (667 g/kg mancozeb)	Foliar	0.12		0.012	4	7
Tomato	Poland	WG	62 g/kg (689 g/kg mancozeb)	Foliar	0.12	700-800	[0.017]	5	4
Tomato	Romania	WG	88 g/kg (685 g/kg mancozeb)	Foliar	0.13	1000	0.013	3	3
Tomato	Rep. Korea	WP	100 g/kg (120 g/kg cymoxanil)	Foliar		Spray enough	0.01	4	2

Crop	Country	Formulation		Application of zoxamide					PHI days
		Type	Conc. of zoxamide (conc. of other ingredient)	Method	Rate kg ai/ha	Volume L/ha	Spray conc. kg ai/hL	Number max	
Tomato	Rep. Korea	WG	80 g/kg (90 g/kg famoxadone)	Foliar		Spray enough	0.008	3	2
Tomato	USA	WG	83 g/kg (667 g/kg mancozeb)	Aerial	0.14-0.19	19- (47- in CA)		4 ^e 8	5
Tomato	USA	WG	83 g/kg (667 g/kg mancozeb)	Foliar	0.14-0.19			4 8	5
Potato									
Potato	Argentina	WP	73 g/kg (727 g/kg mancozeb)	Aerial	0.13-0.15	Min. 20		NS	7
Potato	Argentina	WP	73 g/kg (727 g/kg mancozeb)	Foliar	0.15	400-1000		NS	7
Potato	Austria	WG	83 g/kg (667 g/kg mancozeb)	Foliar	0.15	200-600		3	7
Potato	Belgium	WG	83 g/kg (667 g/kg mancozeb)	Foliar	0.15			10	7
Potato	Brazil	WG	330 g/kg (330 g/kg cymoxanil)	Foliar	0.10-0.13	650		NS	7
Potato	Brazil	WP	73 g/kg (727 g/kg mancozeb)	Foliar	0.10-0.13	400-1000		NS	7
Potato	Canada	WG	84 g/kg zoxamide (667 g/kg mancozeb)	Aerial	0.19 or 0.14	90 or 45-90		6	3
Potato	Canada	WG	84 g/kg zoxamide (667 g/kg mancozeb)	Foliar	0.19 or 0.14			6	3
Potato	Chile	WP	73 g/kg (727 g/kg mancozeb)	Foliar	0.11-0.18			NS	7
Potato	Colombia	WP	330 g/kg (330 g/kg cymoxanil)	Foliar	0.10		0.033 in 2 hL	NS	NS
Potato	Croatia	WG	83 g/kg (667 g/kg mancozeb)	Foliar	0.15-0.17			4	7
Potato	Cyprus	WG	83 g/kg (667 g/kg mancozeb)	Foliar	0.12-0.15			3	7
Potato	Czech Republic	WG	83 g/kg (667 g/kg mancozeb)	Foliar	0.15	300-600		3	7
Potato	France	WG	83 g/kg (667 g/kg mancozeb)	Foliar	0.15			4	7
Potato	Germany	WG	83 g/kg (667 g/kg mancozeb)	Foliar	0.15	200-600		3	7
Potato	Honduras	WG	83 g/kg (667 g/kg mancozeb)	Foliar	0.12-0.15	200-400		4	14
Potato	Hungary	WG	83 g/kg (667 g/kg mancozeb)	Foliar	0.12-0.15	400-600		3	7
Potato	Ireland	WG	83 g/kg (667 g/kg mancozeb)	Foliar	0.15	200-600		10	7
Potato	Israel	WG	83 g/kg (667 g/kg mancozeb)	Foliar	0.12-0.17	250-300		NS	14
Potato	Italy	WG	83 g/kg (667 g/kg mancozeb)	Foliar	0.14-0.17		0.014-0.017	5	7
Potato	Mexico	WG	330 g/kg (330 g-cymoxanil/kg)	Foliar	0.10-0.15	200-400		NS	14
Potato	Mexico	WG	83 g/kg (667 g/kg mancozeb)	Foliar	0.10-0.15	200-400		6	7
Potato	Morocco	WG	83 g/kg (667 g/kg mancozeb)	Foliar	0.12		0.012	4	15
Potato	Netherlands	WG	83 g/kg (667 g/kg mancozeb)	Foliar	0.12-0.15			10	7
Potato	Poland	WG	62 g/kg (689 g/kg mancozeb)	Foliar	0.12	200-400		5	14
Potato	Portugal	WG	83 g/kg (667 g/kg mancozeb)	Foliar		-1000 ^b	0.012-0.015	3	7

Crop	Country	Formulation		Application of zoxamide					PHI days
		Type	Conc. of zoxamide (conc. of other ingredient)	Method	Rate kg ai/ha	Volume L/ha	Spray conc. kg ai/hL	Number max	
Potato	Romania	WG	88 g/kg (685 g/kg mancozeb)	Foliar	0.13-0.16			10	7
Potato	Slovakia	WG	83 g/kg (667 g/kg mancozeb)	Foliar	0.15	300-600		10	14
Potato	Slovenia	WG	83 g/kg (667 g/kg mancozeb)	Foliar	0.12-0.15			4	7
Potato	Rep. Korea	WP	80 g/kg (600 g/kg mancozeb)	Foliar		Spray enough	0.008	5	14
Potato	Rep. Korea	WG	80 g/kg (90 g/kg famoxadone)	Foliar		Spray enough	0.008	4	14
Potato	Spain	WG	83 g/kg (667 g/kg mancozeb)	Foliar	0.15-0.17	200-800		3	14
Potato	Sweden	WG	83 g/kg (667 g/kg mancozeb)	Foliar	0.15	200-600		10	30
Potato	Switzerland	WG	86 g/kg (660 g/kg mancozeb)	Foliar	0.15			3	21 ^f
Potato	United Kingdom	WG	83 g/kg (667 g/kg mancozeb)	Foliar	0.15	200-600		10	7
Potato	USA	WG	83 g/kg (667 g/kg mancozeb)	Aerial	0.14-0.19	19-28 (47- in CA)		6	3
Potato	USA	WG	83 g/kg (667 g/kg mancozeb)	Foliar	0.14-0.19			6	3
Potato	USA	WP	800 g/kg	Aerial	0.14-0.18	19-28 (47- in CA)		6	3
Potato	USA	WP	800 g/kg	Foliar	0.14-0.18			6	3

a - NS = Not specified on the product label.

b - When the product is applied using medium or low volume devices, the concentration must be increased so that the portion of product per hectare is the same as it is at high volume (1000 L/ha).

c - Includes cucumber, melons (cantaloupe, casaba, Crenshaw, honeydew, muskmelon), summer squash (including crookneck squash, scallop squash, straightneck squash, vegetable marrow and zucchini), and watermelon.

d - Canned

e - Maximum 4 applications west of the Rocky Mountains; and 8 applications east of the Rocky Mountains.

f - For new potatoes, 14 days.

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

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

Table 31 - 34	Grapes
Table 35 - 37	Fruiting Vegetables, Cucurbits (Cantaloupe, Cucumber and Squash)
Table 38 - 40	Tomato
Table 41 - 45	Potato

In these tables, application rates are reported for zoxamide only. Unless otherwise noted, the residue concentrations being reported are also for zoxamide. When residues were not detected (i.e., they were below the limit of detection of the method) they are shown as "ND". When residues were detected at concentrations below the LOQ, they were reported as less than the LOQ (e.g. < 0.1 mg/kg). Residues, application rates and spray concentrations have generally been rounded to two significant figures but for residues near the LOQ to one significant figure. Residue values from the trials conducted according to maximum GAP have been used for the estimation of maximum residue levels and STMRs. These results are double underlined. If a residue value at the minimum PHI from a decline trial was lower than a value from a subsequent sampling event, the higher residue value was underlined and used in subsequent calculations.

Laboratory reports included method validation with batch recoveries at residue levels similar to those occurring in samples from the supervised trials. Dates of analyses or duration of residue sample storage were also provided. Field reports provided data on the sprayers used and their calibration, plot size, residue sample size and sampling date. Although trials included control plots, no control data are recorded in the tables except where residues in control samples exceeded the LOQ. Residue data are recorded unadjusted for percent recovery.

Grapes

Supervised trials in Europe

Relevant supervised residue trials were conducted over multiple growing seasons in both the Northern and Southern zones of Europe: 12 trials in Germany in 1996, 97, 98 and 99; in 21 trials in Northern France in 1996, 97, 98 and 99; 15 trials in Southern France in 19, 97 and 98; 25 trials in Greece, Italy and Spain in 1996, 97 and 98. Most of the studies conducted used the WG formulation containing 66.7% mancozeb and 8.3% zoxamide and the SC formulation containing 240g/L zoxamide. A WP formulation also containing 66.7% mancozeb and 8.3% zoxamide was introduced into trials from 1998 in parallel with WG trials. No differences were noted between residues arising from the different formulation types.

The maximum labelled GAP for grapes in countries of the European Union, except Germany and the UK, is based on multiple applications (2 – 6 sprays) of zoxamide at 0.17 kg ai/ha with a PHI of 28 or 56 days depending on the country. Most of the supervised grape trials, with the exception of the German trials, were undertaken with 10 applications of zoxamide at 0.15 kg ai/ha giving a total estimated application of 1.5 kg of active substance per year in support of a PHI of 28 days.

The GAP for grapes in Germany and the UK is also based on multiple applications (4 and 6 respectively) with a PHI of 56 days. The use rate is 0.015 kg/hL with the intended kg ai/ha varying with water volume according to the growth stage of the crop at the time of applications. The supervised trials conducted in Germany were performed with multiple applications (6) of 0.015 kg/hL made over the growing season giving a total of approximately 0.6 kg as/ha/year in support of a PHI of 56 days. A typical application scenario under the German/UK GAP would be one application at 400L/ha (0.06 kg ai/ha), two at 800 L/ha (2 × 120 g ai/ha), two at 1200 L/ha (2 × 180 g ai/ha) and one at 1600 L/ha (1 × 240 g ai/ha).

The application timings were designed to cover the full seasonal range of infection from grape downy mildew (*Plasmopara viticola*). In all trials, ripe berries were present at the last application, thus giving a worst-case potential for residues in the RAC. The registrations gained in EU Member States were for less than 10 sprays due to other registration limitations (typically zoxamide resistance management strategies). However, the majority of the residue found at harvest is derived from the contribution of the last 3 – 4 sprays of a spray program. Therefore, it is unlikely that the residues from the 6 spray or 10 spray trials, which included applications before flowering, resulted in different residues at harvest than a program based on 3 – 4 sprays.

Residues of zoxamide in grapes were determined using method TR34-96-128 (Guo, 1996; DERBI 91475). The method was validated prior to and concurrent with analyses by spiking control samples with zoxamide at fortification levels ranging from 0.05 to 5.0 mg/kg. The limit of quantification (LOQ) was 0.01 mg/kg whilst the limit of detection was 0.003 mg/kg.

Table 31. Zoxamide residues in grapes from supervised trials in Germany, Northern and Southern France, Greece, Italy and Spain

Grapes country, year (variety)	Application					PHI Days	Residues, mg/kg	Reference Trial number
	Form	kg ai/ha	kg ai/hL	water, L/ha	no.			
GAP, Germany	WG	0.24 max		800-1600	4	56		
Walluf, Germany, 1998 (Muller-Thurgau)	WG	0.06-0.30	0.015	400-2000	6	0	0.71	Wais,1999, DERBI 221387 A/GE/F/98/86
						28	0.41	
						56	0.41	

Grapes country, (variety)	year	Application				no.	PHI Days	Residues, mg/kg	Reference Trial number
		Form	kg ai/ha	kg ai/hL	water, L/ha				
Walluf, Germany, 1998 (Muller-Thurgau)		WP	0.06-0.30	0.015	400-2000	6	0 28 56	0.71 0.53 <u>0.39</u>	Wais,1999, DERBI 221387 A/GE/F/98/86
Bensheim, Germany, 1998 (Riesling)		WG	0.06-0.30	0.015	400-2000	6	0 28 56	0.43 0.62 <u>0.72</u>	Wais,1999, DERBI 221387 A/GE/F/98/142
Alsbachtal, Germany, 1998 (Riesling)		WG	0.06-0.30	0.015	400-2000	6	0 28 56	1.43 0.52 <u>0.59</u>	Wais,1999, DERBI 221387 A/GE/F/98/141
Walluf, Germany,1998 (Riesling)		WP	0.06-0.30	0.015	400-2000	6	0 28 56	0.73 0.41 <u>0.45</u>	Wais,1999, DERBI 221387 A/GE/F/98/87
Walluf, Germany,1998 (Riesling)		WG	0.06-0.30	0.015	400-2000	6	0 28 56	0.39 0.40 <u>0.60</u>	Wais,1999, DERBI 221387 A/GE/F/98/87
Zwingenberg, Germany, 1996 (Late burgundy)		WG	0.15-0.30	0.015	300-600	6	0 21 42 56	2.53 0.69 0.49 <u>0.41</u>	Wais,1999, DERBI 221867 A/GE/F/96/37
Zwingenberg, Germany, 1996 (Riesling)		WG	0.06-0.30	0.015	400-1600	6	0 21 42 56	3.54 0.43 0.53 <u>0.55</u>	Wais,1999, DERBI 221867 A/GE/F/96/36
Zwingenberg, Germany, 1997 (Riesling)		WG	0.06-0.30	0.015	400-2000	6	0 14 28 43 56	0.75 0.67 0.93 0.58 <u>0.34</u>	Wais,1999, DERBI 221863 A/GE/F/97/1
Zwingenberg, Germany, 1997 (Riesling)		WG	0.15-0.30	0.015	300-600	6	0 28 56	0.98 0.60 <u>0.38</u>	Wais,1999, DERBI 221863 A/GE/F/97/2
Walluf, Germany, 1997 (Burgunder)		WG	0.06-0.30	0.015	400-2000	6	0 14 28 43 56	0.66 0.50 0.89 < 0.01 <u>0.66</u>	Wais,1999, DERBI 221863 A/GE/F/97/3
Kaiserstuhl, Germany, 1999 (Silvaner)		WG	0.06-0.30	0.015	400-1600	6	0 56	0.74 <u>0.49</u>	Wais,2000, DERBI 221384 A/GE/F/99/65
<i>GAP, France</i>		<i>WG</i>	<i>0.12</i>			<i>3</i>	<i>28</i>		
Champagne, France, 1996 (Meunier)		SC	0.15	0.043	350	10	0 7 14 21 28	1.02 1.04 0.70 0.79 <u>0.67</u>	Grolleau,1999, DERBI 220177 EA960110FR01
Champagne, France, 1996 (Meunier)		WG	0.15	0.043	350	10	0 7 14 21 28	1.83 1.72 1.36 1.66 <u>1.31</u>	Grolleau,1999, DERBI 220177 EA960110FR01
Vouvray, France, 1996 (Chenin)		SC	0.15	0.043	350	10	28	<u>0.51</u>	Grolleau,1999, DERBI 220177 EA960110FR02
Vouvray, France, 1996 (Chenin)		WG	0.15	0.043	350	10	28	<u>0.45</u>	Grolleau,1999, DERBI 220177 EA960110FR02
Champagne, France, 1997 (Pinot Noir)		SC	0.15	0.03	500	10	0 28 35	0.76 0.43 <u>0.47</u>	Grolleau,1999, DERBI 146039 EA970130R01
Champagne, France, 1997 (Pinot noir)		WG	0.15	0.03	500	10	0 28 35	0.54 0.42 <u>0.48</u>	Grolleau,1999, DERBI 146039 EA970130FR01

Grapes country, year (variety)	Application					PHI Days	Residues, mg/kg	Reference Trial number
	Form	kg ai/ha	kg ai/hL	water, L/ha	no.			
Champagne, France, 1997 (Chardonnay)	SC	0.15	0.03	500	10	0 14 21 28 35	1.01 0.44 0.51 0.46 <u>0.55</u>	Grolleau,1999, DERBI 146039 EA970130FR02
Champagne, France, 1997 (Chardonnay)	WG	0.15	0.03	500	10	0 14 21 28 35	0.72 0.46 0.38 0.42 <u>0.50</u>	Grolleau,1999, DERBI 146039 EA970130FR02
Pays de Loire, France, 1997 (Chenin)	SC	0.15	0.03	500	10	0 28 35	0.86 0.42 <u>0.50</u>	Grolleau,1999, DERBI 146039 EA970130FR03
Pays de Loire, France, 1997 (Chenin)	WG	0.15	0.03	500	10	0 28 35	0.43 <u>0.56</u> 0.48	Grolleau,1999, DERBI 146039 EA970130FR03
Vouvray, France, 1997 (Cabernet franc)	SC	0.15	0.03	500	10	0 14 21 28 35	0.59 0.67 0.60 0.42 <u>0.81</u>	Grolleau,1999, DERBI 146039 EA970130FR04
Vouvray, France, 1997 (Cabernet franc)	WG	0.15	0.03	500	10	0 14 21 28 35	0.76 0.61 0.48 <u>0.35</u> 0.30	Grolleau,1999, DERBI 146039 EA970130FR04
Champagne, France, 1998 (Pinot noir)	WG	0.15	0.03	500	10	0 21 28	0.84 0.95 <u>1.55</u>	Grolleau,1999, DERBI 146038 EA980117FR01
Champagne, France, 1998 (Pinot noir)	WG	0.125	0.025	500	10	0 21 28	0.86 1.17 <u>0.77</u>	Grolleau,1999, DERBI 146038 EA980117FR01
Champagne, France, 1998 (Chardonnay)	WG	0.15	0.03	500	10	0 21 28	1.02 0.89 <u>0.88</u>	Grolleau,1999, DERBI 146038 EA980117FR02
Champagne, France, 1998 (Chardonnay)	WG	0.125	0.025	500	10	0 21 28	1.22 0.74 <u>0.77</u>	Grolleau,1999, DERBI 146038 EA980117FR02
Pays de Loire, France, 1998 (Chenin)	WG	0.15	0.03	500	10	0 21 28	0.16 0.16 <u>0.17</u>	Grolleau,1999, DERBI 146038 EA980117FR03
Pays de Loire, France, 1998 (Chenin)	WG	0.125	0.025	500	10	0 21 28	0.15 0.11 <u>0.09</u>	Grolleau,1999, DERBI 146038 EA980117FR03
Pays de Loire, France, 1998 (Grolleau)	WG	0.15	0.03	500	10	0 21 28	0.41 0.59 <u>0.33</u>	Grolleau,1999, DERBI 146038 EA980117FR04
Pays de Loire, France, 1998 (Grolleau)	WG	0.125	0.025	500	10	0 21 28	0.26 0.19 <u>0.19</u>	Grolleau,1999, DERBI 146038 EA980117FR04
Noget L'Abbesse, France, 1999 (Chardonnay)	WG	0.125	0.025	500	10	0 28	0.27 <u>0.19</u>	Grolleau,2000, DERBI 221383 EA990175FR01
<i>GAP, France</i>	<i>WG</i>	<i>0.12</i>			3	28		
Aquitaine, France, 1996 (Cabernet Sauvignon)	SC	0.15	0.043	350	10	0 7 14 21 28	2.66 2.23 1.44 1.65 <u>1.53</u>	Grolleau, 1999 DERBI 220177 EA960110FR03

Grapes country, (variety)	year	Application				no.	PHI Days	Residues, mg/kg	Reference Trial number
		Form	kg ai/ha	kg ai/hL	water, L/ha				
Aquitaine, France, 1996 (Cabernet Sauvignon)		WG	0.15	0.043	350	10	0 7 14 21 28	4.95 3.61 3.41 3.49 <u>2.84</u>	Grolleau, 1999 DERBI 220177 EA960110FR03
Provence, France, 1996 (Clairette blanche)		SC	0.15	0.043	350	10	28	<u>1.11</u>	Grolleau, 1999 DERBI 220177 EA960110FR04
Provence, France, 1996 (Clairette blanche)		WG	0.15	0.043	350	10	28	<u>1.07</u>	Grolleau, 1999 DERBI 220177 EA960110FR04
Aquitaine, France, 1997 (Merlot)		WG	0.125	0.025	350	10	0 28 35	0.68 0.33 <u>0.46</u>	Grolleau, 1999 DERBI 146039 EA970130FR05
Aquitaine, France, 1997 (Merlot)		WG	0.125	0.025	500	6	28 35 55	0.29 <u>0.61</u> 0.42	Grolleau, 1999 DERBI 146039 EA970130FR05
Aquitaine, France, 1997 (Sauvignon)		WG	0.125	0.025	500	10	0 14 21 28 35	0.66 0.40 0.17 0.33 <u>0.42</u>	Grolleau, 1999 DERBI 146039 EA970130FR06
Provence, France, 1997 (Grenache)		WG	0.125	0.025	500	10	0 28 35	0.24 <u>0.63</u> 0.21	Grolleau, 1999 DERBI 146039 EA970130FR07
Provence, France, 1997 (Ugni blanc)		WG	0.125	0.025	500	10	0 14 21 28 35	1.03 0.62 0.61 <u>0.54</u> 0.46	Grolleau, 1999 DERBI 146039 EA970130FR08
Aquitaine, France, 1998 (Merlot)		WG	0.125	0.025	500	10	0 21 28	0.16 0.19 <u>0.21</u>	Grolleau, 1999 DERBI 146038 EA980117FR05
Aquitaine, France, 1998 (Merlot)		WG	0.125	0.025	500	6	28 35 56	<u>0.42</u> 0.17 0.14	Grolleau, 1999 DERBI 146038 EA980117FR05
Aquitaine, France, 1998 (Semillion)		WG	0.125	0.025	500	10	0 21 28	0.20 0.12 <u>0.21</u>	Grolleau, 1999 DERBI 146038 EA980117FR06
Provence, France, 1998 (Ugni blanc)		WG	0.125	0.025	500	10	0 21 28	0.59 0.63 <u>0.49</u>	Grolleau, 1999 DERBI 146038 EA980117FR07
Provence, France, 1998 (Cinsaut)		WG	0.125	0.025	500	10	0 21 28	0.47 0.44 <u>0.58</u>	Grolleau, 1999 DERBI 146038 EA980117FR08
Aquitaine, France, 1999 (Cabernet frac)		WG	0.125	0.0357	350	10	0 28	0.49 <u>0.33</u>	Grolleau, 2000 DERBI 221382 EA990176FR01
<i>GAP, Italy</i>		<i>WG</i>	<i>0.14-0.17</i>	<i>0.014- 0.017</i>		<i>NS</i>	28		
Trevison, Italy, 1996 (Cabernet sauvignon)		SC	0.15	0.015	1000	10	0 7 14 21 29	0.41 0.28 0.40 0.49 <u>0.66</u>	Wais, 1999 DERBI 221861 A/IT/F/96/38
Trevison, Italy, 1996 (Cabernet sauvignon)		WG	0.15	0.015	1000	10	0 7 14 20 28	0.53 0.37 0.54 0.52 <u>0.48</u>	Wais, 1999 DERBI 221861 A/IT/F/96/38

Grapes country, year (variety)	Application					PHI Days	Residues, mg/kg	Reference Trial number
	Form	kg ai/ha	kg ai/hL	water, L/ha	no.			
Isola d'Asti, Italy, 1996 (Sauvignon blanc)	SC	0.15	0.015	1000	10	28	<u>0.48</u>	Wais, 1999 DERBI 221861 A/IT/F/96/39
Isola d'Asti, Italy, 1996 (Sauvignon blanc)	WG	0.15	0.015	1000	10	28	<u>0.59</u>	Wais, 1999 DERBI 221861 A/IT/F/96/39
Castigliano, Italy, 1996 (Early Cardinal)	WG	0.15	0.03	500	10	28	<u>0.54</u>	Wais, 1999 DERBI 221385 A/IT/F/96/40
Agliano, Italy, 1996 (Italia)	SC	0.15	0.03	500	10	0 7 14 21 28	2.15 0.86 0.94 0.84 <u>1.37</u>	Wais, 1999 DERBI 221385 A/IT/F/96/41
Agliano, Italy, 1996 (Italia)	WG	0.15	0.03	500	10	0 7 14 21 28	1.08 1.08 1.51 1.59 <u>1.56</u>	Wais, 1999 DERBI 221385 A/IT/F/96/41
Carpento, Italy, 1997 (Barbera)	SC	0.15	0.015	1000	10	0 28 35	0.45 <u>0.82</u> 0.52	Wais, 1999 DERBI 221864 A/IT/F/97/6
Carpento, Italy, 1997 (Barbera)	WG	0.15	0.015	1000	10	0 28 35	0.91 <u>0.65</u> 0.48	Wais, 1999 DERBI 221864 A/IT/F/97/6
Isola d'Asti, Italy, 1997 (Sauvignon)	SC	0.15	0.015	1000	10	0 14 21 28 35	0.45 0.31 0.24 <u>0.30</u> 0.20	Wais, 1999 DERBI 221864 A/IT/F/97/7
Isola d'Asti, Italy, 1997 (Sauvignon)	WG	0.15	0.015	1000	10	0 14 21 28 35	0.54 0.53 0.35 0.26 <u>0.33</u>	Wais, 1999 DERBI 221864 A/IT/F/97/7
Isola d'Asti, Italy, 1998 (Sauvignon)	WG	0.15	0.015	1000	10	0 21 28	0.40 0.55 <u>0.81</u>	Wais, 1999 DERBI 221390 A/IT/F/98/145
Isola d'Asti, Italy, 1998 (Sauvignon)	WP	0.15	0.015	1000	10	0 21 28	0.65 0.46 <u>0.29</u>	Wais, 1999 DERBI 221390 A/IT/F/98/145
Isola d'Asti, Italy, 1998 (Barbera)	WG	0.15	0.015	1000	10	0 21 28	0.41 0.32 <u>0.24</u>	Wais, 1999 DERBI 221390 A/IT/F/98/146
Isola d'Asti, Italy, 1998 (Barbera)	WP	0.15	0.015	1000	10	0 21 28	0.52 0.42 <u>0.28</u>	Wais, 1999 DERBI 221390 A/IT/F/98/146
Alhama Murcia, Spain, 1996 (Italia)	de SC	0.15	0.015	1000	10	0 7 14 21 28	2.34 2.68 1.85 1.03 <u>1.42</u>	Wais, 1999 DERBI 221862 A/SP/F/96/42
Alhama Murcia, Spain, 1996 (Italia)	de WG	0.15	0.015	1000	10	0 7 14 21 28	3.49 4.07 2.40 2.06 <u>1.92</u>	Wais, 1999 DERBI 221862 A/SP/F/96/42
Abaran, Spain, 1996 (Napoleon)	SC	0.15	0.015	1000	10	28	<u>1.21</u>	Wais, 1999 DERBI 221862 A/SP/F/96/43

Grapes country, (variety)	year	Application				no.	PHI Days	Residues, mg/kg	Reference Trial number
		Form	kg ai/ha	kg ai/hL	water, L/ha				
Abaran, Spain, 1996 (Napoleon)		WG	0.15	0.015	1000	10	28	<u>1.17</u>	Wais, 1999 DERBI 221862 A/SP/F/96/43
La Pobla del Duc, Spain, 1998 (Tempranillo)		WG	0.15	0.03	500	10	0 21 28	0.81 0.76 <u>0.53</u>	Wais, 1999 DERBI 221396 A/SP/F/98/143
L'Arbocar, Spain, 1998 (Macabeo)		WG	0.15	0.03	500	10	0 21 28	0.35 0.17 <u>0.36</u>	Wais, 1999 DERBI 221396 A/SP/F/98/144
Orfani, Greece, 1997 (Soultanina)		SC	0.15	0.015	1000	10	0 14 21 28 35	0.49 0.76 0.27 0.31 <u>0.32</u>	Wais, 1999 DERBI 221866 A/GR/F/97/11
Orfani, Greece, 1997 (Soultanina)		WG	0.15	0.015	1000	10	0 14 21 28 35	0.33 0.21 0.35 0.21 <u>0.27</u>	Wais, 1999 DERBI 221866 A/GR/F/97/11
Kardia, Greece, 1997 (Rosaki)		SC	0.15	0.015	1000	10	0 28 35	1.96 <u>0.64</u> 0.55	Wais, 1999 DERBI 221866 A/GR/F/97/12
Kardia, Greece, 1997 (Rosaki)		WG	0.15	0.015	1000	10	0 28 35	0.31 <u>0.34</u> 0.18	Wais, 1999 DERBI 221866 A/GR/F/97/12

The concentration of RH-150721 was also determined in grapes (Wolf, 2001; DERBI 221399). The results of the analyses are presented in the table below.

Table 32. Determination of RH-150721 in samples from selected supervised field trials in France and Germany

Country/Year	PHI (Days)	RH-150721 (mg/kg)	Zoxamide (mg/kg)	Zoxamide/150721	RH-	Reference Trial No.
France N 1999	28	0.074	0.19	2.6		Groleau, 2000, DERBI 221383 EA990175 FR 01
France S 1999	28	0.086	0.33	3.8		Groleau, 2000, DERBI 221382 EA990176 FR01
France 1999	28	0.062	0.19	3.1		Groleau, 2000, DERBI 221381 EA990177 FR01
France 1999	28	0.185	0.27	1.6		Groleau, 2000, DERBI 221381 EA990177 FR02
Italy 1999	28	0.090	1.32	14.6		Wais, 2001, DERBI 221398 A/IT/F/99/66
Germany 1999	56	0.055	0.49	8.9		Wais, 2001, DERBI 221384 A/GE/F/99/65
Germany 1997	57	< 0.03	0.84	28		Wais, 1999, DERBI 221863 A/GE/F/97/1
Germany ^a 1997	57	< 0.03	0.6	20		Wais, 1999, DERBI 221863 A/GE/F/97/2
Germany 1997	57	< 0.03	0.66	22		Wais, 1999, DERBI 221863 A/GE/F/97/3

Country/ Year	PHI (Days)	RH-150721 (mg/kg)	Zoxamide (mg/kg)	Zoxamide/ 150721	RH-	Reference Trial No.
Germany ^a 1997	57	0.03	0.35	11.66		Wais, 1999, DERBI 221863 A/GE/F/97/4

a - Crops were treated with a spray concentration of 45 kg ai/hL instead of 15 kg ai/hL as stated in the proposed GAP

Supervised trials in North and South America

A total of four supervised field trials on grapes were conducted in Canada (Vaughn, 2001; DERBI 110110). Each of the four trials contained one treated plot that received 8 foliar-directed applications of a wettable powder formulation containing 800 g/kg zoxamide at the nominal rates of 0.23 kg ai/ha per application. Two of the four trials included an additional plot treated with six applications of a dry flowable formulation containing 69% mancozeb and 8.3% zoxamide. The nominal application rate was 0.23 kg ai/ha zoxamide. All applications were made using airblast sprayers at 7 – 10 day intervals. For each sample a minimum of 1 kg of grapes were collected from 12 locations within the plot. The maximum period of time from harvest to analysis was 94 days. Analyses of the RAC samples were performed using method TR 34-98-150 (Burdge, 1998; DERBI 92154). The LOQ for this method was 0.01 mg/kg. The average percent recovery from the method evaluation performed and the concurrent recoveries was 94% with a standard deviation of 11%.

A total of 12 supervised field trials on grapes were conducted in 4 major grape producing states in the USA over 2 years (Graves, 1998; DERBI 92215).

Twenty-four supervised trials were conducted in Brazil using either a WP formulation containing approximately 73 g ai/kg zoxamide and 727 g ai/kg of mancozeb or a WG formulation containing approximately 330 g ai/kg zoxamide and 330 g ai/kg of cymoxanil.

Table 33. Zoxamide residues in grapes from supervised trials in the Canada, and USA, and Brazil

Grapes country, year (variety)	Application					PHI days	Residues, mg/kg	Reference Trial number
	Form	kg ai/ha	kg ai/hL	water, L/ha	no.			
<i>GAP, Canada</i>	<i>WG</i>	<i>0.19</i>			<i>6</i>	<i>66</i>		
<i>GAP, USA</i>	<i>WP</i>	<i>0.14-0.22</i>			<i>8</i>	<i>14</i>		
Ontario, Canada, 1999 (Vidal)	WP	0.23		885	8	14	<u>1.52</u>	Vaughn, 2000, DERBI 110110 99RHC15-A
Ontario, Canada, 1999 (Vidal)	DF	0.23		888	6	30	1.24	Vaughn, 2000, DERBI 110110 99RHC15-A
Ontario, Canada, 1999 (SV 23512)	WP	0.23		902	8	14	<u>1.12</u>	Vaughn, 2000, DERBI 110110 99RHC15-B
Ontario, Canada, 1999 (Niagara)	WP	0.23		896	8	14	<u>1.46</u>	Vaughn, 2000, DERBI 110110 99RHC15-C
Ontario, Canada, 1999 (Niagara)	DF	0.23		896	6	30	0.70	Vaughn, 2000, DERBI 110110 99RHC15-C
Ontario, Canada, 1999 (Concord)	WP	0.23		901	8	1 3 7 14 18	2.68 3.54 2.92 1.55 <u>1.69</u>	Vaughn, 2000, DERBI 110110 99RHC15-D
<i>GAP, USA</i>	<i>WP</i>	<i>0.14-0.22</i>			<i>8</i>	<i>14</i>		
Pennsylvania, USA, 1996 (Concord)	WP	0.14		881	10	0 7 14 21	1.87 1.27 1.33 1.84	Graves, 1998, DERBI 92215 21696201

Grapes country, year (variety)	Application					PHI days	Residues, mg/kg	Reference Trial number
	Form	kg ai/ha	kg ai/hL	water, L/ha	no.			
Pennsylvania, USA, 1996 (Concord)	WP	0.28		881	10	0 7 14 21	5.37 3.55 3.66 4.34	Graves, 1998, DERBI 92215 21696201
California, USA, 1996 (Carignane)	WP	0.14		1097	10	14	0.76	Graves, 1998, DERBI 92215 21696202
California, USA, 1996 (Carignane)	WP	0.28		1097	10	14	<u>1.61</u>	Graves, 1998, DERBI 92215 21696202
California, USA, 1996 (French colombard)	WP	0.14		941	10	13	0.60	Graves, 1998, DERBI 92215 21696203
California, USA, 1996 (French colombard)	WP	0.28		941	10	13	<u>1.65</u>	Graves, 1998, DERBI 92215 21696203
California, USA, 1996 (Thompson seedless)	WP	0.14		513	10	14	0.27	Graves, 1998, DERBI 92215 21696204
California, USA, 1996 (Thompson seedless)	WP	0.28		513	10	14	<u>0.52</u>	Graves, 1998, DERBI 92215 21696204
California, USA, 1996 (Flame seedless)	WP	0.14		560	10	14	0.12	Graves, 1998, DERBI 92215 21696205
California, USA, 1996 (Flame seedless)	WP	0.28		560	10	14	<u>0.22</u>	Graves, 1998, DERBI 92215 21696205
California, USA, 1996 (Crimson seedless)	WP	0.14		480	10	0 7 14 21	0.35 0.26 0.21 0.16	Graves, 1998, DERBI 92215 21696206
California, USA, 1996 (Crimson seedless)	WP	0.28		480	10	0 7 14 21	0.80 1.09 <u>0.46</u> 0.41	Graves, 1998, DERBI 92215 21696206
Idaho, USA, 1996 (Concord)	WP	0.14		937	10	14	0.44	Graves, 1998, DERBI 92215 21696207
Idaho, USA, 1996 (Concord)	WP	0.28		937	10	14	<u>0.83</u>	Graves, 1998, DERBI 92215 21696207
Pennsylvania, USA, 1997 (Concord)	WP	0.225		809	10	14	<u>1.08</u>	Graves, 1998, DERBI 92215 21697-101
Pennsylvania, USA, 1997 (Concord)	WP	0.45		819	10	14	2.66	Graves, 1998, DERBI 92215 21697-101
Pennsylvania, USA, 1997 (Concord)	SC	0.225		815	10	14	<u>1.18</u>	Graves, 1998, DERBI 92215 21697-101
California, USA, 1997 (Crimson seedless)	WP	0.225		560	10	14	<u>0.34</u>	Graves, 1998, DERBI 92215 21697-102
California, USA, 1997 (Crimson seedless)	WP	0.45		559	10	14	1.19	Graves, 1998, DERBI 92215 21697-102

Grapes country, (variety)	year	Application				no.	PHI days	Residues, mg/kg	Reference Trial number
		Form	kg ai/ha	kg ai/hL	water, L/ha				
California, USA, 1997 (Crimson seedless)		SC	0.225		557	10	14	<u>0.49</u>	Graves, 1998, DERBI 92215 21697-102
California, USA, 1997 (Thompson seedless)		WP	0.225		552	10	14	<u>0.31</u>	Graves, 1998, DERBI 92215 21697-103
California, USA, 1997 (Thompson seedless)		WP	0.45		554	10	14	1.43	Graves, 1998, DERBI 92215 21697-103
California, USA, 1997 (Thompson seedless)		SC	0.225		553	10	14	<u>0.42</u>	Graves, 1998, DERBI 92215 21697-103
California, USA, 1997 (Thompson seedless)		WP	0.225		954	10	14	<u>0.91</u>	Graves, 1998, DERBI 92215 21697-104
California, USA, 1997 (Thompson seedless)		WP	0.45		949	10	14	4.10	Graves, 1998, DERBI 92215 21697-104
California, USA, 1997 (Thompson seedless)		SC	0.225		954	10	14	<u>0.61</u>	Graves, 1998, DERBI 92215 21697-104
Washington, USA, 1997 (White riesling)		WP	0.225		696	10	14	<u>0.34</u>	Graves, 1998, DERBI 92215 21697-105
Washington, USA, 1997 (White riesling)		WP	0.45		695	10	14	1.6	Graves, 1998, DERBI 92215 21697-105
Washington, USA, 1997 (White riesling)		SC	0.225		696	10	14	<u>0.66</u>	Graves, 1998, DERBI 92215 21697-105
<i>GAP, Brazil</i>		<i>WP</i>	<i>0.10-0.13</i>		<i>600- 2000</i>	<i>NS</i>	<i>7</i>		
Porto Feliz (SP), Brazil, 2002 (Niagra)		WG		0.023	1000	3	7	0.05	Pinheiro, 2003, DERBI 134801 A
Porto Feliz (SP), Brazil, 2002 (Niagra)		WG		0.046	1000	3	7	0.12	Pinheiro, 2003, DERBI 134801 B
Indaiatuba (SP), Brazil, 1999 (Niagra Rosada)		WG	0.13		1000	6	7	<u>0.15</u>	Tornisielo, 2000, DERBI 224088 33199037
Indaiatuba (SP), Brazil, 1999 (Niagra Rosada)		WG	0.27		1000	6	7	1.15	Tornisielo, 2000, DERBI 224088 33199037
Indaiatuba (SP), Brazil, 1999 (Niagra Rosada)		WG	0.13		1000	6	7	<u>0.14</u>	Tornisielo, 2000, DERBI 224089 33100025
Indaiatuba (SP), Brazil, 1999 (Niagra Rosada)		WG	0.27		1000	6	7	0.31	Tornisielo, 2000, DERBI 224089 33100025
Monte Mor(SP), Brazil, 2002 (Niagra)		WP	0.14		1000	8	7	<u>0.36</u> , 0.33	Pinheiro, 2002, DERBI 103349

Grapes country, year (variety)	Application					PHI days	Residues, mg/kg	Reference Trial number
	Form	kg ai/ha	kg ai/hL	water, L/ha	no.			
Monte Mor(SP), Brazil, 2002 (Niagra)	WP	0.27		1000	8	7	1.05, 1.04	Pinheiro, 2002, DERBI 103349
Porto Feliz (SP), Brazil, 2002 (Niagra)	WP	0.14		1000	4	7	0.04, <u>0.07</u>	Pinheiro, 2003, DERBI 134806
Porto Feliz (SP), Brazil, 2002 (Niagra)	WP	0.14		1000	6	7	0.15, <u>0.16</u>	Pinheiro, 2003, DERBI 134806
Porto Feliz (SP), Brazil, 2002 (Niagra)	WP	0.27		1000	4	7	0.12, 0.15	Pinheiro, 2003, DERBI 134806
Porto Feliz (SP), Brazil, 2002 (Niagra)	WP	0.27		1000	6	7	0.43, 0.42	Pinheiro, 2003, DERBI 134806
Porto Feliz (SP), Brazil, 1998 (Niagra)	WP	0.14		1000	8	7	<u>0.08</u>	Tornisielo, 1999, DERBI 221882 BR98F03A
Porto Feliz (SP), Brazil, 1998 (Niagra)	WP	0.27		1000	8	7	0.28	Tornisielo, 1999, DERBI 221882 BR98F03A
Jundiai City (SP), Brazil, 1998 (Niagra)	WP	0.14		1000	8	0 3 7 10 14	0.28 0.16 <u>0.14</u> 0.09 0.07	Tornisielo, 1999, DERBI 221883 BR98F03
Jundiai City (SP), Brazil, 1998 (Niagra)	WP	0.27		1000	8	7	0.24	Tornisielo, 1999, DERBI 221883 BR98F03

Supervised trials in the Republic of Korea

Five trials were conducted using a WP formulation containing 10% zoxamide and 12% cymoxanil.

Table 34. Zoxamide residues in grapes from supervised trials in the Republic of Korea

Grapes Country, year (variety)	Application					PHI days	Residues, mg/kg	Reference
	Form	kg ai/ha	kg ai/hL	water, L/ha	no.			
<i>GAP, Rep. Korea</i>	WP		0.01	<i>Spray enough</i>	3	7		
Rep. Korea, 2000 (Black Olympia)	WP	0.4	0.01	4000	3	14 14 14	0.024 0.032 0.026	DERBI 239921
Rep. Korea, 2000 (Black Olympia)	WP	0.4	0.01	4000	3	7 7 7	<u>0.045</u> 0.042 0.040	DERBI 239921
Rep. Korea, 2000 (Black Olympia)	WP	0.4	0.01	4000	4	14 14 14	0.036 0.038 0.035	DERBI 239921
Rep. Korea, 2000 (Black Olympia)	WP	0.4	0.01	4000	4	7 7 7	0.053 0.056 <u>0.060</u>	DERBI 239921
Rep. Korea, 2000 (Black Olympia)	WP	0.4	0.01	4000	5	7 7 7	0.067 <u>0.081</u> 0.071	DERBI 239921

*Fruiting Vegetables, Cucurbits**Supervised trials in Europe (cucumber)*

Nine indoor trials were conducted in Europe using either a WG formulation containing 67% mancozeb and 8% zoxamide or a SC formulation containing 240 g/L.

Table 35. Zoxamide residues in cucumber from supervised trials in France and Spain

Cucumber country, year (variety)	Application					PHI days	Residues, mg/kg	Reference Trial number
	Form	kg ai/ha	kg ai/hL zox	water, L/ha	no.			
GAP, Poland	WG	0.12-0.15		700-800	3	4		
Handschuheim, N France, 1999, (Danora)	SC	0.15	0.030	500	5	0 1 3 7 9	0.04 0.05 <u>0.06</u> 0.04 0.05	Perny, 2001 DERBI 224320 9063 AN1
Handschuheim, N France, 1999, (Danora)	WG	0.15	0.030	500	5	0 1 3 7 9	0.37 0.33 <u>0.48</u> 0.43 0.32	Perny, 2001 DERBI 224321 9063 AN1
Ploudalmezeau, N France, 1999, (Dasher)	WG	0.15	0.015	500	5	0 1 3 7 10	0.04 0.07 <u>0.06</u> 0.03 0.03	Perny, 2001, DERBI 224321 9063 OB1
Ploudalmezeau, N France, 1999, (Arizona)	WG	0.15	0.020	750	5	0 1 3 7 10	0.03 0.02 <u>0.03</u> 0.02 0.01	Perny, 2001, DERBI 224321 9063 OB2
Daux, S France, 1999 (Gardon)	SC	0.30	0.015	2000	5	0 1 3 7 10	0.04 0.04 <u>0.01</u> < 0.01 < 0.01	Perny, 2001, DERBI 224789 9063 TL1
Daux, S France, 1999 (Danora)	WG	0.30	0.015	2000	5	0 1 3 7 10	0.07 0.06 <u>0.04</u> < 0.01 < 0.01	Perny, 2001, DERBI 224790 9063 TL1
El Ejido, Spain, 1999 (Stela)	SC	0.15	0.015	400	5	0 1 3 7 11	0.15 0.13 <u>0.25</u> 0.13 0.21	Wais, 2000, DERBI 224329 A/SP/F/99/75
El Ejido, Spain, 1999 (Stela)	WG	0.15	0.015	400	5	0 1 3 7 11	0.26 0.49 0.18 <u>0.44</u> 0.23	Wais, 2001, DERBI 238367 A/SP/F/99/75
Balanegra, Spain, 1999/2000 (Bronex)	WG	0.15	0.015	400	5	0 1 3 7 10	0.30 0.22 0.29 <u>0.45</u> 0.06	Wais, 2001, DERBI 238367 A/SP/F/99/76

Supervised trials in the USA

A total of 17 supervised field trials on cucurbits (6 cantaloupe, 5 zucchini, and 6 cucumber trials) were conducted in major melon, squash, and cucumber production areas of the USA in 1999 (Graves, 1999; DERBI 92644).

Table 36. Zoxamide residues in cucurbits from supervised trials in the USA

Cucurbits country, (variety)	year	Application					PHI days	Residues ^a mg/kg	Reference Trial number
		Form	kg ai/ha	kg ai/hL	water, L/ha	no.			
GAP, USA		WP	0.14-0.19			8	5		
Cucumber									
Georgia, USA, 1999 (Poinsett-76)		WP	0.22	0.18	192	8	0 3 5 7	0.12 0.06 <u>0.04</u> 0.04	Graves, 1999, DERBI 92644 60998012
New Jersey, USA, 1999 (Cyclone)		WP	0.22	0.079	284	8	0	0.11	Graves, 1999, DERBI 92644 60998013
Florida, USA, 1999 (Meteor)		WP	0.22	0.081	277	8	0	0.02	Graves, 1999, DERBI 92644 60998014
Michigan, USA, 1999 (Marketmore)		SC	0.22	0.10	215	8	0	0.04	Graves, 1999, DERBI 92644 60998015
Michigan, USA, 1999 (Marketmore)		WP	0.22	0.10	217	8	0	0.04	Graves, 1999, DERBI 92644 60998015
Michigan, USA, 1999 (Marketmore)		WP	0.22	0.10	216	8	0	0.02	Graves, 1999, DERBI 92644 60998016
Texas, USA, 1999 (Straight 8)		WP	0.22	0.17	134	8	0	0.01	Graves, 1999, DERBI 92644 60998017
Cantaloupe									
Georgia, USA, 1999 (Planter's Jumbo)		WP	0.22	0.11	196	8	0	0.61	Graves, 1999, DERBI 92644 60998001
Wisconsin, USA, 1999 (Saticoy)		WP	0.22	0.12	188	8	0	0.05	Graves, 1999, DERBI 92644 60998002
Texas, USA, 1999 (Hales Best)		WP	0.22	0.12	135	8	0	0.07	Graves, 1999, DERBI 92644 60998003
California, USA, 1999 (PMR 45)		WP	0.22	0.10	220	8	0	0.28	Graves, 1999, DERBI 92644 60998004
California, USA, 1999 (Summit)		WP	0.22	0.094	238	8	0	0.44	Graves, 1999, DERBI 92644 60998005
California, USA, 1999 (Summit)		SC	0.22	0.094	238	8	0	0.24	Graves, 1999, DERBI 92644 60998005
Arizona, USA, 1999 (Mission)		WP	0.22	0.062	362	8	0 2 4 6	0.04 0.04 <u>0.04</u> 0.04	Graves, 1999, DERBI 92644 34P-98-43SA-1
Summer squash									
New York, USA, 1999 (Zucchini Select)		WP	0.22	0.097	232	8	0	0.17	Graves, 1999, DERBI 92644 60998007
Florida, USA, 1999 (Zucchini)		SC	0.22	0.080	280	8	0	0.05	Graves, 1999, DERBI 92644 60998009
Florida, USA, 1999 (Zucchini)		WP	0.22	0.091	245	8	0	0.05	Graves, 1999, DERBI 92644 60998009
Wisconsin, USA, 1999 (Dividend)		WP	0.22	0.12	188	8	0	0.25	Graves, 1999, DERBI 92644 60998010

Cucurbits country, year (variety)	Application					PHI days	Residues ^a mg/kg	Reference Trial number
	Form	kg ai/ha	kg ai/hL	water, L/ha	no.			
California, USA, 1999 (Zucchini)	WP	0.22	0.079	284	8	0	0.14	Graves, 1999, DERBI 92644 60998011
Georgia, USA, 1999 (Black Beauty)	WP	0.22	0.077	293	8	0 0.375 3 5 7	0.04 0.06 0.04 0.04 0.09	Graves, 1999, DERBI 92644 34P-98-43SA-2

a - Average of replicate samples from the same plot

Supervised trials in the Republic of Korea

Four trials were conducted using an SC formulation containing 8% zoxamide.

Table 37. Zoxamide residues in cucumber from supervised trials in the Republic of Korea

Cucumber Country, year (variety)	Application					PHI days	Residues, mg/kg	Reference
	Form	kg ai/ha	kg ai/hL	water, L/ha	no.			
<i>GAP, Rep. Korea</i>	<i>WP</i>		<i>0.01</i>	<i>Spray enough</i>	<i>3</i>	<i>3</i>		
Rep. Korea, 1998 (Baedadagi)	SC		0.032	2500	3	7 7 7	< 0.01 < 0.01 < 0.01	DERBI 238418
Rep. Korea, 1998 (Baedadagi)	SC		0.032	2500	3	5 5 5	0.02 0.02 0.02	DERBI 238418
Rep. Korea, 1998 (Baedadagi)	SC		0.032	2500	4	3 3 3	0.05 0.05 0.05	DERBI 238418
Rep. Korea, 1998 (Baedadagi)	SC		0.032	2500	4	1 1 1	0.10 0.10 0.10	DERBI 238418

Tomatoes

Supervised trials Europe

A total of 17 relevant trials on tomatoes were conducted outdoors under typical field conditions in Southern Europe (Spain and Italy) in 1998 and 1999 using either the WG (9 trials) or SC (8 trials) formulations. A total of 16 relevant trials were conducted indoors under glass or polytunnel also in Southern Europe (Spain, Southern France and Greece), mostly using the WG formulations (13 trials) with a small number of them conducted using the SC formulations (3 trials) in 1998 and 1999. Studies conducted in 1998 and 1999 used the WG formulation containing 66.67% mancozeb and 8.33% zoxamide and the SC formulation containing 240 g/L zoxamide. No significant differences were noted between residues arising from the different formulation types or between indoor and outdoor trials.

Table 38. Zoxamide residues in tomatoes from supervised field trials in Spain and Italy, and from supervised indoor trials in France, Greece, Italy and Spain

Tomato country, (variety)	year	Application					PHI days	Residues, mg/kg	Reference Trial number
		Form	kg ai/ha	kg ai/hL	water, L/ha	no.			
<i>GAP, Italy</i>		WG	0.14-0.17	0.014-0.017		5	3		
Indoor									
Elx Spain, 1998 (Victorio)		WG	0.15	0.015	1000	4	0 3 7 14 21	0.08 0.06 0.05 0.08 <u>0.09</u>	Wais, 2000, DERBI 224019 A/SP/F/98/115
El Perello Spain, 1998 (Marman da Raf)		WG	0.15	0.015	1000	4	0 3 7 14 21	0.06 0.05 <u>0.09</u> 0.07 0.09	Wais, 2000, DERBI 224019 A/SP/F/98/116
Pastrana Mazarron Spain, 1999 (Rambo)		WG	0.15	0.015	1000	4	0 3 7 14 21	0.15 0.09 <u>0.12</u> 0.12 0.06	Wais, 2000, DERBI 224019 A/SP/F/98/117
Adra Spain, 1999, (Ron Cardo)		WG	0.15	0.015	1000	4	0 3 7 14 21	0.13 0.06 <u>0.15</u> 0.11 0.11	Wais, 2000, DERBI 224019 A/SP/F/98/118
Vilassar de Mar Spain, 1999 (Bon)		SC	0.15-0.19	0.015	1000 - 1300	5	0 3 14	0.27 <u>0.12</u> 0.02	Galy, 2001, DERBI 224326 9065 ES1
Vilassar de Mar, Spain, 1999 (Bon)		WG	0.16-0.19	0.015	1000 - 1300	5	0 3 14	0.22 <u>0.10</u> 0.06	Galy, 2001, DERBI 224795 9065 ES1
Comarca, Spain, 1999 (Bon)		WG	0.15-0.16	0.015	1000 - 1100	5	0 3 14	0.16 <u>0.08</u> 0.04	Galy, 2001, DERBI 224795 9065 ES2
Comarca Spain, 1999 (Bon)		SC	0.15-0.17	0.015	975 - 1100	5	0 3 14	0.03 <u>0.07</u> 0.03	Galy, 2001, DERBI 224326 9065 ES2
Montornes del Valles Spain, 1999 (Bodar)		WG	0.15	0.015	970 - 1100	5	0 14 21 28	0.06 0.02 0.03 0.01	Wais, 2000, DERBI 224795 9065 ES3
Puerta de Mazarron Spain, 1999 (Vica)		WG	0.15	0.015	1000	5	0 1 3 7 14	0.20 0.22 0.20 <u>0.29</u> 0.24	Wais, 2000, DERBI 238371 A/SP/F/99/68
Aguilas Spain, 1999 (Tomas)		WG	0.15	0.015	500	5	0 1 3 7 14	0.20 0.20 0.15 0.22 <u>0.24</u>	Wais, 2000, DERBI 238371 A/SP/F/99/67
Aucamville S France, 1999 (Labell)		WG	0.31-0.48	0.015	2000 - 2300	5	0 1 3 7 14	0.52 0.27 0.24 <u>0.28</u> 0.26	Galy, 2001, DERBI 224795 9065 TL1
Aucamville S France, 1999 (Abigai)		WG	0.26-0.47	0.015	1800 - 2700	5	0 1 3 7 14	0.51 0.40 <u>0.31</u> 0.21 0.26	Galy, 2001, DERBI 224795 9065 TL2

Tomato country, year (variety)	Application					PHI days	Residues, mg/kg	Reference Trial number
	Form	kg ai/ha	kg ai/hL	water, L/ha	no.			
Profitis Greece, 1999 (622)	WG	0.15	0.015	1000	5	0 1 3 7 14	0.34 0.31 0.10 0.15 <u>0.30</u>	Wais, 2000, DERBI 224330 A/GR/F/99/69
Michaniona Greece, 1999 (Noa)	WG	0.15	0.015	1000	5	0 1 3 7 14	0.08 0.13 0.07 <u>0.15</u> 0.06	Wais, 2000, DERBI 224330 A/GR/F/99/70
Profitis Greece, 1999 (622)	SC	0.15	0.015	1000	5	0 1 3 7 14	0.41 0.30 <u>0.30</u> 0.27 0.28	Wais, 2000, DERBI 224331 A/GR/F/99/69
Outdoor								
Costigliole d'Asti Italy, 1998 (Rio Grande)	SC	0.15	0.030	500	5	0 3 10 14 22 28	0.24 0.11 0.10 0.10 <u>0.13</u> 0.10	Wais, 2000, DERBI 224322 A/IT/F/98/98
Costigliole d'Asti Italy, 1998 (Rio Grande)	WG	0.15	0.030	500	5	0 3 10 14 22 28	0.50 <u>0.24</u> 0.20 0.15 0.18 0.13	Wais, 2000, DERBI 224322 A/IT/F/98/98
S. Anna Italy, 1998 (Rio Grande)	SC	0.15	0.030	500	5	0 3 10 14 22 29	0.22 0.13 <u>0.16</u> 0.12 0.06 0.04	Wais, 2000, DERBI 224322 A/IT/F/98/99
S. Anna Italy, 1998 (Rio Grande)	WG	0.15	0.030	500	5	0 3 10 14 22 29	0.17 <u>0.12</u> 0.08 0.05 0.06 0.06	Wais, 2000, DERBI 224322 A/IT/F/98/99
Canelli Italy, 1998 (Rio Grande)	SC	0.15	0.030	500	5	0 3 10 14 20 29	0.26 <u>0.30</u> 0.17 0.24 0.15 0.06	Wais, 2000, DERBI 224322 A/IT/F/98/111
Canelli Italy, 1998 (Rio Grande)	WG	0.15	0.030	500	5	0 3 10 14 20 29	0.20 0.13 0.14 0.12 0.14 <u>0.15</u>	Wais, 2000, DERBI 224322 A/IT/F/98/111
Cremolino Italy, 1998 (Rio Grande)	SC	0.15	0.030	500	5	0 3 10 14 22 28	0.24 <u>0.20</u> 0.15 0.12 0.09 0.04	Wais, 2000, DERBI 224322 A/IT/F/98/112

Tomato country, year (variety)	Application					PHI days	Residues, mg/kg	Reference Trial number
	Form	kg ai/ha	kg ai/hL	water, L/ha	no.			
Cremolino Italy, 1998 (Rio Grande)	WG	0.15	0.030	500	5	0 3 10 14 22 28	0.28 0.12 0.11 0.14 <u>0.18</u> 0.14	Wais, 2000, DERBI 224322 A/IT/F/98/112
Costigliole Italy, 1999 (Rio Grande)	SC	0.15	0.030	500	5	0 3 14 21 28	0.25 <u>0.18</u> 0.08 0.12 0.06	Wais, 2000, DERBI 224328 A/IT/F/99/71
Costigliole Italy, 1999 (Rio Grande)	WG	0.15	0.030	500	5	0 3 7 14 21 28	0.34 0.23 <u>0.24</u> 0.07 0.10 0.08	Wais, 2001, DERBI 224333 A/IT/F/99/71
Isola d'asti Italy, 1999 (Rio Grande)	SC	0.15	0.030	500	5	0 3 14 21 28	0.18 <u>0.14</u> 0.08 0.09 0.06	Wais, 2000, DERBI 224328 A/IT/F/99/72
Isola d'Asti Italy, 1999 (Rio Grande)	WG	0.15	0.030	500	5	0 3 7 14 28	0.19 <u>0.22</u> 0.22 0.12 0.06	Wais, 2001, DERBI 224333 A/IT/F/99/72
Montornes del Valles Spain, 1999 (Bon)	SC	0.14 – 0.16	0.015	916-1060	5	0 3 14 28	0.20 0.04 <u>0.05</u> 0.02	Galy, 2001, DERBI 224326 9065 ES4
Montornes del Valles Spain, 1999 (Bon)	WG	0.15-0.16	0.015	1000	5	0 3 14 28	0.08 <u>0.04</u> 0.02 0.02	Galy, 2001, DERBI 224795 9065 ES4
Corchuela Spain, 1999 (Toledo)	WG	0.15-0.16	0.030	500	5	0 3 7 14 28	0.01 0.02 < 0.01 < 0.01 <u>0.03</u>	Galy, 2001, DERBI 224795 9065 ES5
Corchuela Spain, 1999 (Bon)	SC	0.14 – 0.15	0.030	500	5	0 3 7 14 28	0.10 0.02 <u>0.04</u> < 0.01 < 0.01	Galy, 2001, DERBI 224326 9065 ES6
Corchuela Spain, 1999 (Bon)	WG	0.14-0.16	0.030	500	5	0 3 7 14 28	0.06 <u>0.05</u> < 0.01 0.01 < 0.01	Galy, 2001, DERBI 224795 9065 ES6

Supervised trials in the United States

Sixteen supervised field residue trials on tomatoes were conducted in six states. The trials were conducted using the WP formulation containing 80% zoxamide.

Table 39. Zoxamide residues in tomatoes from supervised trials in the USA

Tomato country, year (variety)	Application					PHI days	Residues, mg/kg	Reference Trial number
	Form	kg ai/ha	kg ai/hL	water, L/ha	no.			
GAP, USA	WG	0.14-0.19			8	5		
Pennsylvania, USA, 1998 (Better Boy)	WP	0.22	0.12	187	10	5	<u>0.22</u>	Graves, 1999, DERBI 92299 60998018
North Carolina, USA, 1998 (Celebrity)	WP	0.22	0.11	210	10	5	<u>0.11</u>	Graves, 1999, DERBI 92299 60998019
Florida, USA, 1998 (Solar Set)	WP	0.22	0.05	428	10	0 3 5 7	0.06 <u>0.18</u> 0.08 0.06	Graves, 1999, DERBI 92299 60998020
Florida, USA, 1998 (BHN-381 ^a)	WP	0.22	0.07	306	10	5	<u>0.20</u>	Graves, 1999, DERBI 92299 60998021
Illinois, USA, 1998 (Better Boy)	WP	0.22	0.09	242	11	5	<u>0.07</u>	Graves, 1999, DERBI 92299 60998022
California, USA, 1998 (UC 82B)	WP	0.22	0.10	234	10	5	<u>0.10</u>	Graves, 1999, DERBI 92299 60998023
California, USA, 1998 (Halley 3155)	WP	0.22	0.12	187	10	5	<u>0.16</u>	Graves, 1999, DERBI 92299 60998024
Arizona, USA, 1998 (YF-1 Roma)	WP	0.22	0.08	277	10	5	<u>0.12</u>	Graves, 1999, DERBI 92299 60998025
California, USA, 1998 (3155)	WP	0.22	0.08	283	10	5	<u>0.19</u>	Graves, 1999, DERBI 92299 60998026
California, USA, 1998 (3155)	SC	0.22	0.08	282	10	5	<u>0.21</u>	Graves, 1999, DERBI 92299 60998027
California, USA, 1998 (3155)	WP	0.22	0.08	282	10	5	<u>0.32</u>	Graves, 1999, DERBI 92299 60998027
California, USA, 1998 (Red Cherry ^a)	WP	0.22	0.08	283	10	5	<u>1.0</u>	Graves, 1999, DERBI 92299 60998028
California, USA, 1998 (Heinz 8892)	WP	0.22	0.12	190	10	5	<u>0.23</u>	Graves, 1999, DERBI 92299 60998029
California, USA, 1998 (Peto 512)	WP	0.22	0.12	190	10	0 3 5 7	0.20 0.24 <u>0.18</u> 0.10	Graves, 1999, DERBI 92299 60998030
California, USA, 1998 (8892)	WP	0.22	0.08	282	10	5	<u>0.40</u>	Graves, 1999, DERBI 92299 60998031
California, USA, 1998 (8892)	SC	0.22	0.08	282	10	5	<u>0.38</u>	Graves, 1999, DERBI 92299 60998031
California, USA, 1998 (8892)	WP	0.22	0.08	282	13	5	<u>0.13</u>	Graves, 1999, DERBI 92299 60998032
California, USA, 1998 (Heinz 8892)	WP	0.22	0.08	282	11	5	<u>0.21</u>	Graves, 1999, DERBI 92299 60998033

a - Cherry tomato

Supervised trials in Brazil

Sixteen supervised field residue trials on tomatoes were conducted at three locations in Brazil. Eight of the trials were conducted using a WG formulation containing zoxamide and cymoxanil; eight trials were conducted using a WP formulation containing zoxamide and mancozeb.

Table 40. Zoxamide residues in tomatoes from supervised trials in Brazil

Tomato country, year (variety)	Application					PHI days	Residues, mg/kg	Reference Trial number
	Form	kg ai/ha	kg ai/hL	water, L/ha	no.			
<i>GAP, Brazil</i>	WP	0.10-0.13		800	NS	7		
Brazil, 2002 (Carmen)	WP	0.14	0.014	1000	8	7	0.09, <u>0.14</u>	Kalvan, 2002, DERBI 105233
Brazil, 2002 (Carmen)	WP	0.27	0.027	1000	8	7	0.13, 0.14	Kalvan, 2002, DERBI 105233
Brazil, 1998 (Carmen)	WP	0.14	0.014	1000	8	7	<u>0.03</u>	Tornisielo, 1999, DERBI 221855 BR98F02A
Brazil, 1998 (Carmen)	WP	0.27	0.027	1000	8	7	0.05	Tornisielo, 1999, DERBI 221855 BR98F02A
Brazil, 1998 (Santa Clara)	WP	0.14	0.014	1000	9	0 3 7 10 14	0.03 0.03 <u>0.02</u> 0.02 0.01	Tornisielo, 1999, DERBI 221879 BR98F02
Brazil, 1998 (Santa Clara)	WP	0.27	0.027	1000	9	7	0.04	Tornisielo, 1999, DERBI 221879 BR98F02
Brazil, 2003 (Rio Grande)	WG	0.13	0.022	600	3	14	0.03, 0.04	Oliveira, 2003, DERBI 135350
Brazil, 2003 (Rio Grande)	WG	0.26	0.044	600	3	14	0.15, 0.17	Oliveira, 2003, DERBI 135350
Brazil, 1999 (Santa Clara)	WG	0.13	0.013	1000	6	0 3 7 14 21	0.10 0.09 0.01 <u>0.02</u> < 0.01	Tornisielo, 1999, DERBI 224090 31799054
Brazil, 1999 (Santa Clara)	WG	0.26	0.027	1000	6	7	0.13	Tornisielo, 1999, DERBI 224090 31799054
Brazil, 1999 (Carmen)	WG	0.13	0.013	1000	6	7	<u>0.01</u>	Tornisielo, 2000, DERBI 224091 33199036
Brazil, 1999 (Carmen)	WG	0.26	0.027	1000	6	7	0.02	Tornisielo, 2000, DERBI 224091 33199036

Potatoes

Residue data are available from supervised trials conducted in Europe, North America and South America. In Europe and North America the tubers were analyzed for zoxamide and the RH-4952 and RH-4955 metabolites. In South America only residues of zoxamide were determined.

Supervised trials in Europe

A total of 25 trials on potatoes have been conducted in northern Europe from 1996 through 1999. Another 23 trials were conducted in southern Europe in 1996, 1997 and 1999.

Studies conducted in 1996 and 1997 used the WG formulation containing 66.67% mancozeb and 8.33% zoxamide and the SC formulation containing 240g/L zoxamide. A WP formulation also

containing 66.67% mancozeb and 8.33% zoxamide was introduced into trials from 1998 in parallel with WG trials. No differences were noted between residues arising from the different formulation types.

Table 41. Zoxamide residues in potatoes from supervised trials in France, Germany, Greece, Italy, Spain and United Kingdom

Potato country, (variety)	year	Application					PHI days	Residues, mg/kg			Reference Trial number
		Form	kg ai/ha	kg ai/hL	water, L/ha	no.		zoxamide	RH-1452	RH-1455	
<i>GAP, UK, Ireland, Netherlands</i>		WG	0.12-0.15		200-600	10	7				
Pas de Calais, France, (Bintje)	1996	SC	0.15			10	0 7 14 20 28	< 0.02 <u>< 0.02</u> < 0.02 < 0.02 < 0.02	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	0.11 < 0.02 0.03 0.09 0.06	Grolleau, 1999, DERBI 148181 FR01
Pas de Calais, France, (Bintje)	1996	WG	0.15			10	0 7 14 20 28	< 0.02 <u>< 0.02</u> < 0.02 < 0.02 < 0.02	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	Grolleau, 1999, DERBI 148181 FR01
Pas de Calais, France, (Bintje)	1997	WG	0.15			10	0 2 7 14 21	< 0.02 < 0.02 <u>< 0.02</u> < 0.02 < 0.02	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	Grolleau, 1999, DERBI 148183 FR01
Pas de Calais, France, (Bintje)	1997	SC	0.15			10	0 2 7 14 21	< 0.02 < 0.02 <u>< 0.02</u> < 0.02 < 0.02	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	Grolleau, 1999, DERBI 148183 FR01
Dame Marie Les Bois, France, (Nicola)	1999	WG	0.15			10	0 7	< 0.02 <u>< 0.02</u>	< 0.02 < 0.02	< 0.02 < 0.02	Wais, 2000, DERBI 148179 A/NF/F/99/62
Dame Marie Les Bois, France, (Nicola)	1999	WG	0.15			10	0 7	< 0.02 <u>< 0.02</u>	< 0.02 < 0.02	< 0.02 < 0.02	Wais, 2000, DERBI 148182 A/NF/F/99/62
Rossdorf, Germany, 1996 (Agria)		SC	0.15			10	0 7 14 22 28	< 0.02 <u>< 0.02</u> < 0.02 < 0.02 < 0.02	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	Wais, 1999, DERBI 221389 A/GE/F/96/31
Rossdorf, Germany, (Agria)	1996	WG	0.15			10	0 7 14 22 28	< 0.02 <u>< 0.02</u> < 0.02 < 0.02 < 0.02	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	Wais, 1999, DERBI 221389 A/GE/F/96/31
Rossdorf, Germany, (Solara)	1997	WG	0.15			10	0 3 7 14 21	< 0.02 < 0.02 <u>< 0.02</u> < 0.02 < 0.02	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	Wais, 1999, DERBI 221391 A/GE/F/97/5
Rossdorf, Germany, (Solara)	1997	SC	0.15			10	0 3 7 14 21	< 0.02 < 0.02 <u>< 0.02</u> < 0.02 < 0.02	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	Wais, 1999, DERBI 221391 A/GE/F/97/5
Rossdorf, Germany, (Agria)	1998	WG	0.15			10	0 7 14	< 0.02 <u>< 0.02</u> < 0.02	< 0.02 < 0.02 < 0.02	< 0.02 < 0.02 < 0.02	Wais, 1999, DERBI 221380 A/GE/F/98/88

Potato country, (variety)	year	Application					PHI days	Residues, mg/kg			Reference Trial number
		Form	kg ai/ha	kg ai/hL	water, L/ha	no.		zoxamide	RH-1452	RH-1455	
Rossdorf, Germany, (Agria)	1998	WP	0.15			10	0 7 14	< 0.02 <u>< 0.02</u> < 0.02	< 0.02 < 0.02 < 0.02	< 0.02 < 0.02 < 0.02	Wais, 1999, DERBI 221380 A/GE/F/98/88
Rossdorf, Germany, (Quarta)	1998	WG	0.15			10	0 7 14	< 0.02 <u>< 0.02</u> < 0.02	< 0.02 < 0.02 < 0.02	< 0.02 < 0.02 < 0.02	Wais, 1999, DERBI 221380 A/GE/F/98/89
Kent, United Kingdom, 1996 (Romano)		WG	0.15			10	0 7 14 21 28	< 0.02 <u>< 0.02</u> < 0.02 < 0.02 < 0.02	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	Wais, 1999, DERBI 221860 A/UK/F/96/32
Kent, United Kingdom, 1996 (Romano)		SC	0.15			10	0 7 14 21 28	< 0.02 <u>< 0.02</u> < 0.02 < 0.02 < 0.02	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	Wais, 1999, DERBI 221860 A/UK/F/96/32
Kent, United Kingdom, 1996 (Maris Piper)		WG	0.15			10	7	<u>< 0.02</u>	< 0.02	< 0.02	Wais, 1999, DERBI 221860 A/UK/F/96/33
Kent, United Kingdom, 1996 (Maris Piper)		SC	0.15			10	7	<u>< 0.02</u>	< 0.02	< 0.02	Wais, 1999, DERBI 221860 A/UK/F/96/33
Sheldwich, United Kingdom, 1997 (Estima)		WG	0.15			10	0 3 7 14 20	< 0.02 < 0.02 <u>< 0.02</u> < 0.02 < 0.02	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	Wais, 1999, DERBI 221401 A/UK/F/97/9
Sheldwich, United Kingdom, 1997 (Estima)		SC	0.15			10	0 3 7 14	< 0.02 < 0.02 <u>< 0.02</u> < 0.02	< 0.02 < 0.02 < 0.02 < 0.02	< 0.02 < 0.02 < 0.02 < 0.02	Wais, 1999, DERBI 221401 A/UK/F/97/9
East Tilbury, United Kingdom, 1997 (Cara)		WG	0.15			10	0 7 14	< 0.02 <u>< 0.02</u> < 0.02	< 0.02 < 0.02 < 0.02	< 0.02 < 0.02 < 0.02	Wais, 1999, DERBI 221401 A/UK/F/97/10
East Tilbury, United Kingdom, 1997 (Cara)		SC	0.15			10	0 7 14	< 0.02 <u>< 0.02</u> < 0.02	< 0.02 < 0.02 < 0.02	< 0.02 < 0.02 < 0.02	Wais, 1999, DERBI 221401 A/UK/F/97/10
East Tilbury, United Kingdom, 1997 (Cara)		WG	0.15			10	0 7 13	< 0.02 <u>< 0.02</u> < 0.02	< 0.02 < 0.02 < 0.02	< 0.02 < 0.02 < 0.02	Wais, 1999, DERBI 221386 A/UK/F/98/90
United Kingdom, East Tilbury 1997 (Cara)		WP	0.15			10	0 7 13	< 0.02 <u>< 0.02</u> < 0.02	< 0.02 < 0.02 < 0.02	< 0.02 < 0.02 < 0.02	Wais, 1999, DERBI 221386 A/UK/F/98/90
Sheldwich, United Kingdom 1997 (Maris Bard)		WG	0.15			10	0 7 14	< 0.02 <u>< 0.02</u> < 0.02	< 0.02 < 0.02 < 0.02	< 0.02 < 0.02 < 0.02	Wais, 1999, DERBI 221386 A/UK/F/98/91
Ens, Noordoost Polder, Netherlands, 1999 (Agria)		WG	0.15			10	0 7	< 0.02 <u>< 0.02</u>	< 0.02 < 0.02	< 0.02 < 0.02	Wais, 2000, DERBI 148180 A/NL/F/99/64
<i>GAP, Italy</i>		WG	<i>0.14-0.17</i>			NS	7				
Roussillon, France, 1996 (Concurrent)		WG	0.15	0.014-0.017		7	0 7 14 21 28	< 0.02 <u>< 0.02</u> < 0.02 < 0.02 < 0.02	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	Grolleau, 1999, DERBI 148181 FR02

Potato country, (variety)	year	Application					PHI days	Residues, mg/kg			Reference Trial number
		Form	kg ai/ha	kg ai/hL	water, L/ha	no.		zoxamide	RH-1452	RH-1455	
Roussillon, France, (Concurrent)	1996	SC	0.15			7	0	< 0.02	< 0.02	< 0.02	Grolleau, 1999, DERBI 148181 FR02
							7	<u>< 0.02</u>	< 0.02	< 0.02	
							14	< 0.02	< 0.02	< 0.02	
							21	< 0.02	< 0.02	< 0.02	
							28	< 0.02	< 0.02	< 0.02	
Aquitaine, France, (Spunta)	1996	WG	0.15			7	0	< 0.02	< 0.04	< 0.02	Grolleau, 1999, DERBI 148181 FR03
							7	<u>< 0.02</u>	< 0.02	< 0.02	
							14	< 0.02	0.02	< 0.02	
							20	< 0.02	< 0.02	< 0.02	
							28	< 0.02	< 0.02	< 0.02	
Aquitaine, France, (Spunta)	1996	SC	0.15			7	0	< 0.02	< 0.02	< 0.02	Grolleau, 1999, DERBI 148181 FR03
							7	<u>< 0.02</u>	< 0.02	< 0.02	
							14	< 0.02	< 0.02	< 0.02	
							20	< 0.02	< 0.02	< 0.02	
							28	< 0.02	< 0.02	< 0.02	
Midi-Pyrenees, France, 1997 (Bea)	1997	WG	0.15			7	0	< 0.02	< 0.02	< 0.02	Grolleau, 1999, DERBI 148183 FR02
							2	< 0.02	< 0.02	< 0.02	
							7	<u>< 0.02</u>	< 0.02	< 0.02	
							14	< 0.02	< 0.02	< 0.02	
							21	< 0.02	< 0.02	< 0.02	
Midi-Pyrenees, France, 1997 (Bea)	1997	SC	0.15			7	0	< 0.02	< 0.02	< 0.02	Grolleau, 1999, DERBI 148183 FR02
							2	< 0.02	< 0.02	< 0.02	
							7	<u>< 0.02</u>	< 0.02	< 0.02	
							14	< 0.02	< 0.02	< 0.02	
							21	< 0.02	< 0.02	< 0.02	
Hortero, Greece, (Spunta)	1997	WG	0.15			10	0	< 0.02	< 0.02	< 0.02	Wais, 1999, DERBI 221392 A/GR/F/97/13
							7	<u>< 0.02</u>	< 0.02	< 0.02	
							14	< 0.02	< 0.02	< 0.02	
Hortero, Greece, (Spunta)	1997	SC	0.15			10	0	< 0.02	< 0.02	< 0.02	Wais, 1999, DERBI 221392 A/GR/F/97/13
							7	<u>< 0.02</u>	< 0.02	< 0.02	
							14	< 0.02	< 0.02	< 0.02	
Kozani, Greece, (Afroditi)	1997	WG	0.15			10	0	< 0.02	< 0.02	< 0.02	Wais, 1999, DERBI 221392 A/GR/F/97/14
							7	<u>< 0.02</u>	< 0.02	< 0.02	
							14	< 0.02	< 0.02	< 0.02	
Kozani, Greece, (Afroditi)	1997	SC	0.15			10	0	< 0.02	< 0.02	< 0.02	Wais, 1999, DERBI 221392 A/GR/F/97/14
							7	<u>< 0.02</u>	< 0.02	< 0.02	
							14	< 0.02	< 0.02	< 0.02	
Selva Malezzi, Italy, 1996 (Mona Lisa)	1996	WG	0.18			10	7	<u>< 0.02</u>	< 0.02	< 0.02	Wais, 1999, DERBI 221395 A/IT/F/96/34
Selva Malezzi, Italy, 1996 (Mona Lisa)	1996	SC	0.15			10	7	<u>< 0.02</u>	< 0.02	< 0.02	Wais, 1999, DERBI 221395 A/IT/F/96/34
Voghera, Italy, 1996 (Adora)	1996	WG	0.19			10	0	< 0.02	< 0.02	< 0.02	Wais, 1999, DERBI 221395 A/IT/F/96/35
							8	<u>< 0.02</u>	< 0.02	< 0.02	
							15	< 0.02	< 0.02	< 0.02	
							22	< 0.02	< 0.02	< 0.02	
							29	< 0.02	< 0.02	< 0.02	
Voghera, Italy, 1996 (Adora)	1996	SC	0.16			10	0	< 0.02	< 0.02	< 0.02	Wais, 1999, DERBI 221395 A/IT/F/96/35
							8	<u>< 0.02</u>	< 0.02	< 0.02	
							15	< 0.02	< 0.02	< 0.02	
							22	< 0.02	< 0.02	< 0.02	
							29	< 0.02	< 0.02	< 0.02	
Carpento, Italy, 1997 (Bintje)	1997	WG	0.15			10	0	< 0.02	< 0.02	< 0.02	Wais, 1999, DERBI 221394 A/IT/F/97/8
							3	< 0.02	< 0.02	< 0.02	
							7	<u>< 0.02</u>	< 0.02	< 0.02	
							14	< 0.02	< 0.02	< 0.02	
							21	< 0.02	< 0.02	< 0.02	

Potato country, (variety)	year	Application					PHI days	Residues, mg/kg			Reference Trial number
		Form	kg ai/ha	kg ai/hL	water, L/ha	no.		zoxamide	RH-1452	RH-1455	
Carpento, Italy, 1997 (Bintje)	SC	0.15			10	0	< 0.02	< 0.02	< 0.02	Wais, 1999, DERBI 221394 A/IT/F/97/8	
						3	< 0.02	< 0.02	< 0.02		
						7	<u>< 0.02</u>	< 0.02	< 0.02		
						14	< 0.02	< 0.02	< 0.02		
					10	21	< 0.02	< 0.02	< 0.02		
						7	<u>< 0.02</u>	< 0.02	< 0.02	Wais, 2000, DERBI 148184 A/IT/F/99/61	
Centelles, Spain, 1997 (Buffet)	WG	0.15			10	0	< 0.02	< 0.02	< 0.02	Wais, 1999, DERBI 221388 A/SP/F/98/92	
						7	<u>< 0.02</u>	< 0.02	< 0.02		
						14	< 0.02	< 0.02	< 0.02		
Centelles, Spain, 1997 (Buffet)	WP	0.15			10	0	< 0.02	< 0.02	< 0.02	Wais, 1999, DERBI 221388 A/SP/F/98/92	
						7	<u>< 0.02</u>	< 0.02	< 0.02		
						14	< 0.02	< 0.02	< 0.02		
Les Peres, Spain, 1997 (Kennebeck)	WG	0.15			10	0	< 0.02	< 0.02	< 0.02	Wais, 1999, DERBI 221388 A/SP/F/98/93	
						7	<u>< 0.02</u>	< 0.02	< 0.02		
						14	< 0.02	< 0.02	< 0.02		
Les Peres, Spain, 1997 (Kennebeck)	WP	0.15			10	0	< 0.02	< 0.02	< 0.02	Wais, 1999, DERBI 221388 A/SP/F/98/93	
						7	<u>< 0.02</u>	< 0.02	< 0.02		
						14	< 0.02	< 0.02	< 0.02		
Puigcerda, Spain, 1997 (Buffet)	WG	0.15			10	0	< 0.02	< 0.02	< 0.02	Wais, 1999, DERBI 221388 A/SP/F/98/120	
						7	<u>< 0.02</u>	< 0.02	< 0.02		
						14	< 0.02	< 0.02	< 0.02		
Centelles, Spain, 1997 (Kennebeck)	WG	0.15			10	0	< 0.02	< 0.02	< 0.02	Wais, 1999, DERBI 221388 A/SP/F/98/119	
						7	<u>< 0.02</u>	< 0.02	< 0.02		
						14	< 0.02	< 0.02	< 0.02		

Supervised trials in North America

Twelve supervised field residue trials on potatoes were conducted in seven zones across Canada. Each trial consisted of at least 10 applications at 7 – 10 day intervals. While the GAP allows for a maximum of 6 applications these trials were considered in compliance with the GAP since zoxamide is not systemic and the applications were made over a two-month period

Sixteen supervised field residue trials on potatoes were conducted in 10 states in the USA over two growing seasons. The trials were conducted using the WP formulation containing 80% zoxamide and SC formulation containing 23% zoxamide. No differences were noted between residues arising from the different formulation types.

Table 42. Zoxamide residues in potatoes from supervised trials in Canada and the USA

Potato country, (variety)	year	Application					PHI days	Residues, mg/kg			Reference Trial number
		Form	kg ai/ha	kg ai/hL	water, L/ha	no.		zoxamide	RH-1452	RH-1455	
<i>GAP, Canada</i>		WG	0.19		45		3				
Prince Edward Island, Canada, 1998 (Century Russet)		WP	0.20	0.066	301	10	3	<u>< 0.02</u>	< 0.02	< 0.02	Vaughn, 1998, DERBI 92289 RHC04-A
Prince Edward Island, Canada, 1998 (Century Russet)		WP	0.20	0.066	301	10	3	<u>< 0.02</u>	< 0.02	< 0.02	Vaughn, 1998, DERBI 92289 RHC04-B
Prince Edward Island, Canada, 1998 (Russet Burbank)		WP	0.20	0.066	300	10	3	<u>< 0.02</u>	< 0.02	< 0.02	Vaughn, 1998, DERBI 92289 RHC04-C

Potato country, (variety)	year	Application					PHI days	Residues, mg/kg			Reference Trial number
		Form	kg ai/ha	kg ai/hL	water, L/ha	no.		zoxamide	RH-1452	RH-1455	
Prince Edward Island, Canada, 1998 (Russet Burbank)		WP	0.20	0.066	303	10	3	<u>< 0.02</u>	< 0.02	< 0.02	Vaughn, 1998, DERBI 92289 RHC04-D
New Brunswick, Canada, 1998 (Superior)		WP	0.20	0.066	301	10	3	<u>< 0.02</u>	< 0.02	< 0.02	Vaughn, 1998, DERBI 92289 RHC04-E
Quebec, Canada, 1998		WP	0.20	0.079	252	10	3	<u>< 0.02</u>	< 0.02	< 0.02	Vaughn, 1998, DERBI 92289 RHC04-F
Ontario, Canada, 1998 (Chieften)		WP	0.20	0.079	253	10	3	<u>< 0.02</u>	< 0.02	< 0.02	Vaughn, 1998, DERBI 92289 RHC04-G
Ontario, Canada, 1998 (Kennebec)		WP	0.20	0.079	254	10	3	<u>< 0.02</u>	< 0.02	< 0.02	Vaughn, 1998, DERBI 92289 RHC04-H
Manitoba, Canada, 1998 (Russet Burbank)		WP	0.20	0.066	301	10	3	<u>< 0.02</u>	< 0.02	< 0.02	Vaughn, 1998, DERBI 92289 RHC04-I
Alberta, Canada, 1998		WP	0.20	0.066	302	10	3	<u>< 0.02</u>	< 0.02	< 0.02	Vaughn, 1998, DERBI 92289 RHC04-J
Alberta, Canada, 1998 (Russet Burbank)		WP	0.20	0.079	252	10	3	<u>< 0.02</u>	< 0.02	< 0.02	Vaughn, 1998, DERBI 92289 RHC04-K
British Columbia, Canada, 1998 (Russet Burbank)		WP	0.20	0.079	252	10	3	<u>< 0.02</u>	< 0.02	< 0.02	Vaughn, 1998, DERBI 92289 RHC04-L
<i>GAP, USA</i>		<i>WG</i>	<i>0.14-0.19</i>			<i>6</i>	<i>3</i>				
North Carolina, USA, 1996 (La Soda)		WP	0.14	0.08	188	10	3	<u>ND</u>	ND	< 0.02	Graves, 1998, DERBI 92643 21696209
North Carolina, USA, 1996 (La Soda)		WP	0.28	0.15	188	10	3	ND	ND	< 0.02	Graves, 1998, DERBI 92643 21696209
New York, USA, 1996 (Chippewa)		WP	0.14	0.08	187	10	0 3 7 14	ND <u>ND</u> ND ND	< 0.02 < 0.02 ND ND	ND ND ND ND	Graves, 1998, DERBI 92643 21696208
New York, USA, 1996 (Chippewa)		WP	0.28	0.15	187	10	0 3 7 14	ND ND ND ND	< 0.02 < 0.02 < 0.02 < 0.02	< 0.02 ND < 0.02 ND	Graves, 1998, DERBI 92643 21696208
Idaho, USA, 1996 (Russet Burbank)		WP	0.14	0.04	318	10	0 3 7 14	ND <u>ND</u> ND ND	ND ND ND ND	ND ND ND ND	Graves, 1998, DERBI 92643 21696212
Idaho, USA, 1996 (Russet Burbank)		WP	0.28	0.09	318	10	0 3 7 14	ND ND ND ND	ND < 0.02 ND < 0.02	ND ND ND < 0.02	Graves, 1998, DERBI 92643 21696212
Wisconsin, USA, 1996 (Russet)		WP	0.14	0.08	187	10	3	<u>< 0.02</u>	< 0.02	ND	Graves, 1998, DERBI 92643 21696210
Wisconsin, USA, 1996 (Russet)		WP	0.28	0.15	187	10	3	< 0.02	< 0.02	ND	Graves, 1998, DERBI 92643 21696210
Washington, USA, 1996 (Norkotah)		WP	0.14	0.05	291	10	3	<u>ND</u>	< 0.02	< 0.02	Graves, 1998, DERBI 92643 21696211

Potato country, (variety)	year	Application					PHI days	Residues, mg/kg			Reference Trial number
		Form	kg ai/ha	kg ai/hL	water, L/ha	no.		zoxamide	RH-1452	RH-1455	
Washington, USA, 1996 (Norkotah)		WP	0.28	0.10	291	10	3	< 0.02	< 0.02	< 0.02	Graves, 1998, DERBI 92643 21696211
Florida, USA, 1997 (Red Lasoda)		WP	0.22	0.08	278	10	3	<u>< 0.02</u>	ND	< 0.02	Graves, 1998, DERBI 92643 21697002
Florida, USA, 1997 (Red Lasoda)		WP	0.45	0.16	276	10	3	ND	ND	< 0.02	Graves, 1998, DERBI 92643 21697002
Florida, USA, 1997 (Red Lasoda)		SC	0.22	0.08	279	10	3	<u>< 0.02</u>	ND	< 0.02	Graves, 1998, DERBI 92643 21697002
Wisconsin, USA, 1997 (Norland Dark Red)		WP	0.22	0.12	190	10	3	<u>ND</u>	ND	< 0.02	Graves, 1998, DERBI 92643 21697003
Wisconsin, USA, 1997 (Norland Dark Red)		WP	0.45	0.24	189	10	3	ND	ND	< 0.02	Graves, 1998, DERBI 92643 21697003
Wisconsin, USA, 1997 (Norland Dark Red)		SC	0.22	0.12	188	10	3	<u>ND</u>	ND	< 0.02	Graves, 1998, DERBI 92643 21697003
Washington, USA, 1997 (Russet)		WP	0.23	0.11	201	10	3	<u>ND</u>	< 0.02	ND	Graves, 1998, DERBI 92643 21697003
Washington, USA, 1997 (Russet)		WP	0.45	0.22	201	10	3	ND	0.02	< 0.02	Graves, 1998, DERBI 92643 21697003
Washington, USA, 1997 (Russet)		SC	0.22	0.11	201	10	3	<u>ND</u>	< 0.02	ND	Graves, 1998, DERBI 92643 21697003
Washington, USA, 1997 (Russet)		WP	1.12	0.56	200	10	3	ND	0.03	< 0.02	Graves, 1998, DERBI 92643 21697003
Ohio, USA, 1997 (Langlade)		WP	0.22	0.10	215	10	3	<u>ND</u>	ND	< 0.02	Graves, 1998, DERBI 92643 21697004
Ohio, USA, 1997 (Langlade)		WP	0.45	0.20	221	10	3	ND	< 0.02	< 0.02	Graves, 1998, DERBI 92643 21697004
Ohio, USA, 1997 (Langlade)		SC	0.22	0.11	212	10	3	<u>< 0.02</u>	ND	ND	Graves, 1998, DERBI 92643 21697004
California, USA, 1997 (White Rose)		WP	0.23	0.08	282	10	3	<u>ND</u>	< 0.02	< 0.02	Graves, 1998, DERBI 92643 21697007
California, USA, 1997 (White Rose)		WP	0.45	0.16	281	10	3	ND	ND	ND	Graves, 1998, DERBI 92643 21697007
California, USA, 1997 (White Rose)		SC	0.22	0.08	281	10	3	<u>ND</u>	ND	ND	Graves, 1998, DERBI 92643 21697007
Wisconsin, USA, 1997 (Russet Burbank)		WP	0.22	0.12	189	10	3	<u>ND</u>	< 0.02	ND	Graves, 1998, DERBI 92643 21697005
Wisconsin, USA, 1997 (Russet Burbank)		WP	0.45	0.24	189	10	3	< 0.02	< 0.02	< 0.02	Graves, 1998, DERBI 92643 21697005
Wisconsin, USA, 1997 (Russet Burbank)		SC	0.22	0.12	188	10	3	<u>< 0.02</u>	< 0.02	ND	Graves, 1998, DERBI 92643 21697005

Potato country, (variety)	year	Application					PHI days	Residues, mg/kg			Reference Trial number
		Form	kg ai/ha	kg ai/hL	water, L/ha	no.		zoxamide	RH-1452	RH-1455	
Pennsylvania, USA, 1997 (Yukon Gold)		WP	0.23	0.07	312	10	3	<u>ND</u>	< 0.02	ND	Graves, 1998, DERBI 92643 21697001
Pennsylvania, USA, 1997 (Yukon Gold)		WP	0.46	0.14	313	10	3	ND	< 0.02	< 0.02	Graves, 1998, DERBI 92643 21697001
Pennsylvania, USA, 1997 (Yukon Gold)		SC	0.23	0.07	310	10	3	<u>ND</u>	ND	ND	Graves, 1998, DERBI 92643 21697001
Idaho, USA, 1997 (Russet Burbank)		WP	0.22	0.08	280	10	3	<u>ND</u>	ND	ND	Graves, 1998, DERBI 92643 21697010
Idaho, USA, 1997 (Russet Burbank)		WP	0.45	0.16	280	10	3	ND	< 0.02	ND	Graves, 1998, DERBI 92643 21697010
Idaho, USA, 1997 (Russet Burbank)		SC	0.22	0.08	277	10	3	<u>ND</u>	ND	ND	Graves, 1998, DERBI 92643 21697010
Idaho, USA, 1997 (Russet Burbank)		WP	0.22	0.07	303	10	3	<u>ND</u>	ND	ND	Graves, 1998, DERBI 92643 21697011
Idaho, USA, 1997 (Russet Burbank)		WP	0.45	0.15	303	10	3	ND	< 0.02	ND	Graves, 1998, DERBI 92643 21697011
Idaho, USA, 1997 (Russet Burbank)		SC	0.23	0.07	308	10	3	<u>ND</u>	ND	ND	Graves, 1998, DERBI 92643 21697011
Colorado, USA, 1997 (Norkotah)		WP	0.22	0.08	279	10	3	<u>ND</u>	ND	< 0.02	Graves, 1998, DERBI 92643 21697006
Colorado, USA, 1997 (Norkotah)		WP	0.45	0.16	278	10	3	ND	< 0.02	0.02	Graves, 1998, DERBI 92643 21697006
Colorado, USA, 1997 (Norkotah)		SC	0.22	0.08	285	10	3	<u>ND</u>	ND	< 0.02	Graves, 1998, DERBI 92643 21697006
Washington, USA, 1997 (Russet Norkotah)		WP	0.23	0.08	300	10	3	<u>ND</u>	ND	< 0.02	Graves, 1998, DERBI 92643 21697009
Washington, USA, 1997 (Russet Norkotah)		WP	0.45	0.15	299	10	3	ND	ND	ND	Graves, 1998, DERBI 92643 21697009
Washington, USA, 1997 (Russet Norkotah)		SC	0.23	0.08	299	10	3	<u>ND</u>	ND	< 0.02	Graves, 1998, DERBI 92643 21697009

Supervised trials in Central and South America

Two supervised trials were conducted in Mexico. Eleven supervised trials were conducted on potatoes in 2002 and 2003 in two provinces of Argentina. Five supervised trials were conducted over four growing seasons in Brazil.

Table 43. Zoxamide residues in potatoes from supervised trials in Mexico

Potato country, year (variety)	Application					PHI days	Residues, mg/kg			Reference Trial number
	Form	kg ai/ha	kg ai/hL	water, L/ha	no.		zoxamide	RH-1452	RH-1455	
<i>GAP, Mexico</i>	WG	0.10-0.15		200-400	6	7				
Sinaloa, Mexico, 1999 (Alfa)	WP	0.15	0.07	199	8	14	< 0.01	< 0.02	< 0.02	West, 2000, DERBI 109986 220-98-002
Sinaloa, Mexico, 1999 (Alfa)	WP	0.30	0.15	199	8	14	< 0.01	< 0.02	< 0.02	West, 2000, DERBI 109986 220-98-002
Sinaloa, Mexico, 1999 (Alfa)	WP	0.15	0.07	199	8	14	< 0.01	< 0.02	< 0.02	West, 2000, DERBI 109986 220-98-002
Sinaloa, Mexico, 1999 (Alfa)	WP	0.30	0.15	202	8	14	< 0.01	< 0.02	< 0.02	West, 2000, DERBI 109986 220-98-002
Guanajuato, Mexico, 1999 (Alfa)	WP	0.15	0.07	199	8	13	< 0.01	< 0.02	< 0.02	West, 2000, DERBI 109986 220-98-003
Guanajuato, Mexico, 1999 (Alfa)	WP	0.30	0.15	200	8	13	< 0.01	< 0.02	< 0.02	West, 2000, DERBI 109986 220-98-003
Guanajuato, Mexico, 1999 (Alfa)	WP	0.15	0.08	199	8	13	< 0.01	< 0.02	< 0.02	West, 2000, DERBI 109986 220-98-003
Guanajuato, Mexico, 1999 (Alfa)	WP	0.30	0.15	199	8	13	< 0.01	< 0.02	< 0.02	West, 2000, DERBI 109986 220-98-003

Table 44. Zoxamide residues in potatoes from supervised trials in Argentina and Brazil

Potato country, year (variety)	Application					PHI days	Residues, mg/kg	Reference Trial number
	Form	kg ai/ha	kg ai/hL	water, L/ha	no.			
<i>GAP, Argentina</i>	WP	0.15		400-1000	NS	7		
Sante Fe, Argentina, 2002 (Kennebec)	WP	0.11	0.037	300	6	8	≤ 0.05	Kalvan, 2003, DERBI 224603 02
Sante Fe, Argentina, 2002 (Kennebec)	WP	0.22	0.074	300	6	8	< 0.05	Kalvan, 2003, DERBI 224603 02
Buenos Aires, Argentina, 2002 (Kennebec)	WP	0.11	0.037	300	4	7	≤ 0.05	Kalvan, 2003, DERBI 224603 02
Buenos Aires, Argentina, 2002 (Kennebec)	WP	0.22	0.074	300	4	7	< 0.05	Kalvan, 2003, DERBI 224603 02
Sante Fe, Argentina, 2003 (Kennebec)	WP	0.15	0.049	300	4	12	< 0.05	Kalvan, 2003, DERBI 224603 03
Sante Fe, Argentina, 2003 (Kennebec)	WP	0.29	0.097	300	4	12	< 0.05	Kalvan, 2003, DERBI 224603 03

Potato country, year (variety)	Application					PHI days	Residues, mg/kg	Reference Trial number
	Form	kg ai/ha	kg ai/hL	water, L/ha	no.			
Buenos Aires, Argentina, 2003 (Kennebec)	WP	0.15	0.049	300	4	12	< 0.05	Kalvan, 2003, DERBI 224603 03
Buenos Aires, Argentina, 2003 (Kennebec)	WP	0.29	0.097	300	4	12	< 0.05	Kalvan, 2003, DERBI 224603 03
Buenos Aires, Argentina, 2002 (Kennebec)	WP	0.12	0.034	340	1	40	< 0.05	Kalvan, 2003, DERBI 224603 03
Buenos Aires, Argentina, 2002 (Kennebec)	WP	0.15	0.043	340	1	40	< 0.05	Kalvan, 2003, DERBI 224603 03
Buenos Aires, Argentina, 2002 (Kennebec)	WP	0.29	0.086	340	1	40	< 0.05	Kalvan, 2003, DERBI 224603 03A
<i>GAP, Brazil</i>	<i>WP</i>	<i>0.10-0.13</i>		<i>650</i>	<i>NS</i>	<i>7</i>		
Brazil, 2002 (Asterix)	WP	0.14		600	8	7	ND, ND	Oliveira, 2002, DERBI 102061
Brazil, 2002 (Asterix)	WP	0.27		600	8	7	ND, ND	Oliveira, 2002, DERBI 102061
Brazil, 1998 (Jaette Bintje)	WP	0.14		650	8	0 3 7 10 14	< 0.01 < 0.01 <u>< 0.01</u> < 0.01 < 0.01	Tornisielo, 1999, DERBI 221880 BR98F01A
Brazil, 1998 (Jaette Bintje)	WP	0.27		650	8	7	< 0.01	Tornisielo, 1999, DERBI 221880 BR98F01A
Brazil, 1998 (Jaette Bintje)	WP	0.14		625	8	7	<u>< 0.01</u>	Tornisielo, 1999, DERBI 221884 BR98F01A
Brazil, 1998 (Jaette Bintje)	WP	0.27		625	8	7	< 0.01	Tornisielo, 1999, DERBI 221884 BR98F01A
Brazil, 1999 (Bintje)	WG	0.13		648	6	0 7 14 21 28	0.07 <u>0.02</u> 0.01 < 0.01 < 0.01	Tornisielo, 2000, DERBI 204328 3179905
Brazil, 1999 (Bintje)	WG	0.27		648	6	7	0.015	Tornisielo, 2000, DERBI 204328 3179905
Brazil, 1999 (Bintje)	WG	0.13		600	6	7	<u>0.02</u>	Tornisielo, 2000, DERBI 224107 31799056
Brazil, 1999 (Bintje)	WG	0.27		600	6	7	0.04	Tornisielo, 2000, DERBI 224107 31799056
Brazil, 2003 (Monalisa)	WG	0.13		600	3	7	ND, ND	Oliveira, 2003, DERBI 135351
Brazil, 2003 (Monalisa)	WG	0.27		600	3	7	ND, ND	Oliveira, 2003, DERBI 135351

Supervised trials in the Republic of Korea

Six trials were conducted using an SC formulation containing 8% zoxamide.

Table 45. Zoxamide residues in potato from supervised trials in the Republic of Korea

Potato country, year (variety)	Application					PHI Days	Residues mg/kg	Reference
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.			
GAP, Rep. Korea	WG		0.008		4	14		
Rep. Korea, 1997 (Sumi)	SC	0.32	0.016	2000	3	30 30 30	0.025 0.025 0.031	DERBI 239922
Rep. Korea, 1997 (Sumi)	SC	0.32	0.016	2000	3	21 21 21	0.035 < 0.025 0.025	DERBI 239922
Rep. Korea, 1997 (Sumi)	SC	0.32	0.016	2000	4	21 21 21	0.055 0.035 0.035	DERBI 239922
Rep. Korea, 1997 (Sumi)	SC	0.32	0.016	2000	4	14 14 14	0.035 0.035 0.035	DERBI 239922
Rep. Korea, 1997 (Sumi)	SC	0.32	0.016	2000	5	14 14 14	0.21 0.19 0.19	DERBI 239922
Rep. Korea, 1997 (Sumi)	SC	0.32	0.016	2000	5	7 7 7	0.11 0.13 0.11	DERBI 239922

FATE OF RESIDUES IN STORAGE AND PROCESSING

In Storage

No information was provided to the Meeting as zoxamide is not used in stored products.

In Processing

The Meeting received information on processing of grapes to juice, wine and pomace, tomatoes to puree and paste, and potatoes into flakes, chips and peel.

Grapes

Effects on the nature of the residue

In a vinification study [¹⁴C-phenyl]zoxamide (radiochemical purity 97.8%) in acetone was sprayed uniformly onto bunches of fresh white or red grapes in a glass aquarium at the rate of 3 mg ¹⁴C-zoxamide per kg grapes (Mamouni, 1998; DERBI 92294). The grapes were then processed according to commercial practices. Six types of wine were prepared. In addition, three control wines (white wine III and VI and red wine VIII) were prepared from grapes treated only with acetone.

Samples of the must were taken before fermentation and at the end of fermentation. Wine samples were taken after 2, 4, and 8 months of storage at 12 °C. Radioactive residues were determined by LSC after grapes and debris were homogenized and combusted.

The nature of the residue was determined by HPLC analysis using a C18 column with ¹⁴C and/or UV detection. Selected results were confirmed by TLC. The distribution of radioactivity through the pressing step for each wine is shown in Table 46. The radioactive residues in must and wine, in addition to zoxamide and its degradation product, RH-150721, are shown in Table 47 of ten other compounds were found in the HPLC. However, none comprised more than 3.8% of the total applied radioactivity at any time point in any sample. During fermentation, very low amounts of radioactive carbon dioxide were detected (< 0.1 % of the initial dose). No radioactive residues were detected in the control musts.

Table 46. Ratio of recovered radioactivity in wine-making

Wine Sample		Radioactivity recovered (% of applied dose)					
		Fresh juice		Air-dried marc	Washings of aquarium and applicator	Total	
White wine	I & II	49.6		32.2		8.5	90.2
	IX	36.3		45.2		11.38	92.8
Rosé wine	V	25.0		58.2		5.11	88.3
Red wine	VI	20.8		58.9		12.17	91.9
	VII	17.1		69.6		2.0	89.2

Table 47. Radioactive residues in must and wine from treated grapes

Wine	Residue	Fresh juice				End of fermentation		Stored wine					
		before filtration		filtered				2months		4 months		8 months	
		mg/L	%	mg/L	%	mg/L	%	mg/L	%	mg/L	%	mg/L	%
White Wine I	TRR	2.3	49.4	0.93	19.9	1.6	33.6	1.4	29.8	1.46	31.4	1.4	29.6
	Zoxamide	-	-	0.82	17.5	0.62	13.3	0.35	7.6	0.27	5.7	0.12	2.7
	RH-150721	-	-	-	-	0.65	13.9	0.70	15.1	0.83	17.8	0.90	19.4
White Wine II	TRR	2.3	49.4	0.93	19.9	1.4	30.8	1.4	29.3	1.3	28.5	1.3	27.6
	Zoxamide	-	-	0.82	17.5	0.64	13.8	0.40	8.7	0.31	6.6	0.14	3.0
	RH-150721	-	-	-	-	0.45	9.8	0.59	12.7	0.66	14.1	0.79	16.9
White Wine IX	TRR	1.7	36.3	0.77	16.4	1.1	24.1	1.13	24.1	1.2	24.6	1.1	23.1
	Zoxamide	-	-	0.67	14.2	0.51	10.9	0.48	10.1	0.29	6.3	0.12	2.6
	RH-150721	-	-	-	-	0.32	6.9	0.49	10.5	0.49	10.4	0.54	11.5
Rose Wine V	TRR	1.1	25.0	-	-	0.62	13.7	0.62	13.9	0.66	14.6	0.64	14.3
	Zoxamide	0.96	21.3	-	-	0.24	5.4	0.14	3.2	0.11	2.4	0.077	1.7
	RH-150721	-	-	-	-	0.25	5.6	0.33	7.4	0.37	8.2	0.40	8.9
Red Wine VI	TRR	0.90	20.8	0.039	9.1	0.55	12.8	0.53	12.2	0.50	11.6	0.42	9.7
	Zoxamide	0.70	16.1	0.13	3.0	0.14	3.2	0.08	1.9	0.03	0.6	-	-
	RH-150721	0.029	0.7	0.094	2.2	0.27	6.2	0.20	4.7	0.27	6.2	0.13	3.1
Red Wine VII	TRR	0.78	17.1	0.373	8.2	0.49	10.7	0.53	11.5	0.53	11.6	0.40	8.8
	Zoxamide	0.55	12.0	0.125	2.8	0.10	2.3	0.06	1.4	0.03	0.7	-	-
	RH-150721	0.036	0.8	0.139	3.1	0.23	5.0	0.27	5.9	0.23	5.0	0.17	3.8

The total radioactive residue at the end of fermentation was 24 – 34% of the applied activity in the white wines and 11 – 14% of the applied activity in the red wines, with zoxamide and RH-150721 at roughly equal concentrations. Total residues declined slightly during ageing (approximately 10%, average). Zoxamide declined by 69 – 100% (average 84%) over 8 months. In general, RH-150721 increased in concentration over the aging period. However, in red wines where concentrations of zoxamide were low at the end of the fermentation, residues of RH-150721 began to decline toward the end of the aging period.

Clarifying the wine by Bentonite and centrifugation removed significant amounts of residues (up to 49% of the residues in white wines and 31% of the residues in red wines).

Effects on residue levels

Grape juice and dried grapes: In US supervised field trials, grapes were treated with zoxamide 80W formulation. Ten applications were made at rates of either 0.14 or 0.28 kg ai/ha (Graves, 1998; DERBI 92215). Grapes were harvested 14 days after the final application. Samples from one site in California were either processed into juice on the day of harvest or processed into dried grapes over the following few weeks. Processing was in accordance with commercial practice. Processed fractions were frozen until required for analysis.

Table 48. Mean residue levels in grapes and processed products

Commodity analyzed	0.14 kg ai/ha		0.28 kg/ha		Average PF
	14 day PHI		14 day PHI		
	mg/kg	PF ^a	mg/kg	PF	
Fruit (RAC)	0.31	-	0.39	-	-
Unclarified juice	0.050	0.16	0.039	0.10	0.13
Clarified juice	0.014	0.05	0.021	0.05	0.05
Dried grapes	0.70	2.2	1.4	3.5	2.9

a - PF = Processing factor.

Wine: Grapes from twelve residue trials conducted in Europe in 1996 and 1997 were vinified. Samples of must and pomace processed fractions were analyzed for zoxamide. Wine samples were analyzed for zoxamide and the metabolite RH-0721. Wine samples were analyzed as the end of fermentation and after 6 months to 1 year's ageing. .

Table 49. Summary of residues in grapes and wine from European processing trials

Country Variety (Wine type) Year	Application					PHI (days)	Portion analyzed	Residue (mg/kg)		Ref.
	Form	kg ai/ha	kg ai/hL	water L/ha	No.			zoxamide (PF ^a)	RH-0721	
Germany Riesling (White) 1996	WG		0.015	600 1300	-6	56	Grapes Must Young wine Aged wine	0.55 0.16 (0.29) < 0.01 (< 0.02) < 0.01 (< 0.02)	< 0.01 0.02	Wais, 1999, DERBI 221867
Germany Burgunder (Red) 1996	WG		0.015	600 1300	-6	56	Grapes Must Young wine Aged wine	0.41 0.02 (0.05) < 0.01 (< 0.02) < 0.01 (< 0.02)	< 0.01 0.01	Wais, 1999, DERBI 221867
Germany Riesling 1997	WG		0.015	400 2000	-6	56	Grapes Pomace (wet) Must Wine	0.34 0.52 (1.53) 0.08 (0.24) < 0.01 (< 0.03)	0.03	Wais, 1999, DERBI 221863
Germany Burgunder 1997	WG		0.015	400- 2000	6	56	Grapes Pomace (wet) Must Wine	0.66 2.04 (3.09) 0.06 (0.09) < 0.01 (< 0.02)	0.02	Wais, 1999, DERBI 221863
France (N) Meunier 1996	WG	0.15	(0.043)	350	10	28	Grapes Must Pomace (wet) Pomace (dry) Young wine Aged wine	1.44 0.26 (0.18) 0.02 (0.01) 0.02 (0.01) < 0.01 (< 0.01) < 0.01 (< 0.01)	0.03 0.02	Grolleau, 1999, DERBI 220177
France (N) Chenin 1997	WG ^b	0.125	(0.025)	500	10	28	Grapes Must Pomace (wet) Pomace (dry) Young wine Aged wine	0.73 0.21 (0.29) 0.04 (0.05) 0.05 (0.07) < 0.01 (< 0.01) < 0.01 (< 0.01)	< 0.01 0.07	Grolleau, 1999, DERBI 146039
France (S) Cabernet Sauvignon 1996	WG	0.15	(0.043)	350	10	28	Grapes Must Pomace (wet) Pomace (dry) Young wine 6 month wine 25 month wine	3.06 1.66 (0.54) 0.07 (0.02) 0.28 (0.09) 0.07 (0.02) 0.03 (0.01) < 0.01 (< 0.01)	0.49 0.38 0.49	Grolleau, 1999, DERBI 220177

Country Variety (Wine type) Year	Application					PHI (days)	Portion analyzed	Residue (mg/kg)		Ref.
	Form	kg ai/ha	kg ai/hL	water L/ha	No.			zoxamide (PF ^a)	RH-0721	
France (S) Clairette Blanche 1996	WG	0.15	(0.043)	350	10	28	Grapes Must Pomace (wet) Pomace (dry) Young wine 7 month wine 20 month wine	1.45 0.49 (0.34) 1.64 (1.13) 1.42 (0.98) 0.06 (0.04) 0.03 (0.02) < 0.01 (< 0.01)	0.17 0.14 0.19	Grolleau, 1999, DERBI 220177
France (S) Merlot 1997	WG ^b	0.125	(0.025)	500	10	35	Grapes Must Pomace (wet) (Pomace (dry) Young wine Aged wine	0.61 0.44 (0.72) 0.03 (0.05) 0.19 (0.31) < 0.01 (< 0.02) < 0.01 (< 0.02)	0.03 0.06	Grolleau, 1999, DERBI 146039
France (S) Grenache 1997	WG ^b	0.125	(0.025)	500	10	35	Grapes Must Pomace (wet) (Pomace (dry) Young wine Aged wine	0.23 0.08 (0.35) 0.03 (0.13) < 0.01 (< 0.04) < 0.01 (< 0.04) < 0.01 (< 0.04)	0.04 0.03	Grolleau, 1999, DERBI 146039
Italy Cabernet Sauvignon 1996	WG	0.15	0.015	1000	10	28	Grapes Wine before malo-lactic fermentation	0.48 < 0.01 (< 0.02) < 0.01 (< 0.02) < 0.01 (< 0.02)	0.01 0.02 0.03	Wais, 1999, DERBI 221861
Italy Sauvignon 1997	WG	0.15	0.015	1000	10		Grapes Must Pomace (wet) Young wine Aged wine	0.33 0.04 (0.12) 0.26 (0.79) < 0.01 (< 0.03) < 0.01 (< 0.03)	0.05 0.04	Wais, 1999, DERBI 221864

a - PF = Processing factor.

b - zoxamide/mz 75.25 WG

The median concentration factors for the above trials are 0.29 for must, and < 0.02 for wine (young and aged).

Tomatoes

Effects on the nature of the residue

A radiochemical mass balance and identification of degradates were determined in processed tomato products made from tomatoes treated with ¹⁴C-zoxamide (Völkl; 2000, DERBI 92297). Aliquots from the application solution in acetone were spotted onto each tomato separately and distributed on the surface using a syringe. The tomatoes were treated at a rate of 3 mg ¹⁴C-zoxamide per 1 kg tomato. After evaporation of the acetone the tomatoes were processed to either juice, puree or preserved tomatoes.

The amount of radioactive residues after processing was determined directly by liquid scintillation counting in all liquid samples and in solid fractions after combustion. Additionally, the samples were analyzed by HPLC/RAM for parent substance and degradates.

Juice: Based on the total radioactivity applied, the level of residues in the juice amounted to 13.0% of the initial dose or 0.393 mg parent equivalents/kg tomato. In the centrifuged tomato juice 4.7% (0.142 ppm) of the radioactivity applied were recovered. In the pulp which had been separated from the juice, the residues represented 8.3% of the radioactivity applied or 0.250 mg parent equivalents/kg tomato. Most of the radioactivity from the residue could be extracted from the pulp with acetone (i.e., 7.7% of the applied radioactivity, 0.232 ppm).

Puree: The radioactivity amounted to 26.2% of the initial dose or 0.778 mg parent equivalents/kg tomato. The remaining radioactivity (in total 57.8% or 1.715 ppm) was detected in

skins, seeds and cooking water that were separated from the puree. Most of the radioactivity from puree was extracted with acetone (i.e., 23% of the radioactivity applied, 0.683 ppm). Only 3.2% of the radioactivity applied (0.095 ppm) remained unextracted.

Preserved tomatoes: Based on the total radioactivity applied, the level of residues in the preserved tomatoes amounted to only 1.9% of the initial dose or 0.028 mg parent equivalents/kg tomato. Most of the radioactivity was in the separated skins representing 87.0% of the radioactivity applied (2.635 ppm) and in the cooking water amounting to 9.2% (0.279 ppm). Most of the radioactivity applied (0.026 ppm). Only 0.2% of the radioactivity applied (0.002 ppm) remained unextracted.

Degradation pattern: For juice, zoxamide was the main radioactive component recovered amounting to 12.2% (0.369 ppm) of the radioactivity applied. No HPLC was performed for the preserved tomatoes due to the low amount of radioactivity recovered in the extracts (1.8%). Similarly, parent compound was the main radioactive fraction in the extracted skin of the preserved tomatoes and was recovered without significant degradation due to the fact that in the early stages of processing the skin was separated from the remaining tomatoes. However, in the puree where the whole tomatoes were cooked for 20-30 minutes the parent compound was degraded mainly to RH-129151 and RH-150721. The parent compound amounted to 3.1% (0.092 ppm) of the radioactivity applied. The main degradation products reached levels of 10.9% (0.324 ppm) of the radioactivity applied and 6.7% (0.199 ppm), respectively. In addition, four minor fractions were detected not exceeding 1.4% (0.040 ppm) of the radioactivity applied.

Effects on residue levels

A processing trial was included in the trial study. The trial made 10 applications of zoxamide at 0.225 kg ai/ha at 7 – 10 day intervals. Tomatoes (~180 kg for each control and treated sample) were collected 5 days after the last application. They were stored overnight and processed the next day. The samples were then frozen until they were analyzed 8 – 12 months later. The results are contained in Table 50.

Table 50. Summary of residues in tomato and its processed products

	Zoxamide	Processing factor
Tomato used for processing	0.0939	
Tomato puree	0.0408	0.43
Tomato paste	0.0908	0.97

There was no concentration of zoxamide residues in either the puree or paste compared to the unwashed fruit. The processing factor is $0.0408/0.0939=0.43$ for the puree and $0.0908/0.0939=0.97$ for tomato paste.

Potatoes

A processing trial was included in the trial study. The trial made 10 applications of zoxamide at an exaggerated rate of 1.1 kg ai/ha at 7 – 10 day intervals. Potatoes were collected 3 days after the last application. They were stored at 7 °C until processed into chips and flakes. The samples were then frozen until they were analyzed ~12 months later.

Samples were analyzed for residues of zoxamide and two metabolites, RH-1452 and RH-1455 using method TR 34-98-140 (Desai, 1998; DERBI 91465). No quantifiable residues of zoxamide were found in the RAC samples from the trial with the high application rate. So the chips and flakes were not initially analyzed for zoxamide. They were later analyzed for zoxamide; no detectable residues of the parent were found (Graves, 2000; DERBI 91485).

After the initial report for this study was issued, potato peel samples stored frozen from the initial processing were analyzed for residues of zoxamide and the metabolites (Graves, 2000; DERBI 92638).

Table 51. Summary of residues in potato and its processed products and by-product

	Zoxamide	RH-1452	RH-1455
Potato	< 0.02	0.021	0.016
Flakes	ND	0.12	0.056
Chips	ND	0.026	0.020
Peel	0.060	ND	0.015
Processing Factor (Flakes) =	-	5.8	3.4
Processing Factor (Chips) =	-	1.2	1.3
Processing Factor (Peel) =	>3.0	< 0.29	0.94

NA = Not applicable, sample was not analyzed.

ND = Not detected, less than the 0.006 mg/kg.

Note: residues are from a trial with an exaggerated rate. No quantifiable zoxamide residues were found.

When the metabolite residues found in the processing trial are adjusted to a 1× rate they would be below the method LOQ of 0.02 mg/kg.

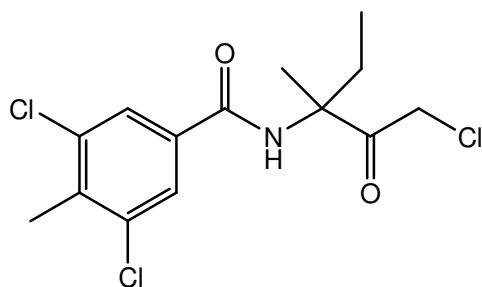
RESIDUES IN ANIMAL COMMODITIES

Zoxamide is intended for use on cucurbits, grapes, potatoes and tomatoes. Wet grape pomace and potato culls or processed potato waste (wet peel) may be fed to cattle and poultry. However, they are not among major contributors to animal burden. Furthermore, on the basis of the goat metabolism study using the dose rate equivalent to 60.7 ppm in the diet, significant residues (≥ 0.01 mg/kg) are unlikely to be present in any edible animal tissue or milk, taking into account the low livestock dietary burden calculated using zoxamide residues in wet grape pomace and potato peel from trials conducted in accordance with the relevant GAP.

No feeding study was provided on ruminants or poultry.

APPRAISAL – RESIDUE AND ANALYTICAL ASPECTS

Zoxamide, a benzamide fungicide, was identified as a priority new compound at the 38th Session of the CCPR (ALINORM 06/29/24) for evaluation by the 2007 JMPR. The Meeting received information on physical and chemical properties, animal and plant metabolism, environmental fate, analytical methods, storage stability, use patterns, supervised trials and processing.



(*RS*)-3,5-dichloro-*N*-(3-chloro-1-ethyl-1-methyl-2-oxopropyl)-4-methylbenzamide

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

RH-127450	3,5-dichloro- <i>N</i> -(1-ethyl-1-methyl-2-oxopropyl)-4-methylbenzamide
RH-129151	2-(3,5-dichloro-4-methylphenyl)-4-ethyl-4-methyl-4H-1,3-oxazin-5(6H)-one
RH-139432	3,5-dichloro-4-methylbenzamide

RH-141288	3,5-dichloro- <i>N</i> -(3-hydroxy-1-ethyl-1-methyl-2-oxopropyl)-4-methylbenzamide
RH-1452	3,5-dichloro-4-hydroxymethylbenzoic acid
RH-1455	3,5-dichloro-1,4-benzene-dicarboxylic acid
RH-149736	3,5-dichloro-4-hydroxymethylbenzamide
RH-149737	4-carboxy-3,5-dichlorobenzamide
RH-150721	(3-amino-3-methyl-2-oxo)pentyl-(3,5-dichloro-4-methyl)benzoate
RH-163353	3,5-dichloro- <i>N</i> -(2-carboxy-1-ethyl-1-methyl-2-oxoethyl)-4-methylbenzamide
RH-24549	3,5-dichloro-4-methylbenzoic acid

Animal metabolism

The Meeting received information on the fate of orally-dosed zoxamide in a lactating goat.

When [U-¹⁴C-phenyl]zoxamide was administered orally at a dose equivalent to a dietary concentration of 60.7 ppm to a lactating goat once a day for 7 consecutive days, 95% of the recovered radioactivity (77.5% of the administered dose) was found in urine (37.1%) and faeces (36.1%). None of individual tissues or cumulative milk sample on day 7 contained more than 3% of the administered dose. On day 4 the radioactive residues in milk was the highest at 0.24 mg/kg in parent equivalents.

Unextracted radioactivity was less than 10% of total radioactive residues (TRR) (< 0.05 mg/kg) in all samples except liver (12%).

No parent compound was found in any of tissues or milk sample. A number of metabolites were detected in milk and tissues. In fat, RH-127450 was found at 0.13 mg/kg in parent equivalent. However, as the dose administered in the study was about 14 times the highest concentration found in any commodity after treatment of the respective crop in accordance with GAP, significant residue concentrations are unlikely to occur in milk or any tissues in practice.

Zoxamide was extensively metabolized and readily eliminated following oral administration to a lactating goat. Once administered orally, zoxamide underwent dechlorination, then oxidation of either position 4 of the benzene ring or the end of the side-chain and further hydrolysis.

The metabolism of zoxamide in the lactating goat was qualitatively similar to that described in the toxicology section of the 2007 Report of the JMPR (see page 280).

Plant metabolism

The Meeting received information on the fate of zoxamide after foliar application of [U-¹⁴C-phenyl]zoxamide to grapes, cucumber, tomato and potato.

When grape vines were sprayed at a rate of 1.9 kg ai/ha three times at 30 day intervals, grapes harvested 1 day after the last application contained 0.74 mg/kg of radioactive residues. The parent compound was the major residue at 0.43 mg/kg (58% of TRR). RH-129151, RH-139432, RH-141288, RH-149736, RH-149737 and RH-150721 were identified but all were less than 0.021 mg/kg in parent equivalents (\leq 2.8% of TRR).

Cucumber plants were sprayed three times at the rate of 1.3 kg ai/ha at a 7 day intervals and foliage and fruit samples were harvested 1 day after the last application. While an average radioactive residue in foliage was 108 mg/kg in parent equivalent, that in fruits was 1.5 mg/kg, which indicates that translocation of zoxamide, was not significant one day after the final application. Extraction of foliage and fruit samples with acetonitrile-water mixture solubilised 100% of the total radioactivity and there were no volatile or unextracted residues. Zoxamide accounted for 87% of TRR in fruits and 89% in foliage indicating that the parent is predominant. Minor metabolites were identified in fruits

and foliage. Among them, RH-150721 and RH-157450 were present at the highest concentrations but still less than 0.1 mg/kg (< 5% of TRR).

Tomato plants received three foliar applications at 0.86 mg/kg with 18 day intervals and tomato fruits were collected 1 day after the last application. The TRR was 0.29 mg/kg in green tomato and 0.50 mg/kg in red tomato. The parent was the major component of residues amounting to 0.14 mg/kg (48% of TRR) in green tomato and 0.22 mg/kg (44% of TRR) in red tomato. Minor amounts of metabolites were identified but none exceeded 3% of TRR. RH-1452 and RH-141288 were identified in two different fractions but their actual concentrations were not determined.

Three foliar applications were made at the rate of 0.9 kg ai/ha on potato plants with the first application at 39 days after planting, and the second and third made at intervals of 21 and 17 days respectively. Mature potato tubers were harvested 14 days after the last application. The TRR was 0.18 mg/kg parent equivalents. Unlike other plants tested, the parent compound was not found in the harvested commodity, i.e., the potato tuber. The metabolites RH-1455 and RH-1452 were found at 0.069 and 0.037 mg/kg accounting for 39% and 21% of the TRR, respectively.

The nature of minor metabolites suggests that zoxamide, when applied to plants, underwent dechlorination and hydrolysis or oxidation. Zoxamide was the major residue in grape, cucumber and tomato when harvested one day after the last application. However, the parent compound was not found in potato sampled 14 days after the last application.

Environmental fate in soil

The Meeting reviewed information on aerobic soil metabolism and rotational crop study as zoxamide was intended for protection of potatoes.

Aerobic soil metabolism studies were conducted using [U-¹⁴C-phenyl]zoxamide applied to various soils which were then incubated under aerobic conditions at 20 or 25 °C. Under aerobic conditions, zoxamide applied to soil was rapidly degraded. After 120 – 122 days, only small amounts (0.6 – 10%) of applied zoxamide remained as the parent. Carbon dioxide was steadily evolved from all soils and accounted for 34 – 58% of the dose applied after 120 – 122 days. RH-127450, RH-129151, RH-24549, RH-139432 and RH-163353 were formed and then degraded during the study periods. Unextracted radioactivity, 0.4-3.3% of the applied dose (3.3% in silt loam dosed at 1.5 mg/kg; for other soils tested 0.4 – 0.8%) on day 0, increased steadily to reach 24 – 38% of the applied dose on day 120 – 122. Several other degradates were observed at very low concentrations. These results indicate that none of zoxamide or its identified metabolites are persistent in soil.

Residues in succeeding crops

In an outdoor confined rotation study, mustard, radish, turnip, sorghum and soya bean were planted at 30, 137, 210, 365 days following the last of four applications of [¹⁴C-phenyl]zoxamide. Zoxamide was applied to bare soil between mid April and early June (18 day intervals) at a rate of 0.5 kg ai/ha. Crops were harvested at an intermediate stage and when mature.

TRRs were very low for all samples at all plant back intervals. In general, the amount of extractable residues was low in all the crop samples. Between 7% and 40% of the TRR was recovered in the polar MeOH/H₂O fractions for all the crops grown on treated soil. About 2 to 36% of the TRR was found in the organic extracts (CHCl₃, CH₃CN and hexane) of all the crops. The concentrations in these samples did not exceed 0.023 mg/kg. The values of extracts for all the crop samples showed a significant fraction of unextracted residues: generally 49% or greater.

Concentrations of RH-1452 and one other metabolite were generally below 0.01 mg/kg. The second metabolite was not fully identified. Other metabolites were detected at lower concentrations in some crops.

Zoxamide residues are not expected to occur in succeeding crops.

Methods of analysis

Analytical methods for determination of residues of zoxamide were developed for a wide range of matrices including cucurbits, grapes, tomato, potato and their processed commodities and byproducts.

In most of the methods for determination of zoxamide only, zoxamide was extracted with organic solvent or a mixture of organic solvent and aqueous solution specific to the matrix; cleaned up with liquid-liquid partition followed by solid phase extraction using carbon, alumina, Florisil and silica singly or in combination; and analysed by gas chromatography using electron capture detection (GC/ECD) for quantitation and mass selective detection (GC/MSD) for confirmation. For detection, ELCD or NPD may also be used. These methods were validated in independent laboratories. Most of the methods were suitable as enforcement methods with the limit of quantification at 0.01 mg/kg. One method for potato and its products has an LOQ of 0.02 mg/kg.

The existing multi-residue enforcement methods, one of FDA screen methods and DFG S19 were also tested to be suitable for analysing zoxamide.

The methods for potato and its processed commodities determine zoxamide and two metabolites, RH-1452 and RH-1455. While zoxamide is extracted in the organic phase in liquid-liquid partition, these metabolites were extracted in the aqueous phase. After methylation of these metabolites using diazomethane, and further clean-up, they were analysed using GC/ECD or GC/MSD. The LOQ was 0.02 mg/kg.

Stability of residues in stored analytical samples

Stability of zoxamide (0.1 – 2 mg/kg) in homogenized samples of grapes (433 days), cucumbers (868 days), tomatoes (810 days), and potatoes (708 days); grape juice (858 days); dried grapes (789 days); wine (8 months); tomato juice (832 days); tomato paste (237 days); and tomato puree (228 days) stored in deep freezer at a temperature below -10 °C was investigated.

No decrease of zoxamide was observed in all samples of cucumbers, tomatoes and its processed products and potatoes during the test periods.

In the case of grapes and its products, in particular grape juice, relatively large fluctuations were observed in the percentage of remaining zoxamide during the test period. However, the Meeting concluded that zoxamide was sufficiently stable for 14 months in grapes, 28 months in grape juice, 26 months in dried grapes and 8 months in wine.

RH-1452 and RH-1455 were shown to be stable for 29 months of storage while frozen.

Definition of the residue

In grapes, zoxamide represented 58% of the TRR with no metabolite exceeding 5% of the TRR. Also in cucumber and tomato, zoxamide is the major residue component: 87% of TRR in cucumber, 48% of TRR in green tomatoes and 44% of TRR in red tomatoes. No metabolite was found to be more than 10% of the TRR in all cucumber and tomato samples. Most metabolite residues were present at less than 5% of the total residues. These indicate that the residue of concern in grapes, cucurbit and tomato be defined as parent although samples analysed were taken only one day after the last application.

In potato, however, no parent zoxamide was detected. RH-1452 and RH-1455, comprising 21% and 39% of the total residue, respectively, were the major components of the residue. Another 16% of the residue was identified as glucose and/or other sugars. No other metabolites were present at or higher than 10% of TRR. In supervised field trials in Northern and Southern Europe, the United States, Canada and Mexico, samples were analysed for zoxamide, RH-1452 and RH-1455. In all trials, residues of parent were below the LOQ and concentrations of the metabolites were also below the LOQ in all but two trials where zoxamide was found at 0.02 mg/kg.

Methods of analysis are available for determination of zoxamide in grapes, cucurbits, tomatoes and potatoes and their processed products. A method is available also for determination of RH-1452 and RH-1455 in potatoes.

The current Meeting concluded that only zoxamide is toxicologically significant.

In the lactating goat study, the main components of residues were RH-127450 in milk and fat, glucuronic acid conjugates of 4-hydroxymethyl-RH-141288 in liver, with the highest concentration of 0.13 mg/kg parent equivalents of RH-12740 in liver. However, as the administered dose was 14 times higher than the highest residue concentration found in the reported trials, no residue was expected to be found in animals given feed with incurred residues of zoxamide. No method of analysis is currently available for these metabolites. For these reasons, the Meeting concluded that it was not in a position to recommend a residue definition for animal commodities.

In the lactating goat study, the concentration of radioactive residues expressed in parent equivalent in fat was about 4 times that in muscle but about one half of that in kidney or liver. Therefore, the Meeting considered residues not fat-soluble.

In countries where there are MRLs for zoxamide, the residue definition was mostly “zoxamide” except in the USA where it is zoxamide including its metabolites RH-1452 and RH-1455 for potato and its products.

The Meeting recommended the following residue definition for zoxamide in plant commodities.

For plants: Definition of the residue (for compliance with the MRL and for estimation of dietary intake): zoxamide

Results of supervised residue trials on crops

The Meeting received supervised trial data for zoxamide uses on grapes, cucurbits, tomato and potato.

Grapes

Numerous residue trials were conducted on grapes in Brazil, Canada, Germany, France, Greece, Italy, Republic of Korea, Spain and the USA.

The trials conducted in Germany used six applications rather than four as on the label. The Meeting decided to use the results of these trials for MRL estimation as the last applications contribute most to the residue concentration at harvest. In 12 German trials in accordance with German GAP (maximum rate of 0.24 kg ai/ha in 800 – 1600 L/ha, 4 applications, with a PHI of 56 days) (except application number), zoxamide residues in rank order were: 0.34, 0.38, 0.39, 0.41, 0.41, 0.45, 0.49, 0.55, 0.59, 0.60, 0.66 and 0.72 mg/kg.

The trials conducted in France used ten applications rather than three on the label. The Meeting decided to use the results of these trials for MRL estimation as it is the last applications that contribute the most to the residue concentration at the harvest. In 21 Northern French trials in accordance with French GAP (0.12 kg ai/ha, 3 applications, PHI 28 days)(except application number), zoxamide residues in rank order were: 0.09, 0.17, 0.19, 0.19, 0.33, 0.35, 0.45, 0.47, 0.48, 0.50, 0.50, 0.51, 0.55, 0.56, 0.67, 0.77, 0.77, 0.81, 0.88, 1.31, 1.55 mg/kg. In 15 Southern French trials conducted in accordance with French GAP, zoxamide residues in rank order were: 0.21, 0.21, 0.33, 0.42, 0.42, 0.46, 0.49, 0.54, 0.58, 0.61, 0.63, 1.07, 1.11, 1.53 and 2.84 mg/kg. Since the residue populations in the Northern and Southern France are similar and there is a uniform GAP for the whole of France, the Meeting considered it appropriate to combine the results from 36 trials in France: 0.09, 0.17, 0.19, 0.19, 0.21, 0.21, 0.33, 0.33, 0.35, 0.42, 0.42, 0.45, 0.46, 0.47, 0.48, 0.49, 0.50, 0.50, 0.51, 0.54, 0.55, 0.56, 0.58, 0.61, 0.63, 0.67, 0.77, 0.77, 0.81, 0.88, 1.07, 1.11, 1.31, 1.53, 1.55 and 2.84 mg/kg.

In 15 Italian trials conducted in accordance with Italian GAP (maximum rate of 0.17 kg ai/ha, 0.017 kg ai/hL, 5 applications, PHI 28 days), zoxamide residues in rank order were: 0.24, 0.28, 0.29, 0.30, 0.33, 0.48, 0.48, 0.54, 0.59, 0.65, 0.66, 0.81, 0.82, 1.37 and 1.56 mg/kg.

In six Spanish trials conducted in accordance with Italian GAP, zoxamide residues in rank order were: 0.36, 0.53, 1.17, 1.21, 1.42 and 1.92 mg/kg.

In four Greek trials conducted in accordance with Italian GAP, zoxamide residues in rank order were: 0.27, 0.32, 0.34 and 0.64 mg/kg.

Combined residues from Italian, Spanish and Greek trials in accordance with Italian GAP in rank order were: 0.24, 0.27, 0.28, 0.29, 0.30, 0.32, 0.33, 0.34, 0.36, 0.48, 0.48, 0.53, 0.54, 0.59, 0.64, 0.65, 0.66, 0.81, 0.82, 1.17, 1.21, 1.37, 1.42, 1.56 and 1.92 mg/kg.

Six trials were conducted in Canada but none was in accordance with Canadian GAP (0.19 kg ai/ha, 6 applications, PHI 66 days). However, four trials were in accordance with US GAP (maximum rate of 0.22 kg ai/ha, 8 applications, PHI 14 days). Residues in rank order were: 1.12, 1.46, 1.52 and 1.69 mg/kg.

Among numerous US trials, 17 trials were conducted in accordance with US GAP. Zoxamide residues in rank order were: 0.22, 0.31, 0.34, 0.34, 0.42, 0.46, 0.49, 0.52, 0.61, 0.66, 0.83, 0.91, 1.08, 1.18, 1.61, 1.65 and 4.34 mg/kg.

Combined residues from the US and Canadian trials conducted in accordance with US GAP (ranked order, median underlined) were: 0.22, 0.31, 0.34, 0.34, 0.42, 0.46, 0.49, 0.52, 0.61, 0.66, 0.83, 0.91, 1.08, 1.12, 1.18, 1.46, 1.52, 1.61, 1.65, 1.69 and 4.34 mg/kg.

In seven Brazilian trials conducted in accordance with Brazilian GAP (maximum rate of 0.13 kg ai/ha, 600 – 1000 L/ha, PHI 7 days), zoxamide residues in rank order were: 0.07, 0.08, 0.14, 0.14, 0.15, 0.16 and 0.36 mg/kg.

Three trials conducted in the Republic of Korea seemed to be in accordance with Korean GAP (0.01 kg ai/ha, three applications, PHI 7 days). The residues were: 0.05, 0.06 and 0.08 mg/kg.

Among results of these trials, residues from US trials would lead to the highest maximum residue level. Based on the results from US and Canadian trials, the Meeting estimated a maximum residue level and an STMR for zoxamide in grapes of 5 and 0.83 mg/kg respectively.

Fruiting vegetables, cucurbits

Protected supervised trials were conducted on cucumber in France and Spain and field trials in the Republic of Korea and the USA. Supervised trials were also conducted in the USA for cantaloupe and squash.

Six supervised indoor trials on cucumber in France were in accordance with Polish GAP (maximum rate of 0.15 kg ai/ha, 700 – 800 L/ha, 3 applications, PHI 4 days) although five applications were made. Residues from these trials in rank order were: 0.01, 0.03, 0.04, 0.06, 0.06 and 0.48 mg/kg. In three Spanish trials conducted in accordance with Polish GAP, zoxamide residues in rank order were: 0.25, 0.44 and 0.45 mg/kg. Combined residues in rank order (median underlined) were: 0.01, 0.03, 0.04, 0.06, 0.06, 0.25, 0.44, 0.45 and 0.48 mg/kg.

Seven outdoor trials were conducted in the USA but only one trial was in accordance with the current US GAP for cucurbits (maximum rate of 0.19 kg ai/ha, 8 applications, PHI 5 days). The residues were: 0.04 mg/kg.

Four trials were conducted in the Republic of Korea on cucumber but none were in accordance with Korean GAP.

Seven trials on cantaloupe and six on summer squash were conducted in the USA but only one each was in accordance with the current US GAP. The residue level in one cantaloupe trial was 0.04 mg/kg and that in one squash trial was 0.09 mg/kg.

On the basis of indoor trials in Europe, the Meeting estimated a maximum residue level and an STMR for zoxamide in cucumber at 1 and 0.06 mg/kg respectively.

Tomato

Protected supervised trials were conducted on tomato in Greece and Spain; and field trials in Brazil, Italy, Spain and the USA.

In 10 Spanish indoor trials conducted in accordance with Italian GAP (maximum rate of 0.17 kg ai/ha, 0.017 kg ai/hL, 5 applications, PHI 3 days), zoxamide residues in rank order were: 0.07, 0.08, 0.09, 0.09, 0.10, 0.12, 0.12, 0.15, 0.24 and 0.29 mg/kg.

In two French indoor trials conducted in accordance with Italian GAP, zoxamide residues in rank order were: 0.28 and 0.31 mg/kg.

In three Greek indoor trials conducted in accordance with Italian GAP, zoxamide residues in rank order were: 0.15, 0.30 and 0.30 mg/kg.

Combined residues from the indoor trials in Spain, France and Greece in accordance with Italian GAP were: 0.07, 0.08, 0.09, 0.09, 0.10, 0.12, 0.12, 0.15, 0.15, 0.24, 0.28, 0.29, 0.30, 0.30 and 0.31 mg/kg.

In 12 Italian outdoor trials conducted in accordance with Italian GAP, zoxamide residues in rank order were: 0.12, 0.13, 0.14, 0.15, 0.16, 0.18, 0.18, 0.20, 0.22, 0.24, 0.24 and 0.30 mg/kg. In five Spanish outdoor trials conducted in accordance with Italian GAP, zoxamide residues in rank order were: 0.03, 0.04, 0.04, 0.05 and 0.05 mg/kg. Combined residues were: 0.03, 0.04, 0.04, 0.05, 0.05, 0.12, 0.13, 0.14, 0.15, 0.16, 0.18, 0.18, 0.20, 0.22, 0.24, 0.24 and 0.30 mg/kg.

Eighteen US outdoor trials were considered to have been conducted in accordance with US GAP (maximum rate of 0.19 kg ai/ha, 8 applications, PHI 5 days) although application number was mostly 10 up to 13 despite the label specification of 8 applications; however the Meeting concluded that the last applications contribute the most to residue concentrations at harvest. Zoxamide residues in ranked order (median underlined) were: 0.07, 0.10, 0.11, 0.12, 0.13, 0.16, 0.18, 0.18, 0.19, 0.20, 0.21, 0.21, 0.22, 0.23, 0.32, 0.38, 0.40 and 1.0 mg/kg.

In five Brazilian outdoor trials conducted in accordance with Brazilian GAP (maximum rate of 0.13 kg ai/ha, applied in 800 L/ha, with a PHI of 7 days), zoxamide residues in rank order were: 0.01, 0.02, 0.02, 0.03 and 0.14 mg/kg.

Among results from the above trials, those from US trials would lead to the highest maximum residue level. Based on the US data, the Meeting estimated a maximum residue level and an STMR for zoxamide in tomato of 2 and 0.195 mg/kg respectively.

Potato

Supervised trials were conducted on potato in Argentina, Brazil, Canada, France, Germany, Greece, Italy, Republic of Korea, Mexico, the Netherlands, Spain, the UK and the USA.

In six trials in Northern France, seven in Germany, one in the Netherlands and 11 from the UK conducted in accordance with GAP in Ireland, the Netherlands and the UK (maximum rate of 0.15 kg ai/ha, 200 – 600 L/ha, 10 applications, PHI 7 days), zoxamide residues were all < 0.02 mg/kg (25).

In six trials in Southern France, four in Greece, seven in Italy and six in Spain conducted in accordance with Italian GAP (maximum rate of 0.17 kg ai/ha (0.017 kg ai/hL), 5 applications, PHI 7 days), zoxamide residues were all < 0.02 mg/kg (23).

Twelve Canadian trials were considered to have been conducted in accordance with Canadian GAP (0.19 kg ai/ha, 6 applications, PHI 3 days) although 10 applications were made; however the Meeting concluded that the later applications contribute the most to residue concentrations at harvest. The residues were all < 0.02 mg/kg (12). A total of 27 USA trials were considered to be in compliance with US GAP (0.19 kg ai/ha, 6 applications, PHI 3 days) although 10 applications were made. The residues were all below the LOQ (0.02 mg/kg) (4) or LOD (0.006 mg/kg) (22) and hence < 0.02 mg/kg (27). Even with double rate applications, residues were below the LOQ.

Eight trials were conducted in Mexico but samples were taken 13 or 14 days after the last application instead of the PHI of 7 days as specified on the label.

In two Argentine trials, conducted in accordance with Argentine GAP (0.15 kg ai/ha, 400 – 1000 L/ha, PHI 7 days), zoxamide residues were < 0.05 mg/kg (2).

In six Brazilian trials, conducted in accordance with Brazilian GAP (maximum rate of 0.13 kg ai/ha, 650 L/ha, PHI 7 days), zoxamide residues (ranked order, median underlined) were: < 0.01 (4) (two were below the LOD) and 0.02 mg/kg (2).

Six trials were conducted in the Republic of Korea but none were in compliance with Korean GAP.

On the basis of the Brazilian trials and the fact that, other than two Brazilian trials, residues from trials done in accordance with respective GAP were all below the LOQ, the Meeting estimated a maximum residue level and an STMR at 0.02 and 0.02 mg/kg.

Fate of residues during processing

The Meeting received information on processing of grapes to dried grapes, juice, wine and pomace, tomatoes to puree and paste, and potatoes into flakes, chips and peel.

Processing factors were calculated for grapes (dried grapes, juice, wine and pomace), tomato (puree and paste) and potato (peel) and are shown in the Table below. Processing factors could not be calculated for potato flakes or chips because the residue concentrations were below the LOQ in both potatoes and processed products.

Mean processing factors and STMR-P for food and feed.

Commodity	Processing factor	Median or best estimate	STMR-P mg/kg
Grapes			0.83
Unclarified juice	0.10, 0.16	0.13	0.11
Dried grapes	2.2, 3.5	2.9	2.4
Wine	< 0.01, < 0.01, < 0.01, < 0.01, < 0.02, < 0.02, < 0.02, < 0.02, < 0.02, < 0.03, < 0.03, < 0.04	< 0.02	0.02
Pomace, wet	0.01, 0.02, 0.05, 0.05, 0.13, 0.79, 1.1, 1.5, 3.1	1.3 ^a	1.1
Tomato			0.195
Puree	0.43	0.43	0.08
Paste	0.97	0.97	0.19
Potato			0.02
Peel	> 3.0	3.0	0.06

a - As the spread of processing factors of wet pomace calculated from trial results is very large, the Meeting decided to take a conservative approach and use four values at the higher end to provide the best estimate of the processing factor.

Dried grapes are expected to contain higher residues than grapes. Multiplying the highest residue concentration found in the supervised trials (4.34 mg/kg) by the processing factor of 2.9, resulted in an estimate of 12.6 mg/kg, the Meeting estimated a maximum residue level at 15 mg/kg.

Residues in animal commodities

Potato wet peel and wet grape pomace may be fed to dairy cattle and beef cattle but not as major feed ingredients. The calculated maximum and mean livestock animal burden was 0.03 – 1.50 ppm.

In the metabolism study, in which zoxamide equivalent to 60.7 ppm in the diet was orally administered to a lactating goat for 7 consecutive days, no parent compound was found in any tissue or milk. A number of metabolites were present in tissues and milk but mostly below 0.1 mg/kg parent equivalents. Given the low estimated animal burden, about one fortieth of the administered level, no zoxamide or its metabolite is expected to be present at detectable levels in tissues or milk.

The Meeting agreed that no maximum residue level was necessary for commodities derived from mammals.

The livestock dietary burden was also calculated for layers and broilers with potato wet peel and wet grape pomace and were 0 – 0.73 ppm. No information was available on the fate of zoxamide in poultry. In addition, no method of analysis was submitted for zoxamide or metabolites in commodities derived from poultry.

The Meeting agreed that no maximum residue level could be estimated for commodities of poultry origin.

RECOMMENDATIONS

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

Plant commodities:

Definition of the residue (for compliance with the MRL and for estimation of dietary intake):
zoxamide

Commodity		Recommended MRL mg/kg		STMR/ STMR-P) mg/kg	HR/HR-P) mg/kg
CCN	Name	New	Previous		
VC 0424	Cucumber	1	-	0.06	
DF 0269	Dried grapes (=currants, raisins and sultanas)	15	-	2.4	
FB 0269	Grapes	5	-	0.83	
JF 0269	Grape juice, unclarified			0.11	
	Wine			0.02	
VR 0589	Potato	0.02	-	0.02	
VO 0448	Tomato	2	-	0.195	
	Tomato puree			0.08	
	Tomato paste			0.19	

DIETARY RISK ASSESSMENT

Long-term intake

The International Estimated Dietary Intakes (IEDIs) of zoxamide were calculated for the 13 GEMS/Food cluster diets using STMRs/STMR-Ps estimated by the current Meeting (Annex 3 of the 2007 Report of the JMPR). The ADI is 0 – 0.5 mg/kg bw and the calculated IEDIs were all 0% of the maximum ADI. The Meeting concluded that the long-term intakes of residues of zoxamide, resulting from the uses considered by the current JMPR, are unlikely to present a public health concern.

Short-term intake

The 2007 JMPR decided that an ARfD is unnecessary. The Meeting therefore concluded that the short-term intake of residues of zoxamide is unlikely to present a public health concern.

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146039	Grolleau, G.	1999	Magnitude of The Residue of RH-7281 and Mancozeb in Grape Raw Agricultural Commodity and of RH-7281 in Wine - Northern and Southern France - 1997
220177	Grolleau, G.	1999	Magnitude of The Residue of RH-7281 and Mancozeb in Grape Raw Agricultural Commodity and Of RH-7281 in Wine and Processed Fractions - Northern and Southern France - 1996
221382	Grolleau, G.	2000	Magnitude of The Residue of RH-7281/Mancozeb 76.25WG in Grapes Raw Agricultural Commodity - Southern France - 1999
221383	Grolleau, G.	2000	Magnitude of The Residue Of RH-7281/Mancozeb 76.25WG in Grapes Raw Agricultural Commodity - Northern France - 1999
221384	Wais, A.	2000	Determination of Residues Of RH-117,281 and Mancozeb in/on Vine Grapes (RAC Grapes) Following Treatment With RH-7281/Mancozeb 75WG From A Field Trial (Semi Residue Decline Study) in Germany, 1999
221385	Wais, A.	1999	Determination of Residues of RH-117,281 and Mancozeb in/on Table Grapes (RAC Grapes) Following Treatment With RH-7281 2F and Dithane/RH-117,281 75DG Blend From Field Trials in Italy, 1996
221387	Wais, A.	1999	Determination Of Residues Of RH-117,281 and Mancozeb in/on Vine Grapes (RAC Grapes) Following Treatment With Dithane/RH-117,281 75DG Blend (8:1) and Dithane/RH-117,281 75WP Blend (8:1) and RH-7281 2F Experimental Fungicide From Four Field Trials in Germany
221390	Wais, A.	1999	Determination of Residues of RH-117,281 and Mancozeb in/on Vine Grapes (RAC Grapes) Following Treatment With Dithane/RH-117,281 75DG Blend (8:1) and Dithane/RH-117,281 75WP Blend (8:1) From Two Field Trials (Semi Residue Decline Studies) in Italy, 1998
221861	Wais, A.	1999	Determination of Residues Of RH-117,281 and Mancozeb in/on Vine (RAC Grapes) Following Treatment With RH-7281 2F and Dithane/RH-117,281 75 DG Blend From Field Trials in Italy, 1996
221862	Wais, A.	1999	Determination of Residues of RH-117,281 and Mancozeb in/on Table Grapes (RAC Grapes) Following Treatment With RH-7281 2F and Dithane/RH-117,281 75 DG Blend From Field Trials in Spain, 1996
221863	Wais, A.	1999	Determination of Residues Of RH-117,281 and Mancozeb in/on Vine (RAC Grapes) Following Treatment With RH-7281 2F and Dithane/RH-117,281 75 DG Blend From Field Trials in Germany, 1997
221864	Wais, A.	1999	Determination Of Residues of RH-117,281 and Mancozeb in/on Vine (RAC Grapes) Following Treatment With RH-7281 2F and Dithane/RH-117,281 75 DG Blend From Field Trials in Italy, 1997
221866	Wais, A.	1999	Determination of Residues Of RH-117,281 and Mancozeb in/on Vine (RAC Grapes) Following Treatment With RH-7281 2F and Dithane/RH-117,281 75 DG Blend From Two Field Trials in Greece, 1997
221867	Wais, A.	1999	Determination Of Residues Of RH-117,281 and Mancozeb in/on Vine (RAC Grapes) Following Treatment With RH-7281 2F and Dithane/RH-117,281 75 DG Blend From Field Trials in Germany, 1996
91475	Guo, I.	1996	Preliminary Residue Analytical Method For Parent RH-7281 in Grapes
92154	Burdge, E. L.	1998	Tolerance Enforcement Method For RH-117,281 in Grapes and Processed Fractions
92215	Graves, D. D.	1998	RH-117,281 80W and 2F Residue Studies in Grapes and Grape Process Fractions 1996 and 1997 Trials
110110	Vaughn, F. C.	2000	Magnitude Of Residue of RH-7281 and Mancozeb in Grapes Following Treatment With RH-7281 80W and RH-7281 Mz 75df Formulation
92154	Burdge, E. L.	1998	Tolerance Enforcement Method For RH-117,281 in Grapes and Processed Fractions
103349	Pinheiro, A. C.	2002	Residues of Zoxamide and Mancozeb in Grapes After Multiple Applications of Stimo PM -
134801	Pinheiro, A. C.	2003	Residues of Zoxamide and Cymoxanil in Grapes After Treatment With Harpon GD, Fungicide - Brazil - 2002-03.
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221882	Tornisielo, V. L.	1999	Determination of Residues Of RH-7281/Dithane PM (RH-7281/Mancozeb) On Grapes - Proto Feliz/Sp (Determinacao De Residuo De RH-7281/Dithane PM (RH-7281/Mancozeb) Em Cultura De Uva - Porto Feliz/Sp

Code	Author	Year	Title, Report reference
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224088	Tornisielo, V. L.	2000	Determinacao De Residuo De RH-7281/Cymoxanil (RH-7281 E Cymoxanil) Em Frutos De Uva – Indaiatuba-Sp (Determination Of Residues Of RH-7281/Cymoxanil in Grape)
224089	Tornisielo, V. L.	2000	Determinacao De Residuo De RH-7281/Cymoxanil (RH-7281 E Cymoxanil) Em Frutos De Uva – Indaiatuba-Sp (Determination Of Residues Of RH-7281/Cymoxanil in Grape)
239921		2000	Zoxamide Crop Residue Study On Grape
92230	Guo, I.	1999	Preliminary Residue Analytical Method For Parent RH-7281 in Cucurbits
224320	Perny, A.	2001	Determination Of Mancozeb (As CS2) and RH-7281 Residues in Cucumber Following Treatments With The Preparations RH-7281 2F and RH-7281/Mancozeb 75WG Under Greenhouse Conditions in France in 1999 / RH-7281 2F, Northern Europe (France)
224321	Perny, A.	2001	Determination Of Mancozeb (As CS2) and RH-7281 Residues in Cucumber Following Treatments With The Preparations RH-7281 2F and RH-7281/Mancozeb 75 WG Under Greenhouse Conditions in France in 1999 / RH-7281/Mancozeb 75 WG, Northern Europe (France)
224329	Wais, A.	2000	Determination Of Residues Of RH-117,281 in/on Cucumber (RAC Fruits) Following Treatment With RH-7281 2F From One Field Trial (Residue Decline Study) Under Protection in Spain; 1999
224789	Perny, A.	2001	Determination Of Mancozeb (As CS2) and RH-7281 Residues in Cucumber Following Treatments With The Preparations RH-7281 2F and RH-7281/Mancozeb 75WG Under Greenhouse Conditions in France in 1999 / RH-7281 2F, Southern Europe (France)
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238367	Wais, A.	2001	Determination Of Residues Of RH-117,281 in/on Cucumber (RAC Fruits) Following Treatment With RH-7281/Mancozeb 75 WG From Two Field Trials (Residue Decline Studies) Under Protection in Spain, 1999
92230	Guo, I.	1999	Preliminary Residue Analytical Method For Parent RH-7281 in Cucurbits
92644	Graves, D. D.	1999	RH-117281 Fungicide Field Residue Trials in The Cucurbit Vegetable Group
238418		1998	Zoxamide Crop Residue Study On Cucumber
92229	Burdge, E. L.	1999	Test Method 34-99-68: Preliminary Residue Analytical Method For Parent RH-117,281 in Tomato
224019	Wais, A.	2000	Determination Of Residues Of RH-117,281 and Mancozeb in/on Tomatoes (RAC Fruit) Following Treatment With Dithane/RH-117,281 75DG Blend (8:1) From Four Field Trials (Residue Decline Studies) Under Plastic Tunnel in Spain; 1998
224322	Wais, A.	2000	Determination Of Residues Of RH-117,281 and Mancozeb in/on Field Tomatoes (RAC Fruits) Following Treatment With Dithane/RH-117,281 75DG Blend (8:1) and RH-7281 2F Experimental Fungicide From Four Field Trials (Residue Decline Studies) in Italy; 1998
224326	Galy, H.	2001	Determination Of RH-7281 and Mancozeb (As CS2) Residues in Tomato Raw Agricultural Commodity and in Processed Tomatoes Following Treatments With The Preparations Dithane M-45, RH-7281 2F, and RH-7281/Mancozeb 75WG Under Field Or Greenhouse Conditions in E
224328	Wais, A.	2000	Determination Of Residues Of RH-117,281 in/on Tomato (RAC Fruits) Following Treatment With RH-7281 2F From Two Field Trials (Residue Decline Studies) in Open Field in Italy (North); 1999
224330	Wais, A.	2000	Determination Of Residues Of RH-117,281 and Mancozeb in/on Tomato (RAC Fruits) Following Treatment With RH-7281/Mancozeb 75WG From Two Field Trials (Residue Decline Studies) Under Plastic Tunnel in Greece; 1999
224331	Wais, A.	2000	Determination Of Residues Of RH-117,281 in/on Tomato (RAC Fruits) Following Treatment With RH-7281 2F From One Field Trial (Residue Decline Study) Under Plastic Tunnel in Greece; 1999
224333	Wais, A.	2001	Determination Of Residues Of RH-117,281 and Mancozeb in/on Tomato (RAC Fruits) Following Treatment With RH-7281/Mancozeb 75WG From Two Field Trials (Residue Decline Studies) in Open Field in Italy (North); 1999

Code	Author	Year	Title, Report reference
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238371	Wais, A.	2000	Determination Of Residues Of RH-117,281 and Mancozeb in/on Tomato (RAC Fruits) Following Treatment With RH-7281/Mancozeb 75WG From Two Field Trials (Residue Decline Studies) Under Plastic Tunnel in Spain, 1999
92229	Burdge, E. L.	1999	Test Method 34-99-68: Preliminary Residue Analytical Method For Parent RH-117,281 in Tomato
92299	Graves, D. D.	1999	RH-117281 Fungicide Field Residue Trials in Tomatoes and Tomato Processed Fractions
105233	Kalvan, H. C.	2002	Residues Of Zoxamide and Mancozeb in Tomato After Multiple Applications Of Stimo PM - Fungicide - Brazil, 2001-02
135350	Oliveira, R. C.	2003	Residues Of Zoxamide and Cymoxanil in Tomatoes After Treatment With Harpon GD, Fungicide - Brazil, 2002-03
221152	Pinheiro, A. C.	2002	Residue Analysis Of Zoxamide in Grapes, Potatoes and Tomatoes Using GC- Ms Detection
221855	Tornisielo, V. L.	1999	Determination Of Residues Of RH-7281/Dithane PM (RH-7281/Mancozeb) On Tomato - Rafard/Sp (Determinacao De Residuo De RH-7281/Dithane PM (RH-7281/Mancozeb) Em Tomate - Rafard/Sp
221879	Tornisielo, V. L.	1999	Determination Of Residues Of RH-7281/Dithane PM (RH-7281/Mancozeb) On Tomato - Paulinia/Sp (Determinacao De Residuo De RH-7281/Dithane PM (RH-7281/Mancozeb) Em Tomate - Paulinia/Sp
224090	Tornisielo, V. L.	2000	Determinacao De Residuo De RH-7281/Cymoxanil (RH-7281 E Cymoxanil) Em Frutos De Tomate - Paulinia/Sp (Determination Of Residues Of RH-7281/Cymoxanil in Tomato)
224091	Tornisielo, V. L.	2000	Determinacao De Residuo De RH-7281/Cymoxanil (RH-7281 E Cymoxanil) Em Frutos De Tomate - Monte Mor/Sp (Determination Of Residues Of RH-7281/Cymoxanil in Tomato)
148179	Wais, A.	2000	Determination of residues of RH-117,281 and its metabolites RH-141,452 and RH-141,455 in/on potatoes (rac tubers) following treatment with RH-7281/mancozeb 75WP from a field trial (semi residue decline study) in Northern France
148180	Wais, A.	2000	Determination Of Residues Of RH-117281 and Its Metabolites RH-141452 and RH-141455 in/on Potatoes (RAC Tubers) Following Treatment With RH-7281/Mancozeb 75WG From A Field Trial (Semi Residue Decline Study) in The Netherlands; 1999
148181	Grolleau, G.	1999	Magnitude Of Residue Of RH-7281 and Its Metabolites RH-1452 and RH-1455 in Potato - Raw Agricultural Commodity - Northern and Southern France - 1996
148182	Wais, A.	2000	Determination Of Residues Of RH-117281 and Its Metabolites RH-141452 and RH-141455 in/on Potatoes (RAC Tubers and Processing Products) Following Treatment With RH-7281/Mancozeb 75WG From A Field Trial (Semi Residue Decline Study) in Northern France; 1999
148183	Grolleau, G.	1999	Magnitude Of The Residue Of RH-7281 and Its Metabolites RH-1452 and RH-1455 in Potato Raw Agricultural Commodity, Northern and Southern France - 1997
148184	Wais, A.	2000	Determination Of Residues Of RH-117,281 and Its Metabolites RH-141,452 and RH-141,455 in/on Potatoes (RAC Tubers and Processing Products) Following Treatment With RH-7281/Mancozeb 75WG From A Field Trial (Semi Residue Decline Study) in Italy; 1999
221380	Wais, A.	1999	Determination Of Residues Of RH-117,281 and Mancozeb in/on Potato (RAC Tubers) Following Treatment With Dithane/RH-117,281 75DG Blend (8:1) and Dithane/RH-117,281 75WP Blend (8:1) From Two Field Trials (Semi Residue Decline Studies) in Germany; 1998
221386	Wais, A.	1999	Determination Of Residues Of RH-117,281 and Mancozeb in/on Potato (RAC Tubers) Following Treatment With Dithane/RH-117,281 75DG Blend (8:1) and Dithane/RH-117,281 75WP Blend (8:1) From Two Field Trials (Semi Residue Decline Studies) in UK, 1998
221388	Wais, A.	1999	Determination of Residues Of RH-117,281 in/on Potato (RAC Tubers) Following Treatment With Dithane/RH-117,281 75DG Blend (8:1) and Dithane/RH-117,281 75WP Blend (8:1) From Four Field Trials (Semi Residue Decline Studies) in Spain, 1998
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Code	Author	Year	Title, Report reference
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221391	Wais, A.	1999	Determination of Residues of RH-117,281 and Mancozeb in/on Potatoes (RAC Tubers) Following Treatment With RH-7281 2F and Dithane/RH-117,281 in Dithane/RH-117,281 75DG Blend From Field Trials in Germany, 1997
221392	Wais, A.	1999	Determination of Residues Of RH-117,281 and Mancozeb in/on Potatoes (RAC Tubers) Following Treatment With RH-7281 2F and Dithane/RH-117,281 75DG Blend From Field Trials in Greece, 1997
221394	Wais, A.	1999	Determination of Residues Of RH-117,281 and Mancozeb in/on Potatoes (RAC Tubers) Following Treatment With RH-7281 2F and Dithane/RH-117,281 75DG Blend From Field Trials in Italy, 1997
221395	Wais, A.	1999	Determination of Residues Of RH-117,281 and Mancozeb in/on Potatoes (RAC Tubers) Following Treatment With RH-7281 2F and Dithane/RH-117,281 75DG Blend From Field Trials in Italy, 1996
221401	Wais, A.	1999	Determination of Residues Of RH-117,281 and Mancozeb in/on Potatoes (RAC Tubers) Following Treatment With RH-7281 2F and Dithane/RH-117,281 75DG Blend From Field Trials in The UK, 1997
221860	Wais, A.	1999	Determination Of Residues Of RH-117,281 and Mancozeb in/on Potatoes (RAC Tubers) Following Treatment With RH-7281 2F and Dithane/RH-117,281 75 DG Blend From Field Trials in The United Kingdom, 1996
224617	Wais, A.	1999	Validation Of The Residue Analytical Method For Parent RH-117,281 and Its Two Acid Metabolites, RH-1452 and RH-1455, in Potatoes
91463	Desai, T.	1998	Preliminary Residue Analytical Method For Parent RH-7281 and Its Two Acid Metabolites, RH-1452 and RH-1455, in Potatoes
92289	Vaughn, F. C.	1998	Magnitude Of Residue Of RH-7281 and Mancozeb in Potatoes Following Treatment With RH-7281 80W and An RH-7281/Mancozeb Pre-Mix Formulation
92643	Graves, D. D.	1998	RH-117,281 80W and 2F Residue Studies in Potatoes and Potato Processed Fractions: 1996 and 1997 Trials
109986	West, S.	2000	RH-117,281 Fungicide Field Residue Study in Potatoes Under Conditions in Mexico
91463	Desai, T.	1998	Preliminary Residue Analytical Method For Parent RH-7281 and Its Two Acid Metabolites, RH-1452 and RH-1455, in Potatoes
102061	Oliveira, R. C.	2002	Residuos De Zoxamide E Mancozeb Em Batata Apos Multiplas Aplicacoes De Stimo PM – Fungicida, Brasil - 2001-02 (Residues Of Zoxamide and Mancozeb in Potatoes After Multiple Applications Of Stimo PM - Fungicide – Brazil, 2001-02)
135351	Oliveira, R. C.	2003	Residues Of Zoxamide and Cymoxanil in Potatoes After Treatment With Harpon GD, Fungicide - Brazil, 2002-03
204328	Tornisielo, V. L.	2000	Determinacao De Residuo De RH-7281/Cymoxanil (RH-7281 E Cymoxanil) Em Tuberculos De Batata – Paulinia-Sp (Determination Of Residues Of RH-7281/Cymoxanil in Potato)
221152	Pinheiro, A. C.	2002	Residue Analysis Of Zoxamide in Grapes, Potatoes and Tomatoes Using GC-MS Detection
221880	Tornisielo, V. L.	1999	Determination Of Residues Of RH-7281/Dithane PM (RH-7281/Mancozeb) On Potato - Paulinia/Sp (Determinacao De Residuo De RH-7281/Dithane PM (RH-7281/Mancozeb) Em Batata - Paulinia/Sp)
221884	Tornisielo, V. L.	1999	Determination Of Residue Of RH-7281/Dithane PM (RH-7281/Mancozeb) in Potatoes - Itapetininga/Sp (Determinacao De Residuo De RH-7281/Dithane PM (RH-7281/Mancozeb) Em Batata - Itapetininga/Sp)
224107	Tornisielo, V. L.	2000	Determinacao De Residuo De RH-7281/Cymoxanil (RH-7281 E Cymoxanil) Em Tuberculos De Batata – Sumare-Sp (Determination Of Residues Of RH-7281/Cymoxanil in Potato)
224603	Kalvan, H. C.	2006	Summary Of Stimo Residue Studies in Potatoes – Argentina, 2001/2002 and 2002/2003 Seasons
239922		1997	Zoxamide Crop Residue Study On Potato
91466	Kendi, M.	1998	Preliminary Residue Analytical Method For Parent RH-7281 in Raisins
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92294	Mamouni, A.	1998	14C-RH-117281: Vinification Study
92234	Kurilla, K. J.	1999	Preliminary Residue Analytical Method For Parent RH-7281 in Tomato Puree and Paste
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Code	Author	Year	Title, Report reference
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91485	Graves, D.D.	2000	RH-117,281 80W and 2F Residue Studies in Potatoes and Potato Processed Fractions 1996 and 1997 Trials - Analysis Of Potato Chips and Flakes For Residues Of Parent RH-117,281
92638	Graves, D.D.	2000	Analysis Of Potato Peel For Residues Of Parent RH-117,281 and Metabolites RH-141,452 and RH-141,455 - Supplement To RH-117,281 80W and 2F Residue Studies in Potatoes and Potato Processed Fractions 1996 and 1997 Trials
92657	Guo, I.	2000	Test Method 34-00-37: Preliminary Residue Analytical Method For Parent RH-7281 and Its Two Acid Metabolites, RH-1452 and RH-1455, in Potato Peel Waste