Kresoxim-methyl (199)

The first draft was prepared by Dr Jochen Heidler, Federal Institute for Risk Assessment, Berlin, Germany

EXPLANATION

Kresoxim-methyl is a strobilurin fungicide for the control of scab and other fungal diseases on a wide range of crops. It acts by inhibiting the mitochondrial respiration. It was first evaluated by JMPR in 1998 (T, R), when an acceptable daily intake (ADI) of 0– 0.4 mg/kg bw was established. An acute reference dose (ARfD) was not considered necessary. In 2001 the JMPR evaluated the compound for residues and recommended several maximum residue levels. Kresoxim-methyl was scheduled by the Forty-ninth Session of the CCPR for the periodic evaluation of residues and toxicology by the 2018 JMPR.

The Meeting received information on identity, physicochemical properties, metabolism (plant, confined rotational crops and animals), environmental fate, methods of residue analysis, freezer storage stability, registered use patterns, supervised residue trials in citrus, apple, peach, black currant, strawberry, grape, mango, leek, onion, garlic, cucumber, squash, gherkin, melon, sweet pepper, tomato, vine leaves, sugar beet, wheat, barley, pecan nuts and olives, fate of residues in processing, and livestock feeding studies.

IDENTITY

| ISO common name | Kresoxim-methyl |
|--------------------|---|
| Chemical name | methyl (aE)-a-(methoxyimino)-2-[(2-methylphenoxy)methyl]benzene acetate |
| IUPAC & CA | methyl (E)-methoxyimino[a-(o-tolyloxy)-o-tolyl]acetate |
| Synonyms | BAS 490 F, |
| CAS No. | 143390-89-0 |
| CIPAC No. | 568 |
| Structural formula | |
| | ^ |

H,C CH₂ CH₃ H₃C 0

Molecular formula Molecular mass C₁₈H₁₉NO₄ 313.36 g/mol

Specifications

Specifications for kresoxim-methyl were not yet developed by FAO.

Physical and chemical properties

Table 1 Physical and chemical properties of pure kresoxim-methyl

| Property | Results | Method (test material) | Reference |
|--|--|---|------------|
| Appearance | Physical state white crystalline solid Odour mildly aromatic | | JMPR, 1998 |
| Melting point | 101.6-102.5°C | OECD 102 Kresoxim-methyl (99.7% purity) | JMPR, 1998 |
| Boiling point & temperature of decomposition | No boiling or sublimation of kresoxim-methyl up to 310 °C decomposition temperature | OECD 113 | JMPR, 1998 |

| Property | Results | Method (test material) | Reference |
|--|--|---|---|
| Relative density | 1.258 g/mL at 20°C | EEC method A 3.1.4.3 | JMPR, 1998 |
| рН | At 1% concentration in CIPAC water D (pH 6.5): 4.4 | CIPAC MT 75 (Lot: COD-001068, 94.4% purity) | Kroehl T., 2008, Kresoxim_001 |
| Vapour pressure | Kresoxim-methyl: 2.3 × 10 –6 Pa at 20 °C (extrapolated) Free acid (BF 490-1): <1 × 10-5 Pa | EEC A.4 Kresoxim-methyl (Lot: CH 39/149-1, 99.6% purity) | JMPR, 2001 Gueckel W., 1992, Kresoxim_001 Kroehl T., 1998, Kresoxim_001 |
| Henry´s Law Coefficient | $3.6 \times 10^{-7} \text{ kPa m}^3 \text{ mol}^{-1} \text{ at } 20^{\circ}\text{C}$ | Calculation | Ohnsorge U., 1999, Kresoxim_004 |
| Partition coefficient n-octanol / water | Kresoxim-methyl: log K _{ow} = 3.40 at 25 °C Free acid (BF 490-1): log K _{ow} = 0.15 (pH 7; 20 °C) log K _{ow} = 2.74 (pH 4; 20 °C) log K _{ow} = -2.85 (pH 9; 20 °C) | Kresoxim-methyl (99.4% purity) | JMPR, 1998 JMPR, 2001 |
| Solubility in water | Kresoxim-methyl: 2.00 \pm 0.08 g/L at 20 °C Free acid (BF 490-1): 91 mg/L at 20 °C | EEC A.6 Kresoxim-methyl (99.4% purity) | JMPR, 1998 |
| Solubility in organic solvents | n-heptane 1.7 g/kg toluene 111 g/kg dichloromethane 939 g/kg methanol 14.9 g/kg acetone 217 g/kg ethyl acetate 123 g/kg all at 20 °C | Kresoxim-methyl (99.7% purity) | JMPR, 1998 |
| Hydrolysis | DT_{50} at pH 5: 822 days (extrapolated) DT_{50} at pH 7: 35 days DT_{50} at pH 9: 0.38 days degradation to 490M1 (80% in 24 hours) | US-EPA-161-1 Kresoxim-methyl (ring B-U- ¹⁴ C), >98% radiochemical purity) | Van Beinum W., Beulke S 2008, Kresoxim_005 |
| Photolysis | At 25 °C for 370 days of continous irradiation in sodium scetat buffer pH 5 DT_{50} : 29.3 days | Kresoxim-methyl (ring B-U ⁻¹⁴ C) Lot: 445-25, 99.3% radiochemical purity | Beulke S., 2008, Kresoxim_006 |
| Lifetime in the top layer of aqueous systems (calculated and real) | Application period April to August: DT_{50} around 1 calendar day | EEC 94/37 | Hassink J., 2008, Kresoxim_007 |
| Estimated photochemical oxidative degradation | Calculated atmospheric degradation half-life of kresoximmethyl: $t_{1/2} = 0.28 \text{ d} (12 \text{ h day})$ | Not relevant | Hassink J., 2008, Kresoxim_008 |
| Dissociation constant | Kresoxim-methyl: pKa >2 and <12; titration for acidic range. Dissociation in alkali claimed not to be possible because of structure. Free acid (BF 490-1): pKa = 4.2 (20 °C). | 0ECD 112 | JMPR, 1998 |
| Flammability including auto- flammability Explosive properties, Oxidizing/reducing properties | Preliminary test: brief burning and rapid extinction. No gas evolution in contact with water. No explosive properties. No oxidizing properties | EEC A. 10 EEC A. 12 EEC A. 14 EEC A. 16 (Lot: COD-000225, 97.8% purity) | Bitterlich S., 2008, Kresoxim_009 |
| Surface tension | 72.3 mN/m at 20 °C. 0.5% (saturated) solution | Kresoxim-methyl (99.7% purity) | JMPR, 1998 |

| Property | Results | Results | | | Method | Reference |
|----------|----------|--------------|-----------|--------|-----------------|---------------|
| | | | | | (test material) | |
| Specta | 13 C-NN | IR | | | Lot: 01171-111 | Daum A. 2000, |
| | solvent/ | reference CE | 030D/CD30 |)D | 99.9 purity% | Kresoxim_010 |
| | ppm | carbon | ppm | carbon | | |
| | 16.2 | 14 | 127.6 | 6 | | |
| | 52.9 | 17 | 128.5 | 3 | | |
| | 63.8 | 18 | 129.0 | 2 | | |
| | 68.1 | 7 | 129.6 | 5 | | |
| | 111.2 | 13 | 130.7 | 10 | | |
| | 120.7 | 11 | 135.8 | 1 | | |
| | 126.7 | 12 | 149.4 | 15 | | |
| | 127.0 | 9 | 156.6 | 8 | | |
| | 127.5 | 4 | 163.3 | 16 | | |

Formulations

Kresoxim-methyl is applied formulated alone or in combination with other active substances. It is formulated as water dispersible granules (WG), suspension concentrate (SC) and suspo-emulsions (SE) products.

Table 2 Examples of formulations registered containing kresoxim-methyl as active ingredient

| Formulation type | WG | SC | SE | SE | SE | SC | SC |
|-----------------------|-----|---------|---------|---------|---------|---------|---------|
| Kresoxim-methyl (g/L) | 50% | 500 g/L | 150 g/L | 125 g/L | 83 g/L | 125 g/L | 100 g/L |
| Fenpropimorph | | | 300 g/L | 150 g/L | 317 g/L | | |
| Epoxiconazole | | | | 125 g/L | 83 g/L | 125 g/L | |
| Boscalid | | | | | | | 200 g/L |

METABOLISM AND ENVIRONMENTAL FATE

Metabolism studies were conducted using either [phenoxy-14C]- or [phenyl-14C]-kresoxim-methyl. Also, [13C]-kresoxim-methyl was used. The position of the label for the various test substances is presented in the following figure:

* [phenoxy-U-¹⁴C]-kresoxim-methyl # [phenyl-¹⁴C]-kresoxim-methyl ° [¹³C]-kresoxim-methyl

Figure 1 Structure of kresoxim-methyl and position of radiolabels

Chemical names, structures and code names of metabolites and degradation products of kresoxim-methyl are shown below.

Table 3 Known metabolites of kresoxim-methyl

| Code Names | Chemical Names (IUPAC) | Structure | Where found |
|---|--|--|---|
| Kresoxim- methyl BAS 409 F LAB 242 009 | methyl (2E)-[2- (hydroxymethyl)phenyl](methoxyimino)acet ate | | Plants (sugar beet, apple, grape, wheat) Animals (goat, hen) Rotational crops (lettuce, radish) |
| | | Molar mass: 313.36 g/mol | |
| | | | Plants (sugar beet, apple, grape, wheat) |
| 490M1 | (2E)-(methoxyimino){2-[(2- | | Animals (goat, hen) |
| BF 490-1 | methylphenoxy)methyl]phenyl}acetic acid | | Rotational crops (carrot greens) |
| | | Molar mass: 299.33 g/mol | |
| | | | Plants (sugar beet, apple, grape, wheat) |
| 490M2 | (2E)-(2-{[2- (hydroxymethyl)phenoxy]methyl}phenyl)(me thoxyimino) acetic acid | | Animals (goat) |
| BF 490-2 | | Molar mass: 315.33 g/mol | Rotational crops (lettuce, carrot greens, bean forage, wheat straw) |
| 490M04 BF 490-5 | 2-({2-[(E)- carboxy(methoxyimino)methyl]phenyl}meth oxy)benzoic acid | CO ₂ H HO ₂ C N OMe | Environment (soil) |
| 490M5 | 2-((2-[(1E)-N,2-dimethoxy-2- oxoethanimidoyl]phenyl}methoxy)benzoic acid | | Animals (hen) |
| 490M6 | (2E)-[2- (hydroxymethyl)phenyl](methoxyimino)aceti c acid | HO O OH O CH ₃ | Animals (goat, hen) Rotational crops (wheat straw) |
| 490M8 BF 490-11 | (2E)-(2-{[4-hydroxy-2- (hydroxymethyl)phenoxy]methyl}phenyl)(me thoxyimino)acetic acid | HO CO ₂ H OME | Animals (hen) |

| Code Names | Chemical Names (IUPAC) | Structure | Where found |
|------------------------------------|---|---|--|
| 490M9 BF 490-9 | (2E)-{2-[(4-hydroxy-2- methylphenoxy)methyl]phenyl}(methoxyimi no)acetic acid | CH ₃ OH OH Molar mass: 315.33 g/mol | Plants (apple, grape, wheat) Animals (goat, hen) Rotational crops (lettuce, carrot greens, bean forage, wheat straw) |
| 490M11 | (2E)-(hydroxyimino)(2-{[2- (hydroxymethyl)phenoxy]methyl}phenyl)ace tic acid | CH ₂ OH O HO ₂ C | Animals (hen) |
| 490M14 (glucoronid of 490M1) | 1-0-[(2E)-2-(methoxyimino)-2-{2-[(2- methylphenoxy)methyl]phenyl}acetyl] glucuronide | | Animals (hen) |
| 490M15 BF 490-4 | methyl (2E)-{2-[(4-hydroxy-2- methylphenoxy)methyl]phenyl}(methoxyimi no)acetate | CH ₃ O H ₃ CO O CH ₃ O O CH ₃ O O O O CH ₃ | Animals (hen) |
| 490M17 | 4E)-1-(4-hydroxy-2-methylphenoxy)-4- (methoxyimino)-1,4-dihydro-3H-2- benzopyran-3-one | | Plants (wheat |
| 490M18 BF 490-8 | (2E)-(hydroxyimino){2-[(2- methylphenoxy)methyl]phenyl}acetic acid | OH CH3 | Animals (goat) |
| 490M19 | (4 <i>E</i>)-2-amino-4-(methoxyimino)-4-{2-[(2- methylphenoxy)methyl]phenyl}-3- oxobutanoic acid | H_2N OH OH | Animals (goat) |

| Code Names | Chemical Names (IUPAC) | Structure | Where found |
|-------------------------------------|--|--|---------------|
| 490M20 BF 490-14 | (4Z)-4-(methoxyimino)-1,4-dihydro-3H-2- benzopyran-3-one | O O O O N O Me | Animals (hen) |
| 490M24 | methyl (2E)-(2-{[2- (hydroxymethyl)phenoxy]methyl}phenyl)(me thoxyimino)acetate | CH ₂ OH 0 H ₃ C ⁰ 0 CH ₃ | Animals (hen) |
| 490M25 | [2-({2-[(E)- carboxy(methoxyimino)methyl]phenyl}meth oxy)phenyl]methyl glucuronide | H H O H H O O H H O O O O O O O O O O O | Animals (hen) |
| 490M26 | 4-{{2-[(E)- carboxy(methoxyimino)methyl]phenyl}meth oxy)-3-methylphenyl glucuronide | CO ₂ H H H OH H H H OH HO ₂ C N OMe | Animals (hen) |
| 490M28 (glucoronid of 490M15) | [2-((2-[(E)- carboxy(methoxyimino)methyl]phenyl}meth oxy)phenyl]methyl glucuronide | | Animals (hen) |
| 490M31 | [2-({2-[(1E)-N,2-dimethoxy-2- oxoethanimidoy]]phenyl}methoxy)phenyl]me thyl glucuronide | CO ₂ H H H O OH H H H OH O MeO ₂ C N OMe | Animals (hen) |
| 490M33 | 4-({2-[(1E)-N-hydroxy-2-methoxy-2- oxoethanimidoyl]phenyl}methoxy)-3- methylphenyl glucuronide | CO ₂ H H H O OH H H MeO ₂ C N ^O H | Animals (hen) |
| 490M39 | [2-({2-[(1E)-N-hydroxy-2-methoxy-2- oxoethanimidoy]]phenyl}methoxy)phenyl]me thyl glucuronide | CO ₂ H H H O OH H H H OH O MeO ₂ C OH | Animals (hen) |

| Code Names | Chemical Names (IUPAC) | Structure | Where found |
|---------------------|---|--|----------------|
| 490M46 | methyl (2E)-(2-{[4-hydroxy-2- (hydroxymethyl)phenoxy]methyl}phenyl)(me thoxyimino)acetate | HO H ₃ C O CH ₃ | Animals (hen) |
| 490M47 | methyl (2E)-(hydroxyimino){2-[(4-hydroxy-2- methylphenoxy)methyl]phenyl}acetate | HO-C-O-O-O-OH MeO ₂ C-N'OH | Animals (hen) |
| 490M48 BF 490-03 | methyl (2 <i>E</i>)-(hydroxyimino){2-[(2- methylphenoxy)methyl]phenyl}acetate | CH OH H ₃ C | Animals (hen) |
| 490M50 | (2E)-(2-{[2-(hydroxymethyl)-4- (sulfooxy)phenoxy]methyl}phenyl)(methoxyi mino)acetic acid | HO ₃ S-O-OH HO ₂ C N OMe | Animals (hen) |
| 490M51 | methyl (2 <i>E</i>)-(2-{[2-(hydroxymethyl)-4- (sulfooxy)phenoxy]methyl}phenyl)(methoxyi mino)acetate | H ₃ C ^O OH OH OH OH OH | Animals (hen) |
| 490M52 | (2E)-(methoxyimino)(2-{[2-methyl-4- (sulfooxy)phenoxy]methyl}phenyl)acetic acid | HO ₃ S-O-O-O-O-O-O-OMe | Animals (hen) |
| 490M54 | (2 <i>E</i>)-{2-[(5-hydroxy-2- methylphenoxy)methyl]phenyl}(methoxyimi no)acetic acid | O O O O H O C H ₃ O H O O H | Plants (grape) |
| 490M56 | (2E)-(hydroxyimino){2-[(4-hydroxy-2- methylphenoxy)methyl]phenyl}acetic acid | HO-O-O-OH HO ₂ C-N-OH | Animals (hen) |

| Code Names | Chemical Names (IUPAC) | Structure | Where found |
|------------|---|---|---------------|
| 490M57 | methyl (2E)-(hydroxyimino)(2-{[2- (hydroxymethyl)phenoxy]methyl}phenyl)ace tate | HO-O-O-OH HO ₂ C-N-OH | Animals (hen) |
| 490M58 | [(1 <i>E</i>)-1-(2-{[2- (hydroxymethyl)phenoxy]methyl}phenyl)-2- methoxy-2-oxoethylidene]azinic acid | OH O H ₃ C | Animals (hen) |
| 490M59 | [(1E)-1-{2-[(4-hydroxy-2- methylphenoxy)methyl]phenyl}-2-methoxy- 2-oxoethylidene]azinic acid | HO-CH ₃ MeO ₂ C N ⁺ O ⁻ OH | Animals (hen) |
| 490M60 | methyl (2E)-[2- (hydroxymethyl)phenyl](methoxyimino)acet ate | | Animals (hen) |
| 490M63 | 4-{{2-[(1E)-N,2-dimethoxy-2- oxoethanimidoyl]phenyl}methoxy)-3- (hydroxymethyl)phenyl glucuronide | H H H MeO ₂ C N OMe | Animals (hen) |
| 490M64 | 2-({2-[(1E)-N-hydroxy-2-methoxy-2- oxoethanimidoyl]phenyl}methoxy)benzoic acid | CO ₂ H O MeO ₂ C OH | Animals (hen) |
| 490M66 | methyl (2 <i>E</i>)-(methoxyimino)(2-{[2-methyl-4- (sulfooxy)phenoxy]methyl}phenyl)acetate | CH ₃ O H ₃ CO CH ₃ O CH ₃ O CH ₃ O CH ₃ O O S=O OH | Animals (hen) |

| Code Names | Chemical Names (IUPAC) | Structure | Where found |
|-------------------|---|--|--|
| 490M67 | [2-({2-[(1E)-N,2-dimethoxy-2- oxoethanimidoyl]phenyl}methoxy)-5- hydroxyphenyl]methyl glucuronide | CO ₂ H H H O OH H H H OH O HO OH H H H OH O HO OH O HO OMe | Animals (hen) |
| 490M68/490 M69 | 2-[carboxy(methoxyimino)methyl]benzoic acid | | Rotational crops (wheat straw) |
| 490M76/490 M77 | Not applicable | | Rotational crops (wheat straw) |
| 490M78 | Not applicable | | Rotational crops (lettuce, radish tops and roots, wheat forage) |

Plant metabolism

The metabolic fate in plants was investigated following foliar application of either [phenoxy-¹⁴C]-kresoxim-methyl to sugar beet and grapes, or [phenyl-¹⁴C]-kresoxim-methyl to apple, grape and wheat.

In all studies kresoxim-methyl was moderately degraded into several metabolites or their respective glucoside conjugates. In all matrices parent kresoxim-methyl was the major identified component. Further metabolites quantified at significant amounts were: 490M1 at up to 9.7% (0.12 mg eq/kg in sugar beet leaves (free), 490M2 at up to 14% TRR (0.55 mg eq/kg in grapes (sum of free and conjugated); 490M9 at up to 11% TRR (1.0 mg eq/kg) in wheat straw (conjugated).

Sugar beet

The metabolic fate of [phenoxy-¹⁴C]-radiolabelled kresoxim-methyl in sugar beet (variety Victoria) after two foliar applications at 0.15 kg ai/ha each was investigated in the study by Veit (1999, Kresoxim_011). The first application took place at BBCH 39 (leaves cover 90% of ground) and the second 3 weeks later or 28 days before harvest (BBCH stage not given). Samples of leaves and root were taken before and directly after the second treatment and at harvest. All samples were stored in a freezer at -18 °C until further processing.

The TRR in the homogenised samples was determined by combustion and LSC. In order to characterise and identify the radioactivity present, all samples were extracted at least three times with methanol, followed by two extractions with water. Radioactivity in the post-extraction solids was additionally extracted with aqueous ammonia solution. HPLC and LC-MS/MS against reference compounds were applied for the characterisation and identification of the radioactivity.

TRR levels in sugar beet leaves were generally higher, compared to roots, indicating only limited translocation into the roots. In leaves, the radioactive residue increased after the second treatment from 0.54–0.61 mg eq/kg to 1.4–1.8 mg eq/kg and remained fairly steady with 1.3–1.7 mg eq/kg until 28 DALT. In roots, the radioactive residue increased after the second treatment from 0.007 mg eq/kg to 0.024–0.053 mg eq/kg, before dropping down to 0.008–0.009 mg eq/kg. A summary of the radioactive residues found in present in Table 4.

| Sampling interval (DALT) | Matrix | TRR determined by direct combustion, mg eq/kg | TRR calculated, mg eq/kg ^a |
|----------------------------------|--------|--|---------------------------------------|
| Before 2 nd treatment | Roots | 0.007 | 0.007 |
| berore z treatment | Leaves | 0.61 | 0.54 |
| 0 | Roots | 0.053 | 0.024 |
| U | Leaves | 1.8 | 1.4 |
| 28 | Roots | 0.008 | 0.009 |
| 28 | Leaves | 1.7 | 1.3 |

Table 4 Total radioactive residues in sugar beet matrices after two foliar application of [14C]- kresoxim-methyl

^a Sum of extracts (methanol and water) and post-extraction residue (PES).

The radioactivity found in the fractions from the initial methanol/water extractions and in the unextracted remainder is presented in Table 5. Extractability of methanol/water was fairly high with 63–93% TRR extracted from sugar beet roots and 91–99% TRR extracted from leaves.

| Sampling | Sampling | | Methanol e | Methanol extract | | Water extract | | ERR ^a | | Unextracted | |
|------------------------|----------|--------------|------------|------------------|--------|---------------|-------|------------------|-------|-------------|--|
| interval (DALT) | Matrix | TRR mg/kg | mg/kg | % TRR | mg/kg | % TRR | mg/kg | % TRR | mg/kg | % TRR | |
| Before 2 nd | Roots | 0.007 | 0.004 | 66 | 0.001 | 10 | 0.005 | 76 | 0.02 | 24 | |
| treatment | Leaves | 0.54 | 0.53 | 97 | 0.05 | 1.0 | 0.53 | 98 | 0.012 | 2.3 | |
| 0 | Roots | 0.024 | 0.022 | 91 | <0.001 | 1.8 | 0.022 | 93 | 0.002 | 6.8 | |
| 0 | Leaves | 1.4 | 1.4 | 98 | 0.008 | 0.6 | 1.4 | 99 | 0.017 | 1.2 | |
| 20 | Roots | 0.009 | 0.005 | 61 | <0.001 | 2.5 | 0.005 | 63 | 0.003 | 37 | |
| 28 | Leaves | 1.3 | 1.1 | 89 | 0.033 | 2.6 | 1.1 | 91 | 0.11 | 8.9 | |

Table 5 Extractability of radioactive residues from sugar beet matrices after two foliar application of [14C]-kresoxim-methyl

^a Extractable radioactive residues: sum of methanol and water extracts

The distribution of radioactivity following 2×0.15 kg ai/ ha foliar applications is presented in Table 6. Parent kresoximmethyl accounted for 67–98% TRR (1.1–1.4 mg eq/kg) in sugar beet leaves. Metabolite 490M1 (free acid metabolite) was identified at up to 9.7% TRR (0.12 mg eq/kg) in leaves at 28 DALT, where the sugar conjugate of 490M2 was identified in similar amounts as well. Radioactivity in sugar beet roots was not further characterized due their low TRR. The proposed metabolic pathway of kresoxim-methyl is shown in Figure 2.

Table 6 Summary of identified/characterized residues in sugar beet following 2×0.15 kg ai/ha foliar application of [¹⁴C]-kresoximmethyl

| Fraction / | Radioactive residues | in mg eq/kg (% TRR) |
|-----------------------------|-------------------------------|--------------------------------|
| Solubilizates | Sugar beet leaves (0 DALT) | Sugar beet leaves (28 DALT) |
| TRR | 1.4 (100%) | 1.3 (100%) |
| Methanol/water extract | 1.4 (99%) | 1.1 (91%) |
| Kresoxim-methyl | 1.4 (98%) | 0.84 (67%) |
| 490M1 | 0.008 (0.6%) | 0.12 (9.7%) |
| Sugar conjugate of 490M2 | n/a | 0.12 (9.2%) |
| Characterized by HPLC | n/a | 0.033 (2.6%) |
| Post extraction solids | 0.017 (1.2%) | 0.011 (8.9%) |
| Aqueous ammonia extract | n/a | 0.029 (2.3%) |
| Total identified | 1.4 (99%) | 1.1 (86%) |
| Total characterized | n/a | 0.062 (4.9%) |
| Unextracted | 0.017 (1.2%) | 0.026 (2.0%) |
| Total | 1.4 (100%) | 1.2 (93%) |

Apples

The metabolic fate of [phenyl-¹⁴C]-radiolabelled kresoxim-methyl in apples (variety Mutsu) was investigated in the study by Grosshans (1994a, Kresoxim_012; 1994b, Kresoxim_013) and Hofmann (1992a, Kresoxim_014; 1992b, Kresoxim_015). Three different application schemes were used:

- Foliar application (whole tree): 6×0.4 kg ai/ ha at the beginning of flowering (1), petal fall (2), application every 28–42 days (3–5) and 2 weeks before harvest (6). Apples were harvested 14 DALT.
- Early application (whole tree): 2×0.4 kg ai/ ha at the beginning of flowering and at petal fall. Apples were harvested 149 DALT.
- 5. Fruit spray treatment (leaves and branches were covered with foil): 2×0.8 kg ai/ ha at 6 weeks before harvest and 2 weeks before harvest. Apples were harvested 14 DALT.

All samples were peeled, cored and stored at -18 °C until further processing.

The TRR in the homogenised samples was determined by combustion and LSC. Samples of apple peel and pulp (fruit flesh and cores) were extracted three times with methanol, followed by liquid-liquid partitioning against ethyl acetate. HPLC, TLC as well as GC-MS and LC-MS against reference compounds were applied for the characterisation and identification of the radioactivity. Methanol extracts from the leaf application scheme were further treated with hesperidinase and β -glucosidase to cleave conjugates. Radioactivity in the post-extraction solids from apple peel of the leaf application only was additionally extracted with aqueous ammonia solution, followed by acid and alkaline hydrolysis. Individual residues in the peel and pulp were recalculated on a whole-apple basis.

TRR levels in apples using three different application schemes are shown in Table 7. Levels were generally highest after fruit treatment with 6.3 mg eq/kg in peel and 0.84 mg eq/kg in the whole apple. Only levels found in apple pulp were slightly higher after foliar application with 0.032 mg eq/kg, compared to 0.023 mg eq/kg after fruit treatment.

| Sampling interval (DALT) | Matrix | TRR determined by direct combustion, mg eq/kg | TRR calculated, mg eq/kg ^a |
|-----------------------------|---------------------------|---|---------------------------------------|
| Foliar application (6× | 0.4 kg ai/ ha) | | |
| | Peel | 1.6 | 2.2 |
| | Fruit flesh | 0.036 | |
| 14 | Core | 0.044 | |
| | Pulp (fruit flesh + core) | | 0.032 |
| | Whole apple (calculated) | | 0.36 |
| Early application (2×0 |).4 kg ai/ ha) | | |
| | Peel | 0.33 / 0.045 | 0.29 |
| | Fruit flesh | 0.006 / 0.007 | |
| | Core | 0.016 / 0.039 | |
| 149 | Leaves | 1.0 | |
| | Brances | 0.41 | |
| | Pulp (fruit flesh + core) | | 0.005 |
| | Whole apple (calculated) | | 0.041 |
| Fruit treatment (2×0. | 8 kg ai/ ha) | | |
| | Peel | 4.6 / 5.7 | 6.3 |
| | Fruit flesh | 0.027 / 0.024 | |
| 14 | Core | 0.023 / 0.016 | |
| 14 | Leaves | 0.23 | |
| | Pulp (fruit flesh + core) | | 0.023 |
| | Whole apple (calculated) | | 0.84 |

Table 7 Total radioactive residues in apples using three different application schemes with [14C]- kresoxim-methyl

^a Sum of extracts (methanol and water) and post-extraction residue (PES).

The radioactivity found in the methanol extracts and in the unextracted remainder is presented in Table 8. Extractability of methanol ranged between 86–98% TRR.

| Sampling interval | Matrix | TDDmm#/lim | Methanol extract | | Unextracted | |
|--------------------------|---------------|------------|------------------|-------|-------------|-------|
| (DALT) | Matrix | TRR mg/kg | mg/kg | % TRR | mg/kg | % TRR |
| Foliar application (6×0 | .4 kg ai/ ha) | | | | | |
| | Peel | 2.2 | 2.1 | 95 | 0.11 | 4.9 |
| 14 | Pulp | 0.032 | 0.030 | 92 | 0.003 | 7.8 |
| | Whole apple | 0.36 | 0.34 | 95 | 0.018 | 5.1 |
| Early application (2×0.4 | 4 kg ai/ ha) | | | | | |
| | Peel | 0.29 | 0.28 | 95 | 0.013 | 4.6 |
| 149 | Pulp | 0.005 | 0.005 | 86 | <0.001 | 14 |
| | Whole apple | 0.041 | 0.038 | 94 | 0.002 | 5.7 |
| Fruit treatment (2×0.8 | kg ai/ ha) | | | | | |
| | Peel | 6.3 | 6.2 | 98 | 0.11 | 1.7 |
| 28 | Pulp | 0.023 | 0.022 | 95 | 0.001 | 5.0 |
| | Whole apple | 0.84 | 0.82 | 98 | 0.015 | 1.8 |

Table 8 Extractability of radioactive residues from apples after treatment with [14C]-kresoxim-methyl

The distribution of radioactivity in the three different application schemes is presented in Table 9. Parent kresoximmethyl accounted for 74–98% TRR (0.038–0.82 mg eq/kg), calculated on whole-apple basis. Identified metabolites were 490M1, 490M2 and 490M9, but none occurred in significant amounts. The proposed metabolic pathway of kresoxim-methyl is shown in Figure 2.

| y of identified/characterized | | |
|-------------------------------|--|--|
| | | |
| | | |

| Fraction / | Radioactive residues in mg eq/kg (% TRR) | | | | | | |
|------------------------------|--|-------------------|-------------------|--|--|--|--|
| Solubilizates | Foliar application | Early application | Fruit treatment | | | | |
| | (6×0.4 kg ai/ ha) | (2×0.4 kg ai/ ha) | (2×0.8 kg ai/ ha) | | | | |
| TRR | 0.36 (100%) | 0.041 (100%) | 0.84 (100%) | | | | |
| Methanol extract | 0.34 (95%) | 0.038 (94%) | 0.82 (98%) | | | | |
| Kresoxim-methyl | 0.28 (78%) | 0.030 (74%) | 0.78 (93%) | | | | |
| Kresoxim-methyl (Z- | 0.012 (3.3%) | <0.001 (1.2%) | 0.018 (2.2%) | | | | |
| isomer) | | | | | | | |
| 490M1 | 0.011 (3.0%) | <0.001 (1.9%) | 0.012 (1.4%) | | | | |
| 490M2 | 0.007 (1.8%) ^a | na | na | | | | |
| 490M9 | 0.008 (2.1%) ^a | na | na | | | | |
| 490M9 (isomer) | 0.003 (0.9%) ^a | na | na | | | | |
| Uncleaved conjugates | 0.001 (0.4%) ^a | 0.003 (6.1%) | 0.009 (1.1%) | | | | |
| MW313 | 0.006 (1.6%) | <0.001 (1.4%) | 0.003 (0.4%) | | | | |
| Characterized by HPLC | 0.013 (3.5%) | 0.004 (9.8%) | 0.001 (0.1%) | | | | |
| Post extraction solids | 0.018 (5.1%) | 0.002 (5.7%) | 0.015 (1.8%) | | | | |
| Aqueous ammonia solubilizate | 0.008 (2.2%) | na | na | | | | |
| Acid solubilizate (lignin) | 0.011 (3.1%) | na | na | | | | |
| Total identified | 0.32 (89%) | 0.032 (77%) | 0.81 (97%) | | | | |
| Total characterized | 0.039 (11%) | 0.008 (17%) | 0.013 (1.6%) | | | | |
| Unextracted | - | 0.002 (5.7%) | 0.015 (1.8%) | | | | |
| Total | 0.36 (100%) | 0.042 (102%) | 0.83 (99%) | | | | |

^a After hesperidinase and β -glucosidase treatment of methanol extract.

Grapes

The metabolic fate of [phenoxy-¹⁴C]- and [phenyl-¹⁴C]-radiolabelled kresoxim-methyl in grapes (variety Carlos) was investigated in the study by Nelsen J.M. *et al.* (1995), Kresoxim_016). Grape vines were treated five times at a rate of 0.5 kg ai/ha per application with intervals of 14–21 days. The grapes were harvested 14 DALT and stored in a freezer at -18 °C until further processing.

For the determination of the TRR, the grapes were rinsed with methanol and then homogenised. The TRR was calculated at the sum of the radioactivity in the methanol rinse and in the homogenised washed grapes, determined by combustion and LSC. To characterize and identify the radioactivity present, the grapes were either extracted with methanol or acetone/water (in parallel), followed by HPLC or TLC against reference compounds or GC-MS. Conjugated metabolites were treated with β -glucosidase and hesperidinase, followed by HPLC and/or LC-MS analysis. No additional attempts were undertaken to further characterize the unextracted radioactivity.

Table 10 shows that TRR levels measured in grapes for both labels were quite similar (~4 mg eq/kg), as well as were the fractions found in the methanol rinse (~35% TRR) and in the homogenised grapes (~65% TRR)

Table 10 Total radioactive residues in grapes after five foliar applications of [14C]-kresoxim-methyl at a rate of 0.5 kg/ha per application

| Sample | [phenoxy-14C]-kresoxim-r | nethyl | [phenyl-14C]-kresoxim-methyl | | |
|---------------------------|--------------------------|--------------|------------------------------|--------------|--|
| Sample | mg eq/kg (mean) | % TRR (mean) | mg eq/kg (mean) | % TRR (mean) | |
| Methanol rinse | 1.3, 1.6 (1.5) | 35, 40 (38) | 1.1, 1,9 (1.5) | 32, 41 (37) | |
| Homogenised rinsed grapes | 2.5, 2.4 (2.5) | 65, 60 (63) | 2.5, 2.8 (2.7) | 68, 59 (64) | |
| TRR | 3.8, 4.0 (3.9) | 100 | 3.6, 4.7 (4.2) | 100 | |

The radioactivity extracted from homogenised fruits by methanol or acetone/water is presented in Table 11. No significant difference between both solvents was recognized. The unextracted residue ranged between 4.5–6.3% TRR.

Table 11 Extractability of radioactive residues from grapes after five foliar applications of [14C]-kresoxim-methyl

| Sample | [phenoxy-14C]-kreso | xim-methyl | [phenyl-14C]-kresoxim-methyl | | |
|-----------------------------------|---------------------|---------------------|------------------------------|---------------------|--|
| Sample | Acetone/water | Methanol (mean) | Acetone/water | Methanol (mean) | |
| TRR (mg eq/kg) | 3.8 | 3.8, 4.0, 4.0 (3.9) | 3.6 | 3.6 4.7, 4.7 (4.4) | |
| Methanol rinse (% TRR) | 35 | 35, 40, 40 (38) | 32 | 32, 41, 41 (38) | |
| Extracted residue (% TRR) | 52 | 52, 51, 49 (51) | 57 | 60, 46, 50 (52) | |
| Sum of extracted residues (% TRR) | 87 | 87, 91, 89 (89) | 89 | 92, 86, 91 (90) | |
| Unextracted residues (% TRR) | 4.3 | 9.4, 3.8, 5.6 (6.3) | 4.5 | 7.8, 4.1, 5.5 (5.8) | |
| Total recovered residue (% TRR) | 91 | 96, 94, 95 (95) | 94 | 100, 90, 96 (96) | |

The distribution of radioactivity both radioactive labels is presented in Table 12 and was quite similar. Parent kresoximmethyl accounted for 55% TRR (2.2 mg eq/kg) and 57% TRR (2.7 mg eq/kg) in the [phenoxy-¹⁴C]- and [phenyl-¹⁴C]-kresoxim-methyl treated grapes, respectively. As a major metabolite, the sum of free and conjugated 490M2 was identified at 14% (0.55 mg eq/kg) and 13% TRR (0.42 mg eq/kg). Additionally metabolites 490M1, 490M2, 490M9 and 490M54 were identified at smaller amounts 0.13–5.8% TRR). Most of the identified metabolites occurred as their glucoside conjugates and were cleaved to their corresponding aglycons by β -glucosidase treatment. The proposed metabolic pathway of kresoxim-methyl is shown in Figure 2.

Table 12 Summary of identified/characterized residues in grapes after five foliar applications of [¹⁴C]-kresoxim-methyl

| Fraction / | Radioactive residues in mg eq/kg (% TRR) | |
|---------------------------------|--|------------------------------|
| Solubilizates | [phenoxy-14C]-kresoxim-methyl | [phenyl-14C]-kresoxim-methyl |
| TRR | 4.0 (100%) | 4.7 (100%) |
| Sum of methanol rinse & extract | 3.6 (91%) | 4.1 (86%) |
| Kresoxim-methyl | 2.2 (55) | 2.7 (57) |
| Kresoxim-methyl (Z-isomer) | 0.14 (3.5%) | 0.18 (3.8%) |
| 490M1 ^a | 0.019 (0.48%) | 0.006 (0.13%) |
| 490M2 ^a | 0.55 (14%) | 0.42 (8.9%) |
| 490M9 ^a | 0.23 (5.8%) | 0.21 (4.4% |
| 490M54 | 0.083 (2.1%) | 0.064 (1.4%) |
| Conjugates | 0.13 (3.2%) | 0.13 (2.8%) |
| Characterized by HPLC | 0.13 (3.1) | 0.22 (4.6%) |
| Background | 0.15 (3.7%) | 0.14 (3.0%) |
| Total identified | 3.2 (81%) | 3.6 (76%) |
| Total characterized | 0.26 (6.3%) | 0.46 (9.7%) |
| Unextracted | 0.15 (3.8%) | 0.19 (4.1%) |
| Total | 3.6 (91%) | 4.3 (90%) |

^a Sum of free and conjugated

Wheat

The metabolic fate of [phenyl-¹⁴C]-radiolabelled kresoxim-methyl in wheat (variety Star) was investigated in the study by Grosshans (1994c, Kresoxim_017) and Hofmann (1991a, Kresoxim_018; 1991b, Kresoxim_019). Plants received two foliar application at 0.25 kg ai/ha or at an exaggerated rate of 1.25 kg ai/ha. The fist treatment occurred for both application rates at growth stage 29 (leaf sheaths lengthen); while the second treatment occurred 56 days later at growth stage 52 (first ears just visible). For both

applications forage samples were taken at 4 h and 55 days after the first treatment and at 4 h after the second treatment. Straw, husk and grain samples were taken 64 days after the second treatment. All samples were stored in a freezer at -18 °C until further processing.

The TRR in the homogenised samples was determined by combustion and LSC. In order to characterise and identify the radioactivity present, all samples were extracted with methanol followed by 0.5% aqueous ammonia solution. The conjugates in the extracts were hydrolyzed with β -glucosidase and hesperidinase. The extractable radioactivity was characterized by liquid-liquid partitioning, solid phase extraction and TLC. HPLC against reference compounds, GC-MS and LC-MS/MS were applied for the identification of the radioactivity. The post-extraction solids were further treated by enzymatic hydrolysis (cellulase, pectinase, β -glucosidase and hesperidinase), as well as by acid (refluxing with 1M HCl and 6M HCl, 1 h each) and alkaline hydrolysis (refluxing with 10% NaOH, 3 h).

TRR levels in wheat matrices after foliar application of two application rates are shown in Table 13. Radioactivity was highest in forage at day 0 of the first application and in straw harvested at 64 DALT. Levels in grain were significantly lower with residues of 0.059–0.063 mg eq/kg resulting from the normal treatment and 0.21–0.31 mg eq/kg resulting from the exaggerated treatment.

| Application rate | Sampling interval DALT (days after first treatment) | Matrix | TRR determined by direct combustion, mg eq/kg | TRR calculated, mg eq/kg ^a |
|------------------|--|--------|---|---------------------------------------|
| | -56 (0) | Forage | 11/8.1 | 12 |
| | -1 (55) | Forage | 1.3 / 2.1 | 1.3 |
| | 0 (56) | Forage | 5.2 / 7.7 | 5.3 |
| 2×0.25 kg ai/ ha | | Husks | 2.7 /1.8 | 2.7 |
| | 64 (120) | Straw | 13 /13 | 9.2 |
| | | Grain | 0.063 / 0.059 | 0.064 |
| | | Roots | 1.4 | - |
| | -56 (0) | Forage | 74 / 53 | 76 |
| | -1 (55) | Forage | 11/6.1 | 12 |
| | 0 (56) | Forage | 27 / 54 | 30 |
| 2×1.25 kg ai/ ha | | Husks | 21/11 | 18 |
| | (4(100) | Straw | 62 /45 | 61 |
| | 64 (120) | Grain | 0.31 / 0.21 | 0.26 |
| | | Roots | 3.1 | - |

Table 13 Total radioactive residues in wheat after two treatments with [phenyl-14C]-kresoxim-methyl

^a Sum of methanol extract and post-extraction residue (PES).

The radioactivity found in the fractions from the initial methanol extraction, the subsequent ammonia extraction and in the unextracted remainder is presented in Table 14. Extractability of radioactive residues from forage, husks and straw ranged between 90–100% TTR, while it was lower for grain with 61–75% TRR.

Table 14 Extractability of radioactive residues from wheat matrices after two foliar application of [phenyl-14C]-kresoxim-methyl at normal and exaggerated rates

| Sampling interval | | | Methanol extract | | Ammonia | Ammonia extract | | ERR ^a | | Unextracted | |
|--------------------------------------|--------|--------------------------|------------------|-------|-------------|-----------------|-------------|------------------|-------------|-------------|--|
| DALT (days after first treatment) | Matrix | TRR ^{mg eq/} kg | mg eq/kg | % TRR | mg eg/kg | % TRR | mg eg/kg | % TRR | mg eq/kg | % TRR | |
| 2×0.25 kg ai/ ha | | | | | | | | | | | |
| -56 (0) | Forage | 12 | 12 | 100 | 0.022 | 0.2 | 12 | 100 | 0.033 | 0.3 | |
| -1 (55) | Forage | 1.3 | 1.2 | 93 | 0.062 | 4.8 | 1.3 | 98 | 0.048 | 3.7 | |
| 0 (56) | Forage | 5.3 | 5.1 | 98 | 0.088 | 1.7 | 5.2 | 100 | 0.061 | 1.2 | |
| | Husks | 2.7 | 1.4 | 54 | 0.95 | 36 | 2.4 | 90 | 0.27 | 10 | |
| 64 (120) | Straw | 9.2 | 7.6 | 82 | 1.5 | 16 | 9.1 | 98 | 0.27 | 2.9 | |
| | Grain | 0.064 | 0.020 | 30 | 0.020 | 31 | 0.040 | 61 | 0.025 | 39 | |
| 2×1.25 kg ai/ ha | | | | | | | | | | | |
| -56 (0) | Forage | 76 | 75 | 99 | 0.10 | 0.1 | 75 | 99 | 0.098 | 0.1 | |
| -1 (55) | Forage | 12 | 11 | 96 | 0.30 | 2.5 | 12 | 99 | 0.19 | 1.6 | |
| 0 (56) | Forage | 30 | 29 | 99 | 0.32 | 1.1 | 30 | 100 | 0.17 | 0.6 | |
| | Husks | 18 | 14 | 75 | 3.4 | 18 | 17 | 93 | 1.0 | 5.6 | |
| 64 (120) | Straw | 61 | 54 | 88 | 6.2 | 10 | 60 | 98 | 1.4 | 2.3 | |
| | Grain | 0.26 | 0.13 | 48 | 0.070 | 27 | 0.20 | 75 | 0.074 | 28 | |

^a Extractable radioactive residue: sum of methanol and ammonia extract

The distribution of radioactivity after treatment with [¹⁴C]-kresoxim-methyl at a normal and exaggerated rate is presented in Table 15 and Table 16. Parent kresoxim-methyl was the major component in forage (75–97% TRR), straw (64–83% TRR) and grain (17–40% TRR). As a major metabolite, the glucoside of 490M9 was identified at 11% TRR (1 mg eq/kg) and 7.9% TRR (0.1 mg eq/kg) in straw and forage (55 d after first appl.), respectively. Additionally, the Z-isomer of kresoxim-methyl and metabolites 490M1, 490M2 and 490M17 were identified at smaller amounts 0.4–7.2% TRR). The proposed metabolic pathway of kresoxim-methyl is shown in Figure 2.

Table 15 Summary of identified/characterized residues in wheat matrices after application of 2×0.25 kg ai/ ha [phenyl-¹⁴C]-kresoxim-methyl

| Fraction / | Radioactive residues | in mg eq/kg (% TRR) | | | |
|--------------------------------|---|--|-----------------|-----------------|--------------------------|
| Solubilizates | Forage (4 h after 1 st appl.) | Forage (55 d after 1 st appl.) | Forage (0 DALT) | Straw (64 DALT) | Grain (64 DALT) |
| TRR | 12 (100%) | 1.3 (100%) | 5.3 (100%) | 9.2 (100%) | 0.064 (100%) |
| Extracts ^a | 12 (100%) | 1.3 (98%) | 5.2 (99%) | 9.1 (98%) | 0.040 (61%) |
| Kresoxim-methyl | 12 (96%) | 0.98 (75%) | 4.9 (93%) | 5.9 (64%) | 0.011 (17%) |
| Kresoxim-methyl (Z- isomer) | 0.11 (0.9%) | 0.016 (1.2%) | 0.087 (1.7%) | 0.36 (3.9%) | <0.001 (0.3%) |
| 490M1 | 0.25 (2.1%) | 0.039 (3.0%) | 0.063 (1.2%) | 0.13 (1.4%) | n/d |
| 490M2 (conjugates) | - | 0.029 (2.2%) | - | 0.39 (4.2%) | - |
| 490M9 (conjugates) | - | 0.10 (7.9%) | - | 1.0 (11%) | - |
| 490M17 | n/d | 0.037 (2.8%) | 0.031 (0.6%) | 0.33 (3.6%) | <0.001 (0.8%) |
| Uncleaved conjugates | n/d | 0.037 (2.9%) | 0.098 (1.9%) | n/d | n/d |
| Characterized by HPLC | 0.12 (1.0%) | 0.036 (2.7%) | 0.038 (1.8%) | 0.72 (7.8%) | 0.020 (30%) ^b |
| Protein precipitation | - | - | - | - | 0.004 (6.3%) |
| Post extraction solids | 0.033 (0.3%) | 0.048 (3.7%) | 0.061 (1.2%) | 0.27 (2.9%) | 0.025 (39%) |
| Starch extraction | - | - | - | - | 0.020 (32%) |
| Lignin fractionation | - | - | - | 0.20 (2.1%) | 0.005 (7.9%) |
| Cellulose fractionation | - | - | - | 0.020 (0.2%) | 0.001 (1.6%) |
| Total identified | 12 (97%) | 1.2 (92%) | 5.1 (96%) | 8.1 (88%) | 0.012 (18%) |
| Total characterized | 0.12 (1.0%) | 0.073 (5.6%) | 0.14 (3.7%) | 0.94 (10%) | 0.050 (78%) |
| Unextracted | 0.033 (0.3%) | 0.048 (3.7%) | 0.061 (1.2%) | - | - |
| Total | 12 (98%) | 1.3 (101%) | 5.3 (101%) | 9.1 (98%) | 0.62 (96%) |

^a Sum of methanol and ammonia extract

^b Sum 13 minor HPLC peaks ranging from 0.0002–0.0036 mg eg/kg (0.3–5.6% TRR)

| Table 16 Summary of | identified/characterized | residues i | in wheat | matrices | after | application | of | 2×1.25 kg ai/ | ha | [phenyl-1- | ⁴ C]- |
|---------------------|--------------------------|------------|----------|----------|-------|-------------|----|---------------|----|------------|------------------|
| kresoxim-methyl | | | | | | | | | | | |

| Fraction / | Radioactive residues | in mg eq/kg (% TRR) | | | |
|-------------------------|------------------------|------------------------|-----------------|-----------------|-----------------|
| Solubilizates | Forage (4 h after | Forage (55 d after | Forage (0 DALT) | Straw (64 DALT) | Grain (64 DALT) |
| | 1 st appl.) | 1 st appl.) | | | |
| TRR | 76 (100%) | 12 (100) | 30 (100%) | 61 (100%) | 0.26 (100%) |
| Extracts ^a | 75 (99%) | 12 (99%) | 30 (100%) | 60 (98%) | 0.20 (75%) |
| Kresoxim-methyl | 73 (97%) | 10 (86%) | 27 (94%) | 51 (83%) | 0.10 (40%) |
| Kresoxim-methyl (Z- | 0.68 (0.9%) | 0.19 (1.6%) | 0.35 (1.2%) | 0.81 (1.3%) | n/d |
| isomer) | | | | | |
| 490M1 | 0.53 (0.7%) | 0.39 (3.3%) | 0.37 (1.3%) | 0.74 (1.2%) | <0.001 (0.4%) |
| 490M2 (conjugates) | - | - | - | - | n/d |
| 490M9 (conjugates) | - | - | - | - | 0.019 (7.2%) |
| 490M17 | n/d | 0.31 (2.6%) | 0.26 (0.9%) | 2.2 (3.6%) | 0.003 (1.2%) |
| Uncleaved conjugates | n/d | 0.53 (4.5%) | 0.34 (1.1%) | 4.7 (7.6%) | 0.003 (1.1%) |
| Characterized by HPLC | 0.84 (1.3%) | 0.036 (0.3%) | 0.29 (1.0%) | 0.50 (0.8%) | 0.026 (9.7%) |
| Protein precipitation | - | - | - | - | 0.013 (4.4%) |
| Post extraction solids | 0.098 (0.1%) | 0.19 (1.6%) | 0.17 (0.6%) | 1.4 (2.3%) | 0.074 (25%) |
| Starch extraction | - | - | - | - | 0.055 (18.5%) |
| Lignin fractionation | - | - | - | 0.95 (1.6%) | 0.008 (2.7%) |
| Cellulose fractionation | - | - | - | 0.070 (0.1%) | 0.002 (0.7%) |

| Fraction / | Radioactive residues | Radioactive residues in mg eq/kg (% TRR) | | | | | |
|---------------------|------------------------|--|-------------|-----------|------------|--|--|
| Solubilizates | Forage (4 h after | | | | | | |
| | 1 st appl.) | 1 st appl.) | | | | | |
| Total identified | 74 (99%) | 11 (94%) | 28 (97%) | 55 (89%) | 0.12 (49%) | | |
| Total characterized | 0.84 (1.3%) | 0.57 (4.8%) | 0.63 (2.0%) | 6.2 (10%) | 0.11 (37%) | | |
| Unextracted | 0.098 (0.1%) | 0.19 (1.6%) | 0.17 (0.6%) | - | - | | |
| Total | 76 (100%) | 12 (100%) | 29 (100%) | 61 (99%) | 0.23 (86%) | | |

^a Sum of methanol and ammonia extract

Figure 2 Proposed metabolic pathway of kresoxim-methyl in primary crops

Animal metabolism

Metabolism studies were provided for lactating goats using [phenyl-¹⁴C]- and [phenoxy-¹⁴C]-radiolabelled kresoxim-methyl and laying hens using [phenyl-¹⁴C]-radiolabelled kresoxim-methyl.

In lactating goats the transfer of radioactivity into milk and tissues was low. Highest TRR levels were found in liver and kidney, while in all other tissues TRR levels were one order of magnitude lower. Parent kresoxim-methyl was only found in fat at a low level. The predominant metabolites were 490M1, 490M2 and 490M9.

In laying hens transfer of radioactivity into tissues and eggs was low as well. Highest TRR levels were found in liver, while in all other tissues TRR levels were significant lower. Parent kresoxim-methyl was only detected in fat in significant amounts. Other metabolites occurring in amounts >10% TRR were 490M9 in liver, 490M15 in fat, 490M28 in liver, 490M48 in eggs, 490M51/490M66 in muscle and egg and 490M58 in skin.

Laboratory animals [WHO]

Lactating goats

The metabolic fate of [phenyl-¹⁴C]-radiolabelled kresoxim-methyl in lactating goats was investigated in the study by Giese (1992, Kresoxim_021), Jonas (1992, Kresoxim_022) and Mayer (1994, Kresoxim_022). The compound was administered orally to two lactating goats. Goat A received 7.1 ppm (0.26 mg/kg bw) for five consecutive days, while goat B received 454 ppm (21 mg/kg bw) for eight consecutive days. Excreta were collected once daily and milk was collected twice daily. The animals were sacrificed approximately 23 hours (goat A) or 4 hours (goat B) after the last dose. Liver, kidney, muscle, fat, bile and gastrointestinal tract were collected. All samples were stored frozen at least -20 °C until further processing.

Total radioactivity in liquid samples such as urine, milk and various extracts were directly measured by LSC. Tissue samples were either solubilized using Fluorosol tissue solubiliser or subjected to combustion prior to the determination of total radioactivity by LSC.

Pooled daily milk samples were extracted with methanol. Proteins and lactose were precipitated with acetone and the extract analysed by HPLC. Tissues samples of muscle, fat, liver and kidney were homogenised followed by extraction with methanol and water. The methanol extracts were further worked-up by partitioning with dichloromethane, ethyl acetate and acetonitrile/n-hexane, separately or in combination. Characterization of the extracts was carried out by HPLC against reference standards. Post extraction solids of fat were further extracted with toluene/water followed by protease treatment. All other tissue sample were directly digested with protease and also solubilized with water/25% ammonia (50+1, v/v).

The total recovery of the administered radioactivity was equal to 95–97%. The majority of the radioactivity was found in urine (59–70% AR), followed by faeces (18–25% AR). Radioactive residues in the edible matrices were highest in liver at 0.06–0.07% AR. A summary of the recovered radioactivity is presented in Table 17.

| Matrix | % AR (m | g eq/kg) |
|----------------------------------|------------------|------------------|
| Wattix | Goat A (7.1 ppm) | Goat B (454 ppm) |
| Faeces | 18 | 25 |
| Urine | 70 | 59 |
| Urine of bladder | 0.91 (2.2) | 0.26 (482) |
| Cage wash | 1.1 (0.11) | 1.5 (17) |
| Milk | 0.031 0.025) | 0.027 (2.7) |
| Liver | 0.06 (0.041) | 0.07 (6.8) |
| Kidney | 0.03 (0.15) | 0.02 (14) |
| Fat | 0.01 (0.001) | <0.005 (0.34) |
| Muscle | 0.01 (0.001) | 0.01 (0.22) |
| Bile | 0.03 (0.49) | 0.03 (14) |
| Stomach skin | 0.17 (0.064) | 0.08 (4.6) |
| Stomach contents | 1.1 (0.19) | 8.9 (95) |
| Intestine skin | 0.23 (0.11) | 0.08 (4.6) |
| Intestine contents | 3.1 (1.1) | 1.6 (48) |
| Stomach/intestine cleaning water | 0.09 (0.033) | 0.44 (6.8) |
| Blood | 0.03 (0.010) | 0.05 (1.9) |
| Vomited material | <0.005 (0.030) | 0.1 (10) |
| Tray water | <0.005 (0.000) | <0.005 (0.024) |
| Total | 95 | 97 |

Table 17 Recovered radioactive residues after oral administration of ¹⁴C-labelled kresoxim-methyl for 5 (goat A) or 8 (goat B) consecutive days lactating goats

In milk the total radioactivity increased over the whole dosing period, starting from 0.001 mg eq/kg (goat A) and 0.068 mg eq/kg (goat B) at day 1 (afternoon) to a terminal concentration of 0.003 mg eq/kg at day five (morning) for goat A and 0.213 mg eq/kg at day 8 (morning). The results are summarised in the following Table 18. Residue levels in milk reached a plateau in goat A after approximately 3 days. However, for goat B, which received the exaggerated dose, no plateau was reached (Figure 3).

Table 18 Recovered radioactive residues in milk after oral administration of ¹⁴C-labelled kresoxim-methyl for 5 (goat A) or 8 (goat B) consecutive days to goats

| TRR in milk | Goat A (7.1 ppm) | Goat B (454 ppm) | |
|---------------|------------------|------------------|--|
| Days | TRR in mg eq/kg | TRR in mg eq/kg | |
| 1 (afternoon) | 0.001 | 0.068 | |
| 2 (morning) | 0.002 | 0.156 | |
| 2 (afternoon) | 0.002 | 0.142 | |
| 3 (morning) | 0.003 | 0.155 | |
| 3 (afternoon) | 0.003 | 0.152 | |
| 4 (morning) | 0.003 | 0.176 | |
| 4 (afternoon) | 0.003 | 0.170 | |
| 5 (morning) | 0.002 | 0.206 | |
| 5 (afternoon) | 0.003 | 0.174 | |
| 6 (morning) | 0.003 | 0.208 | |
| 6 (afternoon) | n/a | 0.172 | |
| 7 (morning) | n/a | 0.215 | |
| 7 (afternoon) | n/a | 0.215 | |
| 8 (morning) | n/a | 0.316 | |
| 8 (afternoon) | n/a | 0.213 | |

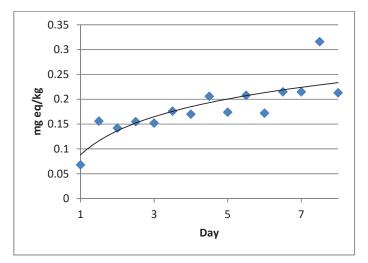


Figure 3 Time course of the concentrations of kresoxim-methyl in milk of goat B

The initial methanol and water extractions released between 63% TRR in liver and 101% TRR in milk. A summary of the results is presented in Table 19.

Table 19 Extractability of residues from milk and tissues of ¹⁴C-labelled kresoxim-methyl dosed lactating goat

| Fraction | Goat B (454 ppm) mg eq/kg (% TRR) | | | | | | |
|-------------------------------|--------------------------------------|------------|-------------|------------|------------|--|--|
| | Milk | Muscle | Fat | Liver | Kidney | | |
| Methanol extract | 0.19 (101) | 0.16 (72) | 0.26 (70) | 3.8 (58) | 13 (96) | | |
| Water extract | n/a | n/a | 0.008 (2.3) | 0.33 (5.0) | 0.32 (2.4) | | |
| Total extracts | 0.19 (101) | 0.16 (72) | 0.27 (72) | 4.1 (63) | 13 (98) | | |
| Post extraction solids | 0.007 (3.5) | 0.061 (27) | 0.064 (18) | 2.8 (43) | 0.34 (2.5) | | |
| Total recovered radioactivity | 0.20 (105) | 0.22 (99) | 0.33 (90) | 6.9 (106) | 14 (101) | | |

Kresoxim-methyl was extensively metabolized with parent only detected in fat at 6.6% TRR (0.024 mg eq/kg. Major metabolites in milk were 490M2 and 490M9 at 20% TRR (0.039 mg eq/kg) and 63% TRR (0.039 mg eq/kg), respectively. In tissues, 490M1 ranged between 14 and 26% TRR (0.059–3.0 mg eq/kg), 490M2 between 11 and 34% TRR (0.022–4.6 mg eq/kg) and 490M9 between 10 and 30% TRR (0.023–4.0 mg eq/kg). Other identified metabolites occurring at smaller fractions were 490M6, 490M18

and 490M19. Unextracted residues ranged between 0.3–9.0% TRR (0.007–0.46 mg eq/kg) (Table 20). The proposed metabolic pathway of kresoxim-methyl is shown in Figure 4.

| Fraction | | Goat B (454 ppm) mg eq/kg (% TRR) | | | | | | |
|--------------------------|-------------|--------------------------------------|-------------|-------------|-------------|--|--|--|
| | Milk | Muscle | Fat | Liver | Kidney | | | |
| TRR | 0.19 (100) | 0.23 (100) | 0.36 (100) | 6.6 (100) | 14 (100) | | | |
| Kresoxim-methyl | n/d | n/d | 0.024 (6.6) | n/d | n/d | | | |
| 490M1 | 0.003 (1.6) | 0.059 (26) | 0.084 (23) | 0.94 (14) | 3.0 (22) | | | |
| 490M2 | 0.039 (20) | 0.032 (14) | 0.089 (24) | 0.69 (11) | 4.6 (34) | | | |
| 490M6 | n/d | n/d | 0.029 (8.0) | 0.25 (3.7) | 0.49 (3.6) | | | |
| 490M9 | 0.12 (63) | 0.023 (10) | 0.001 (0.3) | 1.9 (29) | 4.0 (30) | | | |
| 490M18 | n/d | 0.017 (7.2) | 0.002 (0.7) | 0.22 (3.3) | 0.027 (0.2) | | | |
| 490M19 | n/d | 0.015 (6.6) | n/d | 0.022 (0.3) | n/d | | | |
| Characterized by HPLC | 0.031 (16) | 0.007 (3.2) | 0.012 (3.3) | 1.2 (18) | 0.92 (6.7) | | | |
| Aqueous extract | n/a | 0.011 (4.6) | 0.008 (2.3) | 0.12 (1.8) | n/a | | | |
| Ethyl acetate phase | n/a | 0.021 (9.0) | n/a | n/a | n/a | | | |
| Hexane phase | n/a | 0.009 (3.9) | n/a | n/a | n/a | | | |
| Post extraction solids | 0.007 (3.5) | 0.061 (27) | 0.064 (18) | 2.8 (43) | 0.34 (2.5) | | | |
| Toluene extract | n/a | n/a | 0.018 (5.1) | n/a | n/a | | | |
| Protease extract | n/a | n/a | 0.041 (11) | 0.77 (11) | 0.050 (0.4) | | | |
| Ammonia extract | n/a | 0.026 (11) | n/a | 0.32 (4.9) | n/a | | | |
| Total identified | 0.16 (85) | 0.15 (64) | 0.23 (63) | 4.1 (61) | 12 (90) | | | |
| Total characterized | 0.031 (16) | 0.074 (32) | 0.079 (22) | 2.4 (36) | 1.0 (7.1) | | | |
| Unextracted | 0.007 (3.5) | 0.021 (9.0) | 0.013 (3.5) | 0.46 (6.9) | 0.034 (0.3) | | | |
| Total ^a | 0.20 (105) | 0.24 (105) | 0.33 (88) | 7.0 (104) | 13 (97) | | | |

Table 20 Summary of identified/characterized residues in milk and tissues from ¹⁴C-labelled kresoxim-methyl dosed lactating goat

^a Includes part of different work-ups

In a second study by Kirkpatrick (1996, Kresoxim_020), the metabolic fate of [phenoxy-¹⁴C]-radiolabelled kresoximmethyl was investigated. The compound was administered for five consecutive days to one lactating goat by gavage at 13.9 ppm (1.6 mg/kg bw). Excreta were collected once daily, while milk was collected twice daily. The animals were sacrificed approximately 23 hours after the last dose. Liver, kidney, muscle, fat (omental, perirenal and subcutaneous), rumen, bile and gastrointestinal tract contents were collected. All samples were stored frozen at least -15 °C until further processing.

Total radioactivity in liquid samples such as urine, cage wash, milk and various extracts was directly measured by LSC. Tissue samples were either solubilized using NCSII tissue solubiliser or subjected to combustion prior to the determination of total radioactivity by LSC. Initial analysis was carried out within 6 weeks after sacrifice of the goat, while identification of metabolites and characterization of tissues occurred within 10 month after sample collection.

Urine samples were acidified with to pH 2 with hydrochloric acid prior to partitioning with dichloromethane. After semipreparative HPLC the fractions were cleaned-up by SPE on C18 columns and the eluate characterized by LC-MS/MS. Tissues samples of liver and kidney were homogenised followed by extraction with acetonitrile/water (1+1, v/v). Characterization of the extracts was carried out by HPLC against reference standards or TLC. Post extraction solids from liver were additionally digested with protease, followed by solubilisation with water/25% ammonia (50+1, v/v). Due to the low levels of radioactivity, milk, muscle and fat samples were not further analysed. Additionally, an aliquot of the liver homogenate was dialyzed with phosphate buffer.

The total recovery of the administered radioactivity was equal to 88%. The majority of the radioactivity was found in urine (67%), followed by faeces (20%) and the cage wash (1.5%). Radioactive residues in the edible matrices were highest in liver and kidney at 0.064 mg eg/kg and 0.052 mg eq/kg, respectively. A summary of the recovered radioactivity is presented in Table 21.

Table 21 Recovered radioactive residues after oral administration of ¹⁴C-labelled kresoxim-methyl for 5 consecutive days to a lactating goat

| Matrix | % AR | TRR in mg eq/kg |
|-----------|------|-----------------|
| Faeces | 20 | N/A |
| Urine | 67 | N/A |
| Cage wash | 1.5 | N/A |
| Milk | <0.1 | 0.003 |
| Liver | 0.1 | 0.064 |
| Kidney | <0.1 | 0.052 |

| Muscle | N/A | <0.003 |
|------------------|-----|--------|
| Omental fat | N/A | <0.003 |
| Perirenal fat | N/A | <0.003 |
| Subcutaneous fat | N/A | <0.003 |
| Bile | N/A | 0.718 |
| Whole blood | N/A | 0.015 |
| Plasma | N/A | 0.021 |
| Total | 88 | |

In milk the total radioactivity increased only slightly over the time of the study (Table 22). Residue levels in milk reached a plateau after approximated 3 days.

Table 22 Recovered radioactive residues in milk after oral administration of ¹⁴C-labelled kresoxim-methyl for 5 consecutive days to a lactating goat

| TRR in milk (hours after first dose) | TRR in mg eq/kg |
|---|-----------------|
| 0-24 (afternoon) | 0.003 |
| 0-24 (morning) | 0.002 |
| 24-48 (afternoon) | 0.004 |
| 24-48 (morning) | 0.002 |
| 48-72 (afternoon) | 0.005 |
| 48-72 (morning) | 0.002 |
| 72-96 (afternoon) | 0.005 |
| 72-96 (morning) | 0.003 |
| 96-119 (afternoon) | 0.004 |
| 96-119 (morning) | 0.003 |

The initial acetonitrile/water extractions released 86% TRR and 43% TRR in kidney and liver, respectively. Protease digestion and ammonia extraction of the liver residues remaining after aqueous acetonitrile extraction liberated another 14% TRR (0.009 mg eq/kg) and 23% TRR (0.014 mg eq/kg), respectively. A summary of the results is presented in Table 23.

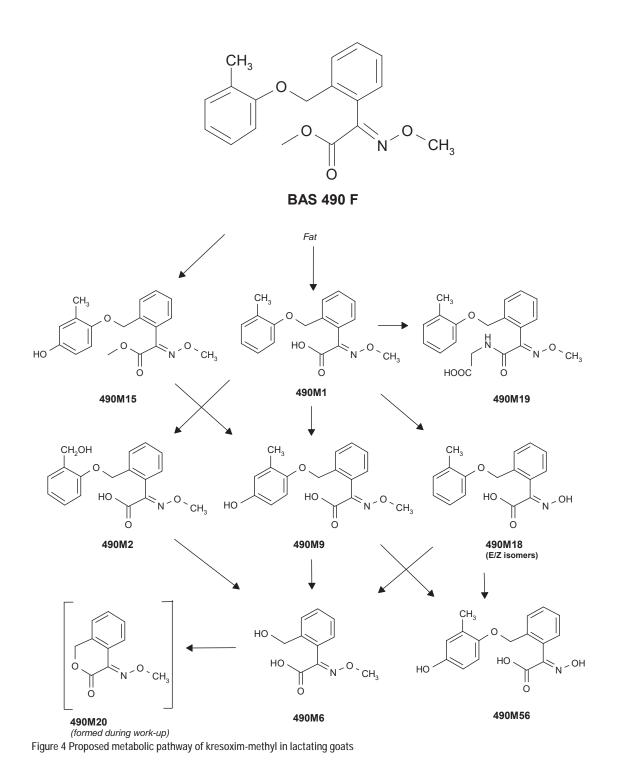
Table 23 Characterization of radioactivity in kidney and liver from a ¹⁴C-labelled kresoxim-methyl dosed lactating goat

| Fraction | Kidney | | Liver | | |
|--------------------------------------|----------|-------|----------|-------|--|
| Flaction | mg eq/kg | % TRR | mg eq/kg | % TRR | |
| Combined acetonitrile/water extracts | 0.045 | 86 | 0.027 | 43 | |
| Protease treatment | n/a | n/a | 0.009 | 14 | |
| Ammonia extraction | n/a | n/a | 0.014 | 23 | |
| Total extracts | 0.045 | 85 | 0.050 | 80 | |
| Unextracted | 0.008 | 15 | 0.013 | 20 | |
| Total recovered radioactivity | 0.053 | 100 | 0.063 | 100 | |

Kresoxim-methyl was extensively metabolized with no parent detected in edible tissues. The predominant metabolites in liver and kidney were 490M9 (17% TRR, 0.011 mg eq/kg) and 490M2 (34% TRR, 0.018 mg eq/kg), respectively. In liver, 58% TRR (0.036 mg eq/kg) remained unextracted (Table 24). As the result of the dialysis experiment resulted in a similar fraction of unextracted radioactivity (52% TRR), it was suggested that the unextracted radioactivity is covalently bound to liver macromolecules. The proposed metabolic pathway of kresoxim-methyl is shown in Figure 4.

Table 24 Summary of identified/characterized residues in kidney and liver from a 14C-labelled kresoxim-methyl dosed lactating goat

| Fraction | Kidney | | Liver | |
|-----------------------|----------|-------|----------|-------|
| Fraction | mg eq/kg | % TRR | mg eq/kg | % TRR |
| TRR | 0.052 | 100 | 0.064 | 100 |
| Kresoxim-methyl | n/d | n/d | n/d | n/d |
| 490M1 | 0.006 | 11 | 0.001 | 2.2 |
| 490M2 | 0.018 | 34 | 0.005 | 8.5 |
| 490M9 | 0.008 | 15 | 0.011 | 17 |
| Characterized by HPLC | 0.013 | 25 | 0.010 | 15 |
| Total identified | 0.032 | 60 | 0.017 | 28 |
| Total characterized | 0.013 | 25 | 0.010 | 15 |
| Unextracted | 0.008 | 15 | 0.036 | 58 |
| Total | 0.053 | 100 | 0.063 | 101 |



Laying hens

The metabolic fate of [phenyl-¹⁴C]-radiolabelled kresoxim-methyl in laying hens was investigated in the study by Burke (1994, Kresoxim_024) and Grosshans (1994, Kresoxim_025). The compound was administered for 6 consecutive days to 2 groups of laying hens by gavage. Group A was dosed daily at 10 ppm (1 mg/kg bw) and group B at 180 ppm (18 mg/kg bw). Excreta were collected

once daily while eggs were collected twice daily. The hens were sacrificed approximately 23 hours (group A) and 3 hours (group B) after the last dose. Liver, muscle, fat, kidney, skin and gastrointestinal tract with contents were collected. All samples were pooled per treatment group, homogenised and stored frozen at least -18 °C until further processing.

Total radioactivity in liquid samples such as egg, plasma, cage wash and various extracts were directly measured by LSC. Fat samples were solubilized using Biolute-S tissue solubiliser followed by LSC. All other samples were subjected to combustion prior to the determination of total radioactivity by LSC.

Pooled samples of egg, liver, muscle and excreta were extracted with methanol and water. The radioactivity in the extracts was characterized by liquid/liquid partition with cyclohexane, dichloromethane, ethyl acetate and acetonitrile/n-hexane, separately or in combination, and analysed by HPLC. Bound radioactivity in the post extraction solids was released by pronase treatment.

The total recovery of the administered radioactivity was identical in both treatment groups with 88% AR recovered. The majority of the radioactivity was found in excreta (72–83%). In the high dose group, radioactivity in tissues accounted for 11%, while in the low dose group for less than 1%.

Table 25 Recovered radioactive residues after oral administration of ¹⁴C-labelled kresoxim-methyl for 6 consecutive days to laying hens

| Matrix | % AI | R (mg eq/kg) |
|----------------|---------------|---------------|
| IVIALITX | Group A (ppm) | Group B (ppm) |
| Excreta | 83 | 72 |
| Cage wash | 4.9 | 5.2 |
| Eggs | 0.02 | 0.02 |
| Tissues | 0.62 | 11 |
| Peritoneal fat | n/d | <0.01 (0.76) |
| Kidney | 0.01 (0.065) | 0.04 (6.4) |
| Liver | 0.02 (0.082) | 0.14 (7.0) |
| Skin | <0.01 (0.009) | <0.01 (0.68) |
| Muscle | n/d | <0.01 (0.20) |
| GI tract | 0.59 | 11 |
| Total | 88 | 88 |

TRR levels in eggs did not reach a plateau after 6 days of consecutive dosing of ¹⁴C-labelled kresoxim-methyl. A summary of the recovered radioactivity is presented in Table 26.

Table 26 Recovered radioactive residues in eggs after oral administration of ¹⁴C-labelled kresoxim-methyl for 6 consecutive days to laying hens

| Days | TRR in mg eq/kg | | | |
|----------------------------|------------------|-------------------|--|--|
| Days | Group A (10 ppm) | Group B (180 ppm) | | |
| 1 | n/d | 0.1 | | |
| 2 | 0.009 | 0.086 | | |
| 3 | 0.007 | 0.13 | | |
| 4 | 0.009 | 0.16 | | |
| 5 | 0.011 | 0.22 | | |
| 6 | 0.012 | n/a | | |
| Pooled sample ^a | 0.009 | 0.16 | | |

^a Used for characterization

The initial methanol and water extractions released 65–66% TRR from liver and skin of the low dose group and 76–92% TRR from eggs, liver, muscle, skin and fat of the high dose group. A summary of the results for the low and high dose group is presented in Table 27 and Table 28, respectively.

| Fraction | Group A (10 ppm) mg eq/kg (% TRR) | |
|-------------------------------|--------------------------------------|-------------|
| | Liver | Skin |
| Methanol extract | 0.042 (59) | 0.006 (57) |
| Water extract | 0.004 (5.7) | 0.001 (9.0) |
| Total extracts | 0.046 (65) | 0.007 (66) |
| Post extraction solids | 0.025 (35) | 0.004 (34) |
| Total recovered radioactivity | 0.071 (100) | 0.011 (100) |

Table 27 Extractability of residues from eggs and tissues of ¹⁴C-labelled kresoxim-methyl dosed laying hens Group A)

Table 28 Extractability of residues from eggs and tissues of ¹⁴C-labelled kresoxim-methyl dosed laying hens Group B)

| Fraction | | Group B (180 ppm) mg eg/kg (% TRR) | | | | |
|-------------------------------|--------------|---------------------------------------|-------------|-------------|-------------|--|
| | Egg (pooled) | Liver | Muscle | Skin | Fat | |
| Methanol extract | 0.11 (76) | 5.3 (77) | 0.14 (81) | 0.57 (83) | 0.68 (90) | |
| Water extract | n/d | 0.38 (5.5) | 0.005 (2.7) | 0.026 (3.7) | 0.012 (1.6) | |
| Total extracts | 0.11 (76) | 5.7 (82) | 0.15 (84) | 0.60 (87) | 0.69 (92) | |
| Post extraction solids | 0.035 (24) | 1.2 (18) | 0.027 (16) | 0.090 (13) | 0.061 (8.1) | |
| Total recovered radioactivity | 0.15 (100) | 6.9 (100) | 0.18 (100) | 0.69 (100) | 0.75 (100) | |

Parent kresoxim-methyl was identified in muscle, skin fat and eggs, ranging between 2.7% TRR in muscle and 41% TRR in fat. Metabolites occurring in significant amounts were 490M9 in liver (20% TRR), 490M15 (free and conjugated) in fat (17% TRR) and liver (14%), 490M48 in eggs (11% TRR), 490M51 (sulphate of 490M46) or 490M66 (sulphate of 490M15) in muscle (20% TRR) and egg (16% TRR) and 490M58 in skin (10% TRR) (Table 29). The proposed metabolic pathway of kresoxim-methyl is shown in Figure 5.

Table 29 Summary of identified/characterized residues in eggs and tissues from ¹⁴C-labelled kresoxim-methyl dosed laying hens (high dose group)

| Fraction | Group B (180 ppm) mg eg/kg (% TRR) | | | | | |
|--|---------------------------------------|--------------|-------------|-------------|--------------|--|
| | Liver | Muscle | Skin | Fat | Eggs | |
| TRR | 6.7 (100) | 0.17 (100) | 0.76 (100) | 0.74 (100) | 0.12 (100) | |
| Identified | | | | | | |
| Kresoxim-methyl | n/d | 0.0047 (2.7) | 0.082 (11) | 0.31 (41) | 0.010 (8.3) | |
| 490M1 | n/d | n/d | n/d | n/d | 0.0049 (4.0) | |
| 490M14 (glucuronide of 490M1) | 0.18 (2.7) | n/d | n/d | n/d | n/d | |
| 490M5 | 0.30 (4.5) | 0.0064 (3.7) | 0.017 (2.2) | n/d | n/d | |
| 490M6 | n/d | n/d | 0.015 (2.0) | n/d | n/d | |
| 490M9 | 1.4 (20) | n/d | 0.034 (4.5) | n/d | n/d | |
| 490M50 (sulphate of 490M8) /490M52 (sulphate of 490M9 | n/d | 0.0085 (4.9) | n/d | 0.019 (2.6) | 0.006 (4.9) | |
| 490M25 (glucuronide of 490M2) /490M26 (glucuronide of 490M9) | n/d | 0.0038 (2.2) | n/d | n/d | 0.004 (3.3) | |
| 490M8/490M11 | n/d | 0.0019 (1.1) | n/d | n/d | n/d | |
| 490M15 | 0.18 (2.6) | 0.0041 (2.4) | 0.031 (4.1) | 0.12 (17) | n/d | |
| 490M28 (glucuronide of 490M15 | 0.92 (14) | 0.0026 (1.5) | n/d | n/d | n/d | |
| 490M20/490M60 | n/d | 0.015 (8.6) | n/d | n/d | n/d | |
| 490M24 | n/d | n/d | 0.013 (1.8) | 0.011 (1.4) | n/d | |
| 490M31 (glucuronide of 490M24) | n/d | n/d | n/d | n/d | 0.012 (9.7) | |
| 490M33 (glucoronide of 490M4) /490M39 (glucuronide of 490M57) | 0.20 (3.0) | n/d | n/d | n/d | 0.0051 (4.2) | |

| | Group B (180 pp | | | | |
|--|-----------------|--------------|-------------|-------------|--------------|
| Fraction | mg eq/kg (% TRF | · 1 | Chin | L E et | L Fano |
| 490M46 | Liver | Muscle | Skin | Fat | Eggs |
| | n/d | 0.0068 (3.9) | 0.043 (5.6) | 0.033 (4.5) | n/d |
| 490M63/490M67 (glucuronides of 490M46) | 0.62 (9.2) | n/d | n/d | n/d | 0.003 (2.5) |
| 490M51 (sulphate of 490M46) /490M66 (sulphate of 490M15) | n/d | 0.035 (20) | 0.030 (4.0) | 0.014 (1.9) | 0.019 (16) |
| 490M47 | 0.21 (3.2) | 0.0030 (1.7) | 0.014 (1.9) | n/d | 0.0028 (2.3) |
| 490M48 | n/d | 0.0032 (1.8) | 0.044 (5.7) | 0.057 (7.7) | 0.014 (11) |
| 490M56 | n/d | n/d | n/d | 0.024 (3.2) | n/d |
| 490M57 | n/d | 0.0028 (1.6) | 0.010 (1.3) | n/d | n/d |
| 490M58 | 0.076 (1.1) | 0.0097 (5.6) | 0.079 (10) | 0.013 (1.7) | n/d |
| 490M59 | 0.053 (0.8) | 0.0036 (2.1) | 0.060 (7.9) | 0.006 (0.8) | n/d |
| 490M64 | n/d | n/d | n/d | n/d | 0.0038 (3.1) |
| Methanol extract | | | | | |
| Characterized by HPLC | 1.1 (16) | 0.0086 (5.0) | 0.12 (16) | 0.037 (5.2) | 0.016 (13) |
| Aqueous extract | • | • | • | | I |
| Characterized by HPLC | 0.23 (3.5) | n/d | n/a | n/a | n/a |
| Liquid/liquid partitioning | | | | | |
| Aqueous phase | n/a | 0.010 (5.8) | n/a | n/a | n/a |
| Hexane phase | n/a | 0.011 (6.4) | n/a | n/a | n/a |
| Post extraction solids | 1.3 (19) | 0.037 (21) | n/a | n/a | 0.023 (19) |
| Protease incubation | 1.1 (16) | 0.031 (17) | 0.093 (12) | 0.053 (7.1) | 0.031 (21) |
| Total identified | 4.1 (61) | 0.11 (64) | 0.47 (62) | 0.61 (82) | 0.084 (69) |
| Total characterized | 2.4 (36) | 0.061 (34) | 0.21 (28) | 0.090 (12) | 0.047 (34) |
| Unextracted | 0.23 (3.3) | 0.007 (3.8) | 0.040 (5.3) | 0.008 (1.1) | 0.008 (5.5) |
| Total ^a | 6.7 (100) | 0.18 (102) | 0.72 (95) | 0.71 (95) | 0.14 (109) |

^a Includes part of different work-ups

Figure 5 Proposed metabolic pathway of kresoxim-methyl in laying hens

ENVIRONMENTAL FATE

For the investigation of the environmental fate of kresoxim-methyl, the Meeting received new studies on the degradation of kresoxim-methyl in soil and the behaviour in confined rotational crops.

Studies provided in the context of the first JMPR evaluation in 1998 were not resubmitted. Therefore, it is referred to the respective evaluation report.

Anaerobic and aerobic degradation in soil

In the study by Platz & Schriever (2008, Kresoxim_026) and its addendum by Schriever (2010, Kresoxim_027), kinetic evaluations were preformed to investigate rate of degradation of parent kresoxim-methyl and metabolite 490M1. These kinetic evaluations followed the latest guidance of FOCUS and were based on laboratory soil degradation studies provided during the first JMPR evaluation in 1998.

The results of the kinetic evaluation showed that the Single First Order (SFO) kinetic model was generally appropriate to describe the observed laboratory anaerobic and aerobic degradation behaviour of kresoxim-methyl. Degradation behavior observed from Holy Springs data was best described by the First Order Multi Compartment (FOMC) kinetic model for the parent and SFO kinetic model for the metabolite. For the Karup soil data, a Double first-order in parallel (DFOP) kinetic model was applied.

The estimated degradation parameters DT_{50} and DT_{90} for parent and metabolite are summarised in Table 30. For kresoxim-methyl and its metabolite 490M1 in aerobic soil, the DT_{50} ranged from 0.48–3.1 days and 23–86 days, respectively. In anaerobic soil DT_{50} was similar for kresoxim-methyl with 0.98 days, while for 490M1 the DT_{50} was significantly longer with 396 days.

| | Kresoxim-methy | /I | | 490M1 | | | | Defense |
|-----------------|----------------|-----------------------------|-------------------------|----------------|-----------------------------|-----------------------------|--|----------------------------|
| Site | Best-fit model | SFO-DT ₅₀ [d] | DT ₉₀ [d] | Best-fit model | SFO-DT ₅₀ [d] | SFO-DT ₉₀ [d] | Formation fraction (%) ^b | Reference (BASF DocID) |
| Anaerobic stud | y data | | | | | | | |
| Bruch West | SF0 | 0.29 | 0.98 | SF0 | 396 | >1000 | 93 | 1994/10165 |
| Aerobic study d | ata | | | | | | | |
| Bruch West | SF0 | 0.56 | 1.8 | SF0 | 46 | 153 | 89 | 1994/10165 |
| Holly Springs | FOMC | 0.36 ^a | 10 | SF0 | 59 | 196 | 94 | 1994/10512 |
| Bruch West | SF0 | 0.48 | 1.6 | SF0 | 36 | 121 | 90 | 1994/10729; 1995/10431; |
| Borris | - | - | - | SF0 | 51 | 169 | - | 1998/11195 |
| Karup | - | - | - | DFOP | 23 | 288 | - | 1998/11195 |
| Langvad | - | - | - | SF0 | 86 | 285 | - | 1998/11195 |

Table 30 Kinetic endpoints for kresoxim-methyl and metabolite 490M1 according to FOCUS

^a SFO-DT50 back-calculated from the bi-phasic DT90, back-calculation of SFO value would be 3.11

^b Faction of 490M1 formed from kresoxim-methyl

Kinetic evaluations to investigate the rate of degradation of parent kresoxim-methyl and metabolite 490M1 were performed in the study by van Beinum & Beulke (2008, Kresoxim_029), using field dissipation data provided during the first JMPR evaluation in 1998. These kinetic evaluations followed the latest guidance of FOCUS.

Kresoxim-methyl transformed rapidly to its metabolite 490M1. Dissipation endpoints for 490M1 were derived from fitting single first-order kinetics (SFO), the First-Order Multi-Compartment model (FOMC) and the Double-First-Order in Parallel model (DFOP).

Table 31 shows the DT_{50} and DT_{90} values for metabolite 490M1 from the best model description for each data set. DT_{50} values ranged from 2.9–37 days and DT_{90} values ranged from 16–286 days.

| Country | Location | DT ₅₀ (days) | DT ₉₀ (days) | Model | Reference (BASF DocID) |
|-----------|------------------|-------------------------|-------------------------|------------------|---------------------------|
| | Birkenheide | 7.3 | 24 | SF0 ^a | 1994/10878 |
| Commonwei | Brockhausen | 4.9 | 16 | SF0 ^a | 1994/10878 |
| Germany | Oberding | 37 | 124 | SF0 | 1994/10878 |
| | Niederhofen | 14 | 47 | SFO | 1994/10878 |
| | British Columbia | 30 | 286 | DFOP | 1997/5071 |
| Canada | Nova Scotia | 18 | 60 | SF0 | 1997/5071 |
| | Ontario | 2.9 | 54 | DFOP | 1997/5071 |
| | California | 7.6 | 25 | SF0 | 1996/5172 |
| USA | New York | 13 | 127 | FOMC | 1996/5172 |
| | Oregon | 7.7 | 51 | FOMC | 1996/5172 |

^a Bi-phasic models not evaluated due to limited number of data points

In a second the study by Gottesbueren (2008, Kresoxim_028), kinetic evaluations were performed using normalized degradation data from field dissipation studies provided during the first JMPR evaluation in 1998, in order to investigate rate of degradation of metabolite 490M1 and formation and degradation of 490M04. These kinetic evaluations were following the latest guidance of FOCUS. The dissipation of kresoxim-methyl was very fast (DT_{50} of < 1 day). Therefore, a meaningful evaluation of the dissipation rate of kresoxim-methyl and hence formation rate of metabolite 490M1 was not possible.

Degradation rate constants of 490M1 and 490M04 from the field were normalization to a reference temperature of 20 °C and reference soil moisture at pF2. Half-lives were calculated on the basis of these normalized degradation rate constants.

The normalized DT_{50} values of the acid metabolite 490M1 ranged from 3.6–26 days, with a geometric mean of 8.8 days. The fractions of 490M4, which is formed from 490M1, ranged from 32–61% with an arithmetic mean of 51%. The normalized DT_{50} values of 490M4 ranged from 1.0–3.9 days, with a geometric mean of 2.7 days (Table 32).

| Site | 490M1 DegT _{50, norm} [d] | 490M4 DegT _{50, norm} [d] | Formation fraction of BF 490-5 (%) | Reference (BASF DocID) |
|------------------|--|--|--|---------------------------|
| Niederhofen | 11 | n | ı/a | 1994/10878 |
| Birkenheide | 4.7 | n | ı/a | 1994/10878 |
| Oberding | 26 | n | ı/a | 1994/10878 |
| Brockhausen | 3.6 | n | n/a | 1994/10878 |
| New York | 12 | 3.5 | 61 | 1996/5172 |
| Oregon | 8.3 | 3.7 | 50 | 1996/5172 |
| California | 9.2 | n | n/a | 1996/5172 |
| Nova Scotia | 12 | 3.9 | 61 | 1997/5071 |
| Ontario | 6.8 | r | n/a | 1997/5071 |
| British Columbia | 8.1 | 1.0 | 32 | 1997/5071 |
| Geometric mean | 8.8 | 2.7 | | |
| Arithmetic mean | | | 51 | |

Table 32 Normalised SFO half-lives of 490M1 and 490M4 as well as formation fractions for 490M4

Confined rotational crops

A confined rotational crop study by Hofmann (1993, Kresoxim_031, 1993a, Kresoxim_032) and Grosshans (1994, Kresoxim_033) was conducted with [phenyl-¹⁴C]-radiolabelled kresoxim-methyl applied at a rate of 0.3 kg ai/ha to a sandy loam soil. After plantback intervals (PBIs) of 30 days the nature and level of radioactive residues were investigated in spring wheat (variety Star, Ralle), green beans (variety Maxi, Marona), carrots (variety Nantaise Frühbund) and lettuce (variety Ultra, Ovation).

Crops were harvested at immature stages and at maturity. All samples were stored in a freezer at -18 °C until further processing.

Homogenised plant samples were combusted prior to the determination of total radioactivity by LSC, while liquid samples such as extracts were directly measured by LSC. In order to characterise and identify the radioactivity present, all samples were extracted with methanol followed by dilute aqueous ammonia. The extractable radioactivity was characterized by liquid-liquid partitioning with cyclohexane or dichloromethane (wheat straw) and ethyl acetate. Conjugates were cleaved by enzymes (hesperidinase and b-glucoside). HPLC against reference compounds was applied for the identification of the radioactivity.

At harvest, the highest total radioactive residues were found in the roots of lettuce, beans and wheat, ranging from 0.23– 1.1 mg eq/kg. This was followed by bean forage at 0.21 mg eq/kg and wheat straw at 0.15 mg eq/kg. In edible commodities the radioactive residues were lower ranging from 0.006 mg eq/kg in carrot root to 0.02 mg eq/kg in lettuce heads (Table 33). Due to the low radioactivity only immature lettuce, carrot greens, bean forage, wheat forage and straw were further characterized.

Table 33 Total radioactive residues in rotational crops after application of [phenyl-¹⁴C]-radiolabelled kresoxim-methyl to bare soil at 0.3 kg ai/ha and 30 days PBI

| Sample | Sampling at days after sowing/planting | TRR determined by direct combustion, mg eq/kg | TRR calculated, mg eq/kg ^a |
|------------------------|--|--|---------------------------------------|
| Soil | | | |
| Initial | | 0.92, 0.83, 1.0 | n/a |
| After 30 days ageing | | 0.049, 0.066, 0.063 | n/a |
| Lettuce | | | |
| Leaves/head (immature) | 26, 30, 56 | 0.053, 0.051, 0.028 | 0.030-0.032 |
| Head | 66, 49 | 0.010, 0.020 | n/a |
| Roots (immature) | 56 | 0.57 | n/a |
| Roots | 66 | 0.23 | n/a |
| Soil (immature) | 56 | 0.033 | n/a |
| Soil | 66 | 0.032 | n/a |
| Carrots | | | |
| Plant (immature) | 50 | 0.025 | n/a |
| Greens (immature) | 52, 89 | 0.061, 0.056 | 0.026 |
| Greens | 111,90 | 0.047, 0.010 | 0.035 |
| Tap root (immature) | 52,89 | 0.052, 0.005 | n/a |
| Tap root | 111, 90 | 0.006, 0.003 | n/a |
| Soil | 111, 90 | 0.036, 0.034 | n/a |
| Beans | | | |
| Forage (immature) | 26 | 0.36 | n/a |

| Sample | Sampling at days after sowing/planting | TRR determined by direct combustion, mg eq/kg | TRR calculated, mg eq/kg ^a |
|------------------------|--|--|---------------------------------------|
| Forage | 77, 75 | 0.21, 0.067 | 0.057-0.20 |
| Green beans (immature) | 56 | 0.004 | n/a |
| Green beans | 77, 75 | 0.009, 0.004 | n/a |
| Root | 77 | 1.1 | n/a |
| Dry beans | 77 | 0.011 | n/a |
| Pods | 77 | 0.062 | n/a |
| Soil | 77, 75 | 0.042, 0.028 | n/a |
| Wheat | | | |
| Forage (immature) | 42, 61 | 0.067, 0.23 | 0.050 |
| Grain | 124, 125 | 0.006, 0.007 | n/a |
| Husks | 124 | 0.030 | n/a |
| Straw | 124, 125 | 0.12, 0.068 | 0.077–0.15 |
| Chaff | 125 | 0.027 | n/a |
| Roots | 124, 125 | 0.73, 0.18 | n/a |
| Soil | 124, 125 | 0.050, 0.029 | n/a |

^a Sum of extracts (methanol and ammonia) and post-extraction residue (PES).

The radioactivity found in the fractions from the initial methanol extractions and in the unextracted remainder is presented in Table 34. Except for wheat forage at least 54% TRR or more was extracted from immature lettuce, carrot greens, bean forage and wheat straw.

Table 34 Extractability of radioactive residues from rotational crops after application of [phenyl-14C]-radiolabelled kresoxim-methyl to bare soil at 0.3 kg ai/ha and 30 days PBI

| Sampla | TDD mg/kg | Methanol extract | | Post extraction solids | |
|--------------------|-------------|------------------|-------|------------------------|-------|
| Sample | TRR mg/kg | mg/kg | % TRR | mg/kg | % TRR |
| Lettuce (immature) | 0.030-0032 | 0.021-0.023 | 71–77 | 0.007-0.009 | 23–29 |
| Carrot greens | 0.026-0.035 | 0.014-0.020 | 54–57 | 0.012-0.015 | 43–46 |
| Bean forage | 0.057-0.20 | 0.047-0.13 | 67–82 | 0.010-0.064 | 18–33 |
| Wheat forage | 0.050 | 0.019 | 38 | 0.031 | 62 |
| Wheat straw | 0.077-0.15 | 0.041-0.076 | 54-59 | 0.035-0.053 | 41–46 |

The results of the identification and characterization of radioactive residues are presented in Table 35. Parent kresoximmethyl was only found in lettuce (0.003 mg eq/kg, 9.4% TRR). As major metabolites, 490M2 (or conjugate thereof) was found in bean forage (0.038 mg eq/kg, 20% TRR) and 490M9 (or conjugate thereof) in carrot forage (0.010 mg eq/kg, 28% TRR), bean forage (0.036 mg eq/kg, 19% TRR) and wheat straw (0.013 mg eq/kg, 10% TRR). Additionally, multiple other unknown metabolites were characterized by HPLC. Metabolite 490M78, detected in significant amounts in the second study, was not analysed since no reference standard was available.

Table 35 Summary of identified/characterized residues in rotation crops following application of [phenyl-¹⁴C]-radiolabelled kresoxim-methyl

| Fraction / | Radioactive residues in mg eq/kg (% TRR) | | | | | |
|------------------------------|--|---------------|-------------|---------------|--------------|--|
| Solubilisate | Lettuce | Carrot greens | Bean forage | Wheat forage | Wheat straw | |
| | (immature) | | | | | |
| TRR | 0.030 (100%) | 0.035 (100%) | 0.20 (100%) | 0.050 (100%) | 0.13 (100%) | |
| Kresoxim-methyl | 0.003 (9.4%) | n/d | n/d | n/d | n/d | |
| 490M1 | n/d | 0.005 (13%) | n/d | n/d | n/d | |
| 490M2 (or conjugate thereof) | 0.009 (30%) | 0.002 (5.2%) | 0.038 (20%) | 0,0012 (2.5%) | 0.006 (4.8%) | |
| 490M9 (or conjugate thereof) | 0.004 (14%) | 0.010 (28%) | 0.036 (19%) | 0.0030 (6.2%) | 0.013 (10%) | |
| Total identified | 0.016 (53%) | 0.016 (46%) | 0.074 (38%) | 0.0042 (8.7%) | 0.020 (15%) | |
| Characterized by HPLC | 0.006 (20%) | 0.012 (35%) | 0.096 (49%) | 0,0098 (19%) | 0.086 (67%) | |
| Ammonia solubilizate | 0.006 (20%) | n/a | n/a | 0.017 (34%) | n/a | |
| Total characterized | 0.012 (40%) | 0.012 (35%) | 0.096 (49%) | 0.027 (53%) | 0.086 (67%) | |
| Unextracted | 0.004 (13%) | 0.005 (14%) | 0.027 (14%) | 0.019 (38%) | 0.020 (16%) | |
| Total | 0.032 (106%) | 0.033 (95%) | 0.20 (101%) | 0.050 (100%) | 0.13 (98%) | |

A second confined rotational crop study by Kampke-Thiel & Deppermann (2011, Kresoxim_030) was conducted with [phenyl-¹⁴C]-radiolabelled kresoxim-methyl applied at a rate of 1.5 kg ai/ha to a sandy loam soil. After plant-back intervals (PBIs) of 30, 120 and 365 days the nature and level of radioactive residues were investigated in lettuce (variety Matilda and Lucan), white radish (variety April Cross) and wheat (variety Thasos).

Lettuce plants were sampled when immature (28 days after planting), and at normal maturity (53–59 days after planting). Radish plants were sampled when immature (14 days after sowing) and at maturity (77–84 days after sowing). Spring wheat was sampled at forage stage (50–61 days after sowing), and at maturity (123–158 days after sowing). All samples were stored in a freezer at -18 °C until further processing.

Homogenised plant samples were combusted prior to the determination of total radioactivity by LSC, while liquid samples such as extracts were directly measured by LSC. In order to characterize and identify the radioactivity present, all samples were extracted with three times methanol followed by two times with water. The methanol extract of plant matrices from the 30 day PBI was additionally evaporated to the aqueous remainder partitioned with dichloromethane and ethyl acetate. Post extraction solids were further extracted with 1% ammonia followed by solubilization with enzymes (macerozyme, amylase/amyloglucosidase, β -glucosidase/hesperidinase, tyrosinase/laccase) as well as acidic (1 M or 6 M HCl) and alkaline hydrolysis (1 M NaOH). HPLC against reference compounds were applied for the characterisation and identification of the radioactivity.

TRR levels found in the model crops generally declined with longer PBIs. Levels for all PBI were highest in wheat straw, followed by wheat chaff and radish root. A summary of all TRRs found is presented in Table 36.

| Plant back | Sampling at days after | TRR determined by direct | TRR calculated, mg eg/kg ^a |
|-------------------|------------------------|--------------------------|---------------------------------------|
| interval | sowing/planting | combustion, mg eq/kg | TRR Calculated, mg eq/kg |
| Immature lettuce | | | |
| 30 DAT | 28 | 0.094 | 0.089 |
| Mature lettuce | | | |
| 30 DAT | 56 | 0.098 | 0.049 |
| 120 DAT | 59 | 0.096 | 0.094 |
| 365 DAT | 53 | 0.012 | 0.012 |
| Immature radish | (whole plant) | | |
| 30 DAT | 14 | 0.41 | 0.44 |
| White radish top | | | |
| 30 DAT | 84 | 0.54 | 0.51 |
| 120 DAT | 81 | 0.095 | 0.092 |
| 365 DAT | 77 | 0.046 | 0.042 |
| White radish root | | | |
| 30 DAT | 84 | 0.18 | 0.21 |
| 120 DAT | 81 | 0.040 | 0.038 |
| 365 DAT | 77 | 0.011 | 0.012 |
| Wheat forage | | | |
| 30 DAT | 50 | 0.33 | 0.30 |
| 120 DAT | 61 | 0.062 | 0.056 |
| 365 DAT | 61 | 0.022 | 0.021 |
| Wheat chaff | | | |
| 30 DAT | 123 | 0.63 | 0.64 |
| 120 DAT | 145 | 0.16 | 0.17 |
| 365 DAT | 158 | 0.099 | 0.099 |
| Wheat straw | | | |
| 30 DAT | 123 | 2.0 | 1.6 |
| 120 DAT | 145 | 0.36 | 0.33 |
| 365 DAT | 158 | 0.17 | 0.16 |
| Wheat grain | | | |
| 30 DAT | 123 | 0.41 | 0.38 |
| 120 DAT | 145 | 0.10 | 0.10 |
| 365 DAT | 158 | 0.066 | 0.066 |

Table 36 Total radioactive residues in rotational crops after application of [phenyl-¹⁴C]-radiolabelled kresoxim-methyl to bare soil at 1.5 kg ai/ha

^a Sum of extracts (methanol and water) and post-extraction residue (PES).

The radioactivity found in the fractions from the initial methanol/water extractions and in the unextracted remainder is presented in Table 37. At least 49% TRR or more was extracted from immature and mature lettuce, radishes, wheat forage and

chaff at 30 days PBI and wheat straw at 30 and 120 days PBI. Extractability was lowest for wheat grain for all PBIs, ranging between 16–17% TRR.

Table 37 Extractability of radioactive residues from rotational crops after application of [phenyl-14C]-radiolabelled kresoxim-methyl to bare soil at 1.5 kg ai/ha

| Plant back | TRR | Methanol | extract | Water extr | act | ERR ^a | | Post extraction solids | |
|---------------|-----------------|----------|---------|------------|-------|------------------|-------|------------------------|-------|
| interval | mg/kg | mg/kg | % TRR | mg/kg | % TRR | mg/kg | % TRR | mg/kg | % TRR |
| Immature let | tuce | | | | | | | | |
| 30 DAT | 0.089 | 0.058 | 64 | 0.003 | 2.9 | 0.060 | 67 | 0.029 | 33 |
| Mature lettuc | e. | • | | • | • | | | | • |
| 30 DAT | 0.049 | 0.033 | 67 | 0.003 | 5.5 | 0.036 | 73 | 0.013 | 27 |
| 120 DAT | 0.094 | 0.041 | 44 | 0.005 | 4.8 | 0.046 | 49 | 0.049 | 51 |
| 365 DAT | 0.012 | 0.005 | 44 | 0.001 | 5.3 | 0.006 | 49 | 0.006 | 51 |
| Immature rad | lish (whole pla | ant) | | | | | | | |
| 30 DAT | 0.44 | 0.33 | 75 | 0.014 | 3.2 | 0.342 | 78 | 0.097 | 22 |
| White radish | top | | | | | | | | |
| 30 DAT | 0.51 | 0.384 | 76 | 0.058 | 12 | 0.44 | 87 | 0.065 | 13 |
| 120 DAT | 0.092 | 0.053 | 58 | 0.009 | 9.3 | 0.062 | 68 | 0.030 | 32 |
| 365 DAT | 0.042 | 0.027 | 64 | 0.003 | 8.2 | 0.030 | 72 | 0.012 | 28 |
| White radish | root | | | | | | | | |
| 30 DAT | 0.21 | 0.15 | 71 | 0.004 | 1.9 | 0.15 | 73 | 0.056 | 27 |
| 120 DAT | 0.038 | 0.024 | 62 | 0.002 | 4.2 | 0.025 | 66 | 0.013 | 34 |
| 365 DAT | 0.012 | 0.007 | 59 | 0.000 | 2.7 | 0.007 | 62 | 0.004 | 38 |
| Wheat forage | | • | | | | | | | |
| 30 DAT | 0.30 | 0.22 | 74 | 0.015 | 4.9 | 0.24 | 79 | 0.064 | 21 |
| 120 DAT | 0.056 | 0.024 | 43 | 0.002 | 3.4 | 0.026 | 47 | 0.030 | 53 |
| 365 DAT | 0.021 | 0.007 | 33 | 0.001 | 4.6 | 0.008 | 37 | 0.013 | 63 |
| Wheat chaff | | - | | | | | | | |
| 30 DAT | 0.64 | 0.23 | 36 | 0.13 | 20 | 0.35 | 56 | 0.28 | 44 |
| 120 DAT | 0.17 | 0.045 | 27 | 0.018 | 11 | 0.064 | 38 | 0.11 | 62 |
| 365 DAT | 0.099 | 0.012 | 12 | 0.013 | 13 | 0.025 | 25 | 0.074 | 75 |
| Wheat straw | | - | | | | | | | |
| 30 DAT | 1.6 | 0.97 | 60 | 0.25 | 15 | 1.2 | 75 | 0.41 | 25 |
| 120 DAT | 0.33 | 0.13 | 38 | 0.042 | 13 | 0.17 | 51 | 0.16 | 49 |
| 365 DAT | 0.16 | 0.043 | 26 | 0.021 | 13 | 0.064 | 39 | 0.10 | 61 |
| Wheat grain | | | | | | - | | | |
| 30 DAT | 0.38 | 0.031 | 8.3 | 0.029 | 7.8 | 0.061 | 16 | 0.32 | 84 |
| 120 DAT | 0.10 | 0.008 | 7.4 | 0.008 | 8.2 | 0.016 | 16 | 0.087 | 84 |
| 365 DAT | 0.066 | 0.005 | 7.7 | 0.006 | 8.8 | 0.011 | 17 | 0.055 | 83 |

^a Extractable radioactive residues: sum of methanol and water extracts

The results of the identification and characterization of radioactive residues are presented in Tables 38 to 44. Parent kresoxim-methyl was only found in lettuce (up to 0.003 mg eq/kg, 6.1% TRR at 30d PBI) and in radish (up to 0.040 mg eq/kg, 9.1% TRR, immature radish, 30d PBI). As a major metabolite, 490M78 was found in immature radish (30 days PBI) and mature radish leaves (30 days PBI) at up to 0.19 mg eq/kg (44% TRR). In mature radish leaves from later PBIs, radish roots and wheat forage levels of 490M78 were significantly lower

Other identified metabolites comprised of 490M06, 490M68/490M69 and 490M76/490M77 at levels ranging between 0.14–0.26 mg eq/kg (8.7–16% TRR). However, these metabolites were identified only in wheat straw at 30 days PBI, but not at later PBIs.

Multiple other unknown metabolites were characterized by HPLC. In radish tops at 30 d PBI, one unknown component was found at 0.062 mg eq/kg (12% TRR). However, the peak was poorly resolved, overlapping with 490M78 and therefore not further identified. In all matrices unknown metabolites were found at up to 0.013 mg eq/kg (15% TRR) in radish top (120 d PBI) and 0.038 mg eq/kg (13% TRR) in wheat forage (30 d PBI).

748

| radiolabelled kresoxim-methyl | | | | | |
|-----------------------------------|--|----------------|--------------|--------------|--|
| | Radioactive residues in mg eq/kg (% TRR) | | | | |
| Fraction / | Immature lettuce | Mature lettuce | | | |
| Solubilisate | 30 d PBI | 30 d PBI | 120 d PBI | 365 d PBI | |
| TRR | 0.089 (100%) | 0.049 (100%) | 0.094 (100%) | 0.012 (100%) | |
| Kresoxim-methyl | 0.001 (1.3%) | 0.003 (6.1%) | 0.001 (1.1%) | n/d | |
| 490M78 | 0.013 (15%) | 0.006 (12%) | 0.003 (3.0%) | 0.001 (4.5%) | |
| Total identified | 0.014 (16%) | 0.009 (18%) | 0.004 (4.1%) | 0.001 (4.5%) | |
| Characterized by HPLC | 0.037 (41%) | 0.018 (37%) | 0.029 (31%) | 0.005 (39%) | |
| Water extracts | 0.003 (2.9%) | - | 0.005 (4.8%) | 0.001 (5.3%) | |
| Post extraction solids | 0.029 (33%) | 0.013 (27%) | 0.049 (51%) | 0.006 (51) | |
| Ammonia solubilizate | - | 0.001 (1.4%) | 0.003 (2.8%) | - | |
| Macerozyme/Cellulase solubilizate | - | 0.005 (9.2%) | 0.012 (13%) | - | |
| Total characterized | 0.039 (44%) | 0.024 (48%) | 0.049 (52%) | 0.005 (45%) | |
| Unextracted | 0.029 (33%) | 0.009 (17%) | 0.031 (33%) | 0.006 (51%) | |
| Total | 0.082 (93%) | 0.042 (83%) | 0.084 (88%) | 0.012 (100%) | |

Table 38 Summary of identified/characterized residues in lettuce planted in rotation following application of [phenyl-¹⁴C]radiolabelled kresoxim-methyl

Table 39 Summary of identified/characterized residues in immature radish and radish tops planted in rotation following application of [phenyl-14C]-radiolabelled kresoxim-methyl

| | Radioactive residues in mg eq/kg (% TRR) | | | |
|-----------------------------------|--|--------------------|--------------|--------------|
| Fraction / | Immature radish | Mature radish tops | | |
| Solubilisate | 30 d PBI | 30 d PBI | 120 d PBI | 365 d PBI |
| TRR | 0.44 (100%) | 0.51 (100%) | 0.092 (100%) | 0.042 (100%) |
| Kresoxim-methyl | 0.040 (9.1%) | 0.018 (3.5%) | n/d | n/d |
| 490M78 | 0.19 (44%) | 0.14 (28%) | 0.012 (13%) | 0.009 (22%) |
| Total identified | 0.23 (53%) | 0.16 (31%) | 0.012 (13%) | 0.009 (22%) |
| Characterized by HPLC | 0.066 (15%) | 0.29 (56%) | 0.041 (44%) | 0.016 (39%) |
| Water extracts | 0.014 (3.2%) | - | - | 0.003 (8.2%) |
| Post extraction solids | 0.097 (22%) | 0.065 (13%) | 0.030 (32%) | 0.012 (28) |
| Ammonia solubilizate | - | 0.017 (3.3%) | 0.002 (2.4%) | - |
| Macerozyme/Cellulase solubilizate | - | 0.004 (0.8%) | 0.011 (12%) | - |
| Total characterized | 0.080 (18%) | 0.31 (60%) | 0.054 (58%) | 0.019 (47%) |
| Unextracted | 0.097 (22%) | 0.039 (7.7%) | 0.017 (19%) | 0.012 (28%) |
| Total | 0.41 (93%) | 0.51 (99%) | 0.083 (90%) | 0.040 (97%) |

Table 40 Summary of identified/characterized residues in radish roots planted in rotation following application of [phenyl-¹⁴C]-radiolabelled kresoxim-methyl

| Fraction / | Radio | Radioactive residues in mg eq/kg (% TRR) | | | |
|------------------------|--------------------|--|---------------|--|--|
| | Mature radish root | | | | |
| Solubilisate | 30 d PBI | 120 d PBI | 365 d PBI | | |
| TRR | 0.21 (100%) | 0.038 (100%) | 0.012 (100%) | | |
| Kresoxim-methyl | n/d | n/d | n/d | | |
| 490M78 | 0.073 (35%) | 0.007 (17%) | 0.001 (11%) | | |
| Total identified | 0.073 (35%) | 0.007 (17%) | 0.001 (11%) | | |
| Characterized by HPLC | 0.068 (32%) | 0.013 (35%) | 0.006 (48%) | | |
| Water extracts | - | 0.002 (4.2%) | <0.001 (2.7%) | | |
| Post extraction solids | 0.056 (27%) | 0.013 (34%) | 0.004 (38%) | | |
| Ammonia solubilizate | 0.002 (1.0%) | 0.002 (4.0%) | - | | |
| Total characterized | 0.073 (34%) | 0.020 (52%) | 0.007 (51%) | | |
| Unextracted | 0.013 (6.3%) | 0.006 (17%) | 0.004 (38%) | | |
| Total | 0.16 (75%) | 0.033 (86%) | 0.012 (100%) | | |

| Fraction / | Radioactive residues in mg eq/kg (% TRR) | | | | |
|-----------------------------------|--|--------------|--------------|--|--|
| | | Wheat forage | | | |
| Solubilisate | 30 d PBI | 120 d PBI | 365 d PBI | | |
| TRR | 0.30 (100%) | 0.056 (100%) | 0.021 (100%) | | |
| Kresoxim-methyl | n/d | n/d | n/d | | |
| 490M78 | 0.033 (11%) | 0.002 (3.0%) | 0.001 (6.2%) | | |
| Total identified | 0.033 (11%) | 0.002 (3.0%) | 0.001 (6.2%) | | |
| Characterized by HPLC | 0.19 (64%) | 0.014 (26%) | 0.006 (27%) | | |
| Water extracts | | 0.002 (3.4%) | 0.001 (4.6%) | | |
| Post extraction solids | 0.064 (21%) | 0.030 (53%) | 0.013 (63%) | | |
| Ammonia solubilizate | 0.005 (1.8%) | 0.001 (1.3%) | - | | |
| Macerozyme/Cellulase solubilizate | 0.021 (7.0%) | 0.009 (17%) | - | | |
| Amylase/amyloglucosidase | 0.002 (0.6%) | - | - | | |
| Tyrosinase/laccase | 0.001 (0.3%) | - | - | | |
| Total characterized | 0.22 (74%) | 0.026 (48%) | 0.007 (32%) | | |
| Unextracted | 0.022 (7.3%) | 0.015 (27%) | 0.013 (63%) | | |
| Total | 0.28 (92%) | 0.043 (78%) | 0.021 (101%) | | |

Table 41 Summary of identified/characterized residues in wheat (forage) planted in rotation following application of [phenyl-¹⁴C]radiolabelled kresoxim-methyl

Table 42 Summary of identified/characterized residues in wheat (chaff) planted in rotation following application of [phenyl-¹⁴C]radiolabelled kresoxim-methyl

| Fraction / | Radioactive residues in mg eq/kg (% TRR) | | | |
|-----------------------------------|--|--------------|--------------|--|
| | | Wheat chaff | | |
| Solubilisate | 30 d PBI | 120 d PBI | 365 d PBI | |
| TRR | 0.64 (100%) | 0.17 (100%) | 0.099 (100%) | |
| Kresoxim-methyl | n/d | n/d | n/d | |
| 490M78 | n/d | n/d | n/d | |
| Total identified | n/a | n/a | n/a | |
| Characterized by HPLC | 0.33 (51%) | 0.058 (34%) | 0.025 (25%) | |
| Post extraction solids | 0.28 (44%) | 0.11 (62%) | 0.074 (75%) | |
| Ammonia solubilizate | 0.023 (3.6%) | 0.008 (4.5%) | - | |
| Macerozyme/Cellulase solubilizate | 0.035 (5.6%) | 0.012 (7.1%) | - | |
| Amylase/amyloglucosidase | 0.016 (2.5%) | 0.006 (3.8%) | - | |
| Tyrosinase/laccase | 0.004 (0.6%) | - | - | |
| Microwave solubilizate 1&2 | 0.009 (1.5%) | - | - | |
| 1 M HCl solubilizate | 0.01 (0.2%) | - | - | |
| 6 M HCl solubilizate | 0.040 (6.3%) | - | - | |
| 1 M NaOH solubilizate | 0.076 (12%) | 0.024 (14%) | - | |
| Total characterized | 0.53 (83%) | 0.11 (64%) | 0.025 (25%) | |
| Unextracted | 0.017 (2.7%) | 0.041 (25%) | 0.074 (75%) | |
| Total | 0.55 (86%) | 0.15 (89%) | 0.099 (100%) | |

Table 43 Summary of identified/characterized residues in wheat (straw) planted in rotation following application of [phenyl-¹⁴C]radiolabelled kresoxim-methyl

| Fraction / | Radio | Radioactive residues in mg eq/kg (% TRR) Wheat straw | | | | |
|------------------------|--------------|---|-------------|--|--|--|
| Solubilisate | 30 d PBI | 120 d PBI | 365 d PBI | | | |
| TRR | 1.6 (100%) | 0.33 (100%) | 0.16 (100%) | | | |
| Kresoxim-methyl | 0.035 (2.2%) | n/d | n/d | | | |
| 490M06 | 0.14 (8.7) | n/d | n/d | | | |
| 490M68/490M69 | 0.26 (16) | n/d | n/d | | | |
| 490M76/490M77 | 0.15 (9.4) | n/d | n/d | | | |
| 490M78 | n/d | n/d | n/d | | | |
| Total identified | 0.59 (36%) | n/a | n/a | | | |
| Characterized by HPLC | 0.63 (39%) | 0.13 (38%) | 0.064 (39%) | | | |
| Post extraction solids | 0.41 (25%) | 0.16 (49%) | 0.10 (61%) | | | |
| Ammonia solubilizate | 0.072 (4.4%) | 0.014 (4.3%) | - | | | |

| Fraction / | Radioactive residues in mg eq/kg (% TRR) Wheat straw | | | |
|-----------------------------------|---|--------------|-------------|--|
| Solubilisate | 30 d PBI | 120 d PBI | 365 d PBI | |
| Macerozyme/Cellulase solubilizate | 0.041 (2.5%) | 0.020 (6.1%) | - | |
| Amylase/amyloglucosidase | 0.012 (0.7%) | 0.005 (1.7%) | - | |
| Tyrosinase/laccase | 0.014 (0.8%) | - | - | |
| 6 M HCI solubilizate | 0.063 (3.8%) | - | - | |
| 1 NaOH solubilizate | 0.044 (2.7%) | 0.043 (13%) | - | |
| Total characterized | 0.88 (54%) | 0.21 (63%) | 0.064 (39%) | |
| Unextracted | 0.017 (1.1%) | 0.066 (20%) | 0.10 (61%) | |
| Total | 1.5 (91%) | 0.28 (83%) | 0.16 (100%) | |

Table 44 Summary of identified/characterized residues in wheat (grain) planted in rotation following application of [phenyl-¹⁴C]radiolabelled kresoxim-methyl

| Fraction / | Radioactive residues in mg eq/kg (% TRR) Wheat grain | | |
|-----------------------------------|---|--------------|--|
| Solubilisate | 30 d PBI | 120 d PBI | |
| TRR | 0.38 (100%) | 0.10 (100%) | |
| Kresoxim-methyl | n/d | n/d | |
| 490M78 | n/d | n/d | |
| Total identified | n/a | n/a | |
| Characterized by HPLC | 0.051 (14%) | 0.016 (17%) | |
| Post extraction solids | 0.32 (84%) | 0.087 (84%) | |
| Ammonia solubilizate | 0.026 (6.9%) | 0.004 (3.5%) | |
| Macerozyme/Cellulase solubilizate | 0.13 (34%)ª | 0.037 (36%) | |
| Amylase/amyloglucosidase | 0.070 (19%) | - | |
| β-glucosidase/hesperidinase | 0.010 (2.6%) | 0.007 (6.4) | |
| Tyrosinase/laccase | 0.011 (2.8%) | - | |
| Total characterized | 0.30 (79%) | 0.063 (62%) | |
| Unextracted | 0.026 (6.9%) | 0.030 (29%) | |
| Total | 0.28 (86%) | 0.093 (91%) | |

^a Identified as carbohydrates

Figure 6 Proposed metabolic pathway of [phenyL14C]-radiolabelled kresoxim-methyl in rotational crops

RESIDUE ANALYSIS

Analytical methods

For the analysis of kresoxim-methyl and metabolites in various plant and animal matrices, analytical methods suitable for enforcement and data generation purposes were submitted. In the following table an overview of these methods is presented.

| Method | Matrix | Extraction | Clean-Up | Analyte, Detection, LOQ |
|-------------------|-------------------|-----------------------------|-------------------|----------------------------------|
| BASF method 445/0 | Apple, leek, sour | Methanol/water/hydrochloric | Partitioning with | HPLC-MS/MS |
| | cherry, grapes, | acid (70/25/5, v/v/v) | dichloromethane | Kresoxim-methyl: m/z |
| (BASF method | strawberry, | | | $314 \rightarrow 116$ and/or m/z |
| L0010/01) | carrot, onion, | | | 314→131 |
| | tomato, broccoli, | | | |
| | white cabbage, | | | LOQ: 0.01 mg/kg or 0.05 |
| | beans, pods with | | | mg/kg |
| | bean, lettuce, | | | |
| | oilseed rape, | | | |

Table 45 Overview of analytical methods for kresoxim-methyl and metabolites

| Method | Matrix | Extraction | Clean-Up | Analyte, Detection, LOQ |
|---|---|----------------------------|---|--|
| | wheat plant, wheat ear, wheat grain, wheat straw, citrus | | | |
| BASF method 351/1 | Apple, wheat straw, wheat grain | Methanol | Partitioning with iso- octane, SPE silica gel, SPE C18 | GC-ECD Kresoxim-methyl: LOQ: 0.05 mg/kg |
| BASF method 351/2 | Apple, wheat straw, wheat grain, wheat forage | Methanol | Partitioning with iso- octane, SPE silica gel, SPE C18 | GC-ECD or GC-MS Kresoxim-methyl: m/z 206 |
| BASF method 350/1 | Wheat straw, wheat grain, wheat forage | Methanol/water (8/2, v/v) | Kresoxim-methyl: partitioning with iso- octane, SPE silica gel, SPE C18 490M2 and 490M9: enzyme hydrolysis, partition with dichloromethane, SPE NH ₂ , HPLC RP-C18 | GC-ECD or GC-MS Kresoxim-methyl: m/z 206 LC-LC-UV 490M2 and 490M9: LOQ: 0.05 mg/kg |
| BASF method 350/3 | Tomato, pepper, melon, cucumber, onion, grape, wine, must, grape pomace, apple, apple juice | Methanol | Kresoxim-methyl: Alkaline hydrolysis to 490M1 490M2 and 490M9: enzyme hydrolysis All analytes: partition with dichloromethane, SPE NH2 | LC-LC-UV Kresoxim-methyl (as 490M1), 490M2 and 490M9 LOQ: 0.05 mg/kg |
| BASF method 350/4 (identical to SOP- PA.0295) | Apple, grapes, dried pea, soya bean, wheat grain (wheat & barley), ear (wheat & barley), straw (wheat & barley), plant (wheat & barley), mango | Methanol | Kresoxim-methyl: Alkaline hydrolysis to 490M1 490M2 and 490M9: enzyme hydrolysis All analytes: partition with dichloromethane | HPLC-MS/MS Kresoxim-methyl (as 490M1): m/z 298 \rightarrow 102, 298 \rightarrow 222 490M2: m/z 314 \rightarrow 132, 314 \rightarrow 102 490M9: m/z 314 \rightarrow 122, 314 \rightarrow 216 LOQ: 0.01 mg/kg |
| Method JP 2015C423 | Unshu mandarin | Water/acetone (20/80, v/v) | Diatomaceous earth column, silica gel column | HPLC-MS/MS Kresoxim-methyl: m/z 314→116 LOQ: 0.01 mg/kg |
| Method Saku 6P-7-175 | Unshu mandarin | Acetone | Diatomaceous earth column, silica gel column, florisil column | GC-NPD Kresoxim-methyl LOQ: 0.2 mg/kg (pulp), 0.4 mg/kg (peel) |
| BASF method L0177/01 | Milk, egg, meat, fat, liver, kidney | Methanol | None, except for fat: partitioning with dichloromethane | HPLC-MS/MS 490M1: m/z 298→102, 222→102 LOO: 0.01 mg/kg |
| BASF method 354/1 | Milk | Acetone | Partitioning with iso- octane, partitioning with dichloromethane, SPE NH ₂ , partitioning | LC-LC-UV 490M2 and 490M9 LOQ: 0.001 mg/kg |
| | | | with dichloromethane | |

| Method | Matrix | Extraction | Clean-Up | Analyte, Detection, LOQ |
|--------|------------|------------|--------------------------------|-------------------------|
| | liver, fat | | partitioning, SPE | 490M1, 490M2 and 490M9 |
| | | | NH ₂ , HPLC RP C18. | |
| | | | | LOQ: 0.001 mg/kg |

Plant materials

BASF method 445/0 (BASF method L0010/01) (Benz & Mackenroth, 2001a, Kresoxim_034: Leite & Santiago, 2010a, Kresoxim_036; Schroth & Martin, 2009a, Kresoxim_037; Schroth & Martin, 2009b, Kresoxim_038)

Plant matrices (high water content, acidic, dry and oily) were extracted with methanol/water/hydrochloric acid (70/25/5, v/v/v). After centrifugation, an aliquot of the supernatant was then partitioned against cyclohexane. An aliquot of cyclohexane phase was taken and evaporated to dryness, followed by reconstitution of the remainder in methanol/water 80/20, v/v).

Final determination of kresoxim-methyl was done by LC-MS/MS in positive ionization mode using a C18 column and monitoring the transition m/z 314+16 and/or m/z 314+31. Quantitation was done with external standards in methanol.

Table 46 Recovery data for BASF method 445/0 measuring kresoxim-methyl in various plant matrices (Benz & Mackenroth, 2001a, Kresoxim 034)

| Matrix | Fortification level (mg/kg) | n | Recovery, mean (%) | RSD (%) | Analyte, MRM transition |
|-------------------|-----------------------------|---|--------------------|---------|---------------------------|
| Apple, fruit | 0.05 | 5 | 81 | 4.9 | |
| | 0.5 | 5 | 86 | 16 | Kresoxim-methyl, |
| Leek | 0.05 | 5 | 86 | 7.1 | m/z: 314 1 31 |
| Leek | 0.5 | 5 | 83 | 15 | |
| Sour cherry | 0.05 | 5 | 88 | 7.2 | |
| Sour cherry | 0.5 | 5 | 83 | 2.6 | |
| Grapes | 0.05 | 5 | 94 | 2.7 | |
| Grapes | 0.5 | 5 | 104 | 17 | |
| Strawberry | 0.05 | 5 | 92 | 4.5 | |
| Strawberry | 0.5 | 5 | 97 | 6.5 | |
| Carrot | 0.05 | 5 | 87 | 3.0 | |
| Carrot | 0.5 | 5 | 83 | 3.4 | |
| Onion | 0.05 | 5 | 96 | 2.0 | |
| UNION | 0.5 | 5 | 95 | 8.9 | |
| Tomato | 0.05 | 5 | 91 | 15 | |
| Tomato | 0.5 | 5 | 81 | 6.3 | Kresoxim-methyl, |
| Broccoli | 0.05 | 5 | 100 | 14 | |
| BIOCCOII | 0.5 | 5 | 88 | 4.1 | m/z: 314 -1 16 |
| White eachbage | 0.05 | 5 | 73 | 18 | |
| White cabbage | 0.5 | 5 | 90 | 4.8 | |
| Dwarf beans, pods | 0.05 | 5 | 88 | 5.2 | |
| with bean | 0.5 | 5 | 90 | 3.1 | |
| Oilseed rape | 0.05 | 5 | 84 | 16 | |
| oliseeu tape | 0.5 | 5 | 90 | 4.3 | |
| Wheat plant | 0.05 | 5 | 87 | 6.0 | |
| | 0.5 | 5 | 83 | 6.3 | |
| Wheat grain | 0.05 | 5 | 86 | 4.8 | |
| | 0.5 | 5 | 90 | 3.0 | |
| Wheat straw | 0.05 | 5 | 93 | 3.6 | |
| WIICAL SUAW | 0.5 | 5 | 85 | 7.8 | |

Table 47 Recovery data for BASF method 445/0 measuring kresoxim-methyl in lettuce (Leite & Santiago, 2010a, Kresoxim_036)

| Matrix | Fortification level (mg/kg) | n | Recovery, mean (%) | RSD (%) | Analyte, MRM transition |
|---------|-----------------------------|---|--------------------|---------|-------------------------------|
| Lettuce | 0.01 | 5 | 92 | 7 | Kresoxim-methyl, m/z: 314 +16 |
| | 1 | 5 | 89 | 8 | Quantitation |
| | 0.01 | 5 | 94 | 11 | Kresoxim-methyl, m/z: 314 +31 |
| | 1 | 5 | 90 | 8 | Confirmation |

Table 48 Recovery data for BASF method No. L0010/01 measuring kresoxim-methyl in wheat matrices (Schroth & Martin, 2009a, Kresoxim_037)

| Matrix | Fortification level (mg/kg) | n | Recovery, mean (%) | RSD (%) | Analyte, MRM transition |
|-----------------------------|-----------------------------|---|--------------------|---------|--------------------------|
| | 0.01 | 1 | 106 | - | |
| Whole plant | 0.1 | 1 | 96 | - | |
| | 10 | 1 | 81 | - | |
| | 0.01 | 1 | 87 | - | |
| Ear | 0.1 | 1 | 94 | - | |
| | 1 | 1 | 79 | - | |
| Dect of plant w/o | 0.01 | 1 | 86 | - | Kresoxim-methyl, |
| Rest of plant, w/o roots | 0.1 | 1 | 71 | - | m/z: 314 1 16 |
| TOOLS | 1 | 1 | 79 | - | Quantitation |
| | 0.01 | 1 | 79 | - | |
| Grain | 0.1 | 1 | 74 | - | |
| | 1 | 1 | 80 | - | |
| | 0.01 | 1 | 71 | - | |
| Straw | 0.1 | 1 | 91 | - | |
| | 1 | 1 | 92 | - | |

Table 49 Recovery data for BASF method No. L0010/01 measuring kresoxim-methyl in barley matrices (Schroth & Martin, 2009b, Kresoxim_038)

| Matrix | Fortification level (mg/kg) | n | Recovery, mean (%) | RSD (%) | Analyte, MRM transition |
|-----------------------------|-----------------------------|---|--------------------|---------|-------------------------|
| Whele plant w/a | 0.01 | 1 | 77 | - | |
| Whole plant, w/o roots | 0.1 | 1 | 107 | - | |
| TOOLS | 1 | 1 | 98 | - | |
| | 0.01 | 1 | 83 | - | |
| Ear | 0.1 | 1 | 87 | - | |
| | 1 | 1 | 72 | - | |
| Deat of alant w/a | 0.01 | 1 | 71 | - | Kresoxim-methyl, |
| Rest of plant, w/o roots | 0.1 | 1 | 81 | - | m/z: 314 1 4 |
| 10015 | 1 | 1 | 80 | - | Quantitation |
| | 0.01 | 1 | 107 | - | |
| Grain | 0.1 | 1 | 120 | - | |
| | 1 | 1 | 104 | - | |
| | 0.01 | 1 | 76 | - | |
| Straw | 0.1 | 1 | 93 | - | |
| | 1 | 1 | 96 | - | |

Independent laboratory validation of BASF method 445/0 (Schwarz & Class, 2007, Kresoxim_035)

The sample preparation was identical to BASF method 445/0. However, two ion transitions (m/z $314\rightarrow116$, $314\rightarrow131$) were monitored for quantitation and confirmation.

Table 50 Recovery data for the ILV of BASF method 445/0 measuring kresoxim-methyl in high water content, acidic, dry and oily plant matrices

| Matrix | Fortification level (mg/kg) | n | Recovery, mean (%) | RSD (%) | Analyte, MRM transition |
|-------------|-----------------------------|---|--------------------|---------|--|
| Tomato | 0.01 | 5 | 83 | 5 | |
| | 0.1 | 5 | 90 | 5 | |
| Citrus | 0.01 | 5 | 83 | 7 | Knoopying mothul |
| | 0.1 | 5 | 100 | 1 | Kresoxim-methyl, m/z: 314 1 16 |
| Wheat grain | 0.01 | 5 | 94 | 5 | Ouantitation |
| | 0.1 | 5 | 99 | 3 | Quantitation |
| Rape seed | 0.05 | 5 | 78 | 2 | |
| | 0.5 | 5 | 74 | 5 | |
| Tomato | 0.01 | 5 | 82 | 8 | |
| | 0.1 | 5 | 89 | 3 | Kresoxim-methyl, m/z: 314 1 31 |
| Citrus | 0.01 | 5 | 81 | 4 | |
| | 0.1 | 5 | 99 | 2 | Confirmation |
| Wheat grain | 0.01 | 5 | 84 | 5 | |

| Matrix | Fortification level (mg/kg) | n | Recovery, mean (%) | RSD (%) | Analyte, MRM transition |
|-----------|-----------------------------|---|--------------------|---------|-------------------------|
| | 0.1 | 5 | 102 | 2 | |
| Rape seed | 0.05 | 5 | 78 | 5 | |
| | 0.5 | 5 | 75 | 7 | |

BASF method 351/1 (Krotzky & Dams, 1993, Kresoxim_040; Schulz, 1994, Kresoxim_041)

Plant matrices were homogenised with methanol. After filtration, aqueous KH₂PO₄ solution was added to the extract, the mixture evaporated to the aqueous remainder and partitioned against iso-octane. The iso-octane phase was dried over sodium sulphate and further cleaned-up on i) a silica gel SPE column and ii) a C18 SPE column. The eluate was evaporated to dryness, followed by reconstitution in iso-octane/ethylacetate (75/25, v/v). Final determination of kresoxim-methyl was done by GC-ECD on a DB 1701 column. Quantitation was done with external standards in iso-octane/ethylacetate (75/25, v/v).

Table 51 Recovery data for BASF method 351/1 measuring kresoxim-methyl in apple and wheat matrices (Krotzky & Dams, 1993, Kresoxim_040)

| Matrix | Fortification level (mg/kg) | n | Recovery, mean (%) | RSD (%) | Analyte |
|-------------|-----------------------------|-----|--------------------|---------|-----------------|
| Apple | 0.05–0.5 | n/a | 91 | 4.8 | |
| Wheat straw | 0.05–0.5 | n/a | 92 | 17 | Kresoxim-methyl |
| Wheat grain | 0.05–0.5 | n/a | 84 | 16 | |

Table 52 Recovery data for BASF method 351/1 measuring kresoxim-methyl in apple and wheat matrices (Schulz, 1994, Kresoxim_041)

| Matrix | Fortification level (mg/kg) | n | Recovery, mean (%) | RSD (%) | Analyte |
|-------------|-----------------------------|----|--------------------|---------|-----------------|
| Apple | 0.05–0.5 | 10 | 98 | 6.4 | Kresoxim-methyl |
| Wheat grain | 0.05-0.5 | 8 | 92 | 7.8 | Kresoxim-metnyi |

BASF method 351/2 (Mackenroth & Krotzky, 1994a, Kresoxim_042; Mackenroth & Krotzky, 1996, Kresoxim_043; Wayman, 1994, Kresoxim_044)

Sample preparation and method of analysis were identical to BASF method 351/1. However, in method 351/2 alternative detectors such as GC-MS (m/z 206) and GC-NPD were used.

Table 53 Recovery data for BASF method 351/2 measuring kresoxim-methyl in apple and wheat matrices (Mackenroth & Krotzky, 1994a, Kresoxim_042)

| Matrix | Fortification level (mg/kg) | n | Recovery, mean (%) | RSD (%) | Analyte |
|--------------|-----------------------------|---|--------------------|---------|-----------------------------|
| | 0.05 | 4 | 88 | 8.0 | Krocovim methyl CC FCD |
| Apple | 5 | 4 | 94 | 5.3 | Kresoxim-methyl, GC-ECD |
| Apple | 0.05 | 4 | 79 | 7.2 | Kresoxim-methyl, GC-MS |
| | 5 | 4 | 88 | 6.0 | Kresoxim-metriyi, GC-WS |
| Wheat straw | 0.05 | 4 | 92 | 7.6 | Krasovim methyl CC MS |
| wheat sti aw | 5 | 4 | 111 | 11 | Kresoxim-methyl, GC-MS |
| Wheat grain | 0.05 | 4 | 98 | 16 | Kresoxim-methyl, GC-NPD |
| wheat grain | 5 | 4 | 99 | 12 | Kiesoxim-metnyi, GC-NPD |
| Wheat forage | 0.05 | 4 | 104 | 9.2 | Kresoxim-methyl, GC-MS |
| Wheat forage | 5 | 4 | 106 | 5.6 | Kiesoxiiii-iiietiiyi, GC-MS |

Table 54 Recovery data for BASF method 351/2 measuring kresoxim-methyl in apples (Wayman, 1994, Kresoxim_044)

| Matrix | Fortification level (mg/kg) | n | Recovery, mean (%) | RSD (%) | Analyte |
|--------|-----------------------------|---|--------------------|---------|-------------------------|
| Annia | 0.05 | 4 | 103 | 9.6 | Kresoxim-methyl, GC-ECD |
| Apple | 5 | 4 | 109 | 7.1 | Klesoxim-methyl, GC-ECD |

BASF method 350/1 (Mackenroth & Krotzky, 1994b, Kresoxim_045; Gillis, 1994, Kresoxim_046)

Parent kresoxim-methyl was extracted from plant matrices by homogenization with methanol/water (8/2, v/v). After filtration, aqueous KH₂PO₄ solution was added to the extract and partitioned against iso-octane. The iso-octane phase was dried over sodium

sulphate and further cleaned-up on i) a silica gel SPE column and ii) a C18 SPE column. The eluate was evaporated to dryness, followed by reconstitution in iso-octane/ethylacetate (75/25, v/v).

Metabolites 490M2 and 490M9 were extracted as their glycosides from the filtration residue of the initial methanol/water filtration using 25% ammonia. After filtration the extract was combined with a fraction of the initial methanol/water extract and evaporated to the aqueous remainder. After washing of the aqueous phase with ethylacetat/MTBE (1/1, v/v), ascorbic acid was added and glycosides cleaved by means of enzymatic hydrolysis using hesperidinase and β -glucosidase. Clean-up was performed by partitioning against dichloromethane, followed by SPE on a NH₂-column. Analytes were eluted with a water/NaCl/formic acid/ascorbic acid/methanol. The eluate was once more partitioned against dichloromethane. After evaporation of the organic phase, the remainder was reconstituted in water/acetonitrile (9/1, v/v) and further cleaned-up on a preparative C18 HPLC column.

Final determination of kresoxim-methyl was done by GC-EI-MS or GC-ECD on a DB 1701 column, while metabolites 490M2 and 490M9 were quantified by LC-LC-UV at 270 nm using column switching with a NH₂-column and a CN-column.

Table 55 Recovery data for BASF method 350/1 measuring kresoxim-methyl and metabolites 490M2 and 490M9 in wheat matrices (Mackenroth & Krotzky, 1994b, Kresoxim_045)

| Matrix | Fortification level (mg/kg) | n | Recovery, mean (%) | RSD (%) | Analyte |
|--------------|-----------------------------|----|--------------------|-----------------|-------------------------|
| | 0.05 | 4 | 92 | 7.6 | Krocovin methyl CC MS |
| | 5 | 4 | 111 | 11 | Kresoxim-methyl, GC-MS |
| Wheat straw | 0.05 | 4 | 62 | 13 | 490M2, LC-LC-UV |
| wheat straw | 5 | 4 | 74 | 7.6 | 490IVIZ, LC-LC-UV |
| | 0.05 4 61 | 61 | 13 | 490M9, LC-LC-UV | |
| | 5 | 4 | 73 | 8.6 | 4901019, LC-LC-UV |
| | 0.05 | 4 | 104 | 9.2 | Krasswim methyl CC CCD |
| | 5 | 4 | 89 | 1.8 | Kresoxim-methyl ,GC-ECD |
| Wheat forage | 0.05 | 4 | 89 | 8.7 | 490M2, LC-LC-UV |
| Wheat forage | 5 | 4 | 75 | 2.8 | 490IVIZ, LC-LC-UV |
| | 0.05 | 4 | 85 | 6.6 | 490M9, LC-LC-UV |
| | 5 | 4 | 79 | 3.2 | 4701V17, LC-LC-UV |

Table 56 Recovery data for BASF method 350/1 measuring kresoxim-methyl and metabolites 490M2 and 490M9 in wheat matrices (Gillis, 1994, Kresoxim_046)

| Matrix | Fortification level (mg/kg) | n | Recovery, mean (%) | RSD (%) | Analyte |
|--|-----------------------------|---|--------------------|---------|-------------------|
| When the set on the set of the se | 0.05 | 4 | 82 | 12.6 | Knoopuing meethod |
| Wheat grain | 5 | 4 | 98 | 4.4 | Kresoxim-methyl |
| | 0.05 | 4 | 97 | 2.3 | Knonovine mothed |
| | 5 | 4 | 74 | 2.9 | Kresoxim-methyl |
| W/h a at atravu | 0.05 | 4 | 92 | 12.1 | 400142 |
| Wheat straw | 5 | 4 | 96 | 3.2 | 490M2 |
| | 0.05 | 4 | 96 | 16.3 | 490M9 |
| | 5 | 4 | 98 | 6.5 | |
| | 0.05 | 4 | 77 | 11.4 | Kracovim mathul |
| | 5 | 4 | 81 | 4.2 | Kresoxim-methyl |
| Wheat forage | 0.05 | 4 | 80 | 11.5 | 490M2 |
| wheat totage | 5 | 4 | 89 | 5.6 | 4901012 |
| | 0.05 | 4 | 84 | 11.4 | 490M9 |
| | 5 | 4 | 84 | 10.6 | 4701017 |

BASF method 350/3 (Rabe & Mackenroth, 1996, Kresoxim_047)

Parent kresoxim-methyl and metabolites 490M2 and 490M9 were extracted from plant matrices by homogenization with methanol after addition of ascorbic acid. After filtration, aqueous KH_2PO_4 solution was added to the extract and the glycoside-conjugates cleaved by enzymatic hydrolysis using hesperidinase and β -glucosidase. In order to hydrolyze parent kresoxim-methyl to 490M1, the pH was adjusted to 13 with 10M KOH and incubated at RT for 1h. Upon completion of the hydrolysis, the pH was set to 2 with formic acid and the extract was partitioned against dichloromethane. Additional clean-up was performed by SPE on a NH2-column. Analytes were eluted with a water/NaCl/formic acid/ascorbic acid/methanol. The eluate was once more partitioned against dichloromethane. After evaporation of the organic phase, the remainder was reconstituted in acetonitrile. Prior to analysis an aliquot was evaporated to dryness and reconstituted in acetonitrile/water +ascorbic acid.

Final determination of kresoxim-methyl (as 490M1) and metabolites 490M2 and 490M9 was done by LC-LC-UV at 270 nm using column switching with a NH2-column and a C18-column.

Table 57 Recovery data for BASF method 350/3 measuring kresoxim-methyl and metabolites 490M2 and 490M9 in plant matrices (Rabe & Mackenroth, 1996, Kresoxim_047)

| Matrix | Fortification level (mg/kg) | n | Recovery, mean (%) | RSD (%) | Analyte |
|--------------|-----------------------------|---|--------------------|---------|-----------------|
| | 0.05 | 5 | 93 | 2.5 | Kresoxim-methyl |
| | 5 | 5 | 88 | 3.0 | Kresoxim-methy |
| Tomata | 0.05 | 5 | 82 | 5.7 | 100M2 |
| Tomato | 5 | 5 | 84 | 1.7 | 490M2 |
| | 0.05 | 5 | 89 | 2.9 | 400140 |
| | 5 | 5 | 84 | 2.6 | 490M9 |
| | 0.05 | 5 | 100 | 3.4 | |
| | 5 | 5 | 100 | 1.4 | Kresoxim-methyl |
| - | 0.05 | 5 | 87 | 2.4 | |
| Pepper | 5 | 5 | 94 | 1.0 | 490M2 |
| | 0.05 | 5 | 91 | 3.4 | |
| | 5 | 5 | 93 | 1.5 | 490M9 |
| | 0.05 | 5 | 101 | 3.7 | |
| | 5 | 5 | 91 | 2.9 | Kresoxim-methyl |
| | 0.05 | 5 | 89 | 3.1 | |
| Melon | 5 | 5 | 92 | 4.1 | 490M2 |
| | 0.05 | 5 | 98 | 3.1 | |
| | 5 | 5 | 92 | 3.5 | 490M9 |
| | 0.05 | 5 | 101 | 4.3 | |
| | 5 | 5 | 109 | 4.6 | Kresoxim-methyl |
| | 0.05 | 5 | 93 | 4.0 | |
| Cucumber | 5 | 5 | 103 | 3.2 | 490M2 |
| | 0.05 | 5 | 79 | 5.9 | |
| | 5 | 5 | 91 | 4.5 | 490M9 |
| | 0.05 | 5 | 105 | 6.8 | |
| | 5 | 5 | 93 | 1.3 | Kresoxim-methyl |
| | 0.05 | 5 | 96 | 3.7 | - 490M2 |
| Onion | 5 | 5 | 95 | 2.3 | |
| | 0.05 | 5 | 94 | 4.4 | 490M9 |
| | 5 | 5 | 95 | 2.0 | |
| | 0.05 | 5 | 97 | 7.7 | |
| | 5 | 5 | 105 | 2.5 | Kresoxim-methyl |
| | 0.05 | 5 | 88 | 7.3 | - |
| Grape | 5 | 5 | 101 | 1.1 | 490M2 |
| | | | | | |
| | 0.05 | 5 | 86 | 8.0 | 490M9 |
| | 5 | 5 | 97 | 2.0 | |
| | 0.05 | 5 | 91 | 1.7 | Kresoxim-methyl |
| | 5 | 5 | 80 | 3.1 | - |
| Wine | 0.05 | 5 | 87 | 4.7 | 490M2 |
| | 5 | 5 | 82 | 2.8 | |
| | 0.05 | 5 | 83 | 4.5 | 490M9 |
| | 5 | 5 | 83 | 2.6 | |
| | 0.05 | 5 | 100 | 2.3 | Kresoxim-methyl |
| | 5 | 5 | 97 | 2.5 | , |
| Must | 0.05 | 5 | 84 | 4.0 | 490M2 |
| | 5 | 5 | 89 | 3.5 | - |
| | 0.05 | 5 | 86 | 4.8 | 490M9 |
| | 5 | 5 | 89 | 3.8 | |
| | 0.05 | 5 | 83 | 6.4 | Kresoxim-methyl |
| | 5 | 5 | 82 | 4.8 | |
| Grape pomace | 0.05 | 5 | 89 | 2.9 | 490M2 |
| orabe homace | 5 | 5 | 94 | 2.0 | 770112 |
| | 0.05 | 5 | 76 | 2.3 | 490M9 |
| | 5 | 5 | 82 | 2.1 | 4701017 |
| Applo | 0.05 | 5 | 85 | 25.7 | Krosovim mothyl |
| Apple | 5 | 5 | 96 | 10.9 | Kresoxim-methyl |

758

| Matrix | Fortification level (mg/kg) | n | Recovery, mean (%) | RSD (%) | Analyte |
|-------------|-----------------------------|---|--------------------|---------|-----------------|
| | 0.05 | 5 | 89 | 1.7 | 490M2 |
| | 5 | 5 | 95 | 9.4 | 4901012 |
| | 0.05 | 5 | 82 | 10.5 | 490M9 |
| | 5 | 5 | 91 | 8.0 | 490019 |
| | 0.05 | 5 | 92 | 11.0 | Krocovin methyl |
| | 5 | 5 | 98 | 1.8 | Kresoxim-methyl |
| Apple iules | 0.05 | 5 | 74 | 5.6 | 490M2 |
| Apple juice | 5 | 5 | 96 | 0.3 | 4901012 |
| | 0.05 | 5 | 80 | 16.6 | 400140 |
| | 5 | 5 | 95 | 1.2 | 490M9 |

BASF method 350/4 (identical to SOP-PA.0295) (Class, 2007, Kresoxim_048; Schroth & Martin, 2009a, Kresoxim_037; Schroth & Martin, 2009b, Kresoxim_038; Silva & Alves, 2011, Kresoxim_049; Silva, 2011 Kresoxim_050)

Method 350/4 is an adaption of method 350/3 to LC-MS/MS detection. In brief, parent kresoxim-methyl and metabolites 490M2 and 490M9 were extracted from plant matrices by homogenization with methanol after addition of ascorbic acid. After filtration, aqueous KH_2PO_4 solution was added to the extract and the glycoside-conjugates cleaved by enzymatic hydrolysis using hesperidinase and β -glucosidase. In order to hydrolyze parent kresoxim-methyl to 490M1, the pH was adjusted to 13 with 10M KOH and incubated at RT for 1h. Upon completion of the hydrolysis, the pH was set to 2 with formic acid and the extract was partitioned against dichloromethane. The organic phase was dried over Na_2SO_4 , evaporation to dryness and the remainder reconstituted in acetonitrile/water (1+1, v/v).

Final determination of kresoxim-methyl (as 490M1) and metabolites 490M2 and 490M9 was done by LC-MS/MS in negative ionization mode using a C18 column and monitoring the transition m/z 298-102, 298-222 (490M1), m/z 314-132, 314-102 (490M2) and m/z 314-22, 314-216 (490M9).

Table 58 Recovery data for BASF method 350/4 measuring kresoxim-methyl (as 490M1) and metabolites 490M2 and 490M9 in plant matrices (Class, 2007, Kresoxim_048)

| Matrix | Fortification level (mg/kg) | n | Recovery, mean (%) | RSD (%) | Analyte |
|-----------|-----------------------------|---|--------------------|---------|---|
| | 0.01 | 5 | 77 | 7 | Kresoxim-methyl (as 490M1), m/z |
| | 0.1 | 5 | 80 | 8 | 298→102, Quantitation |
| | 0.01 | 5 | 76 | 5 | 400M2 = /= 214 122 Overtitation |
| | 0.1 | 5 | 84 | 7 | − 490M2, m/z 314 \rightarrow 132, Quantitation |
| | 0.01 | 5 | 77 | 9 | 490M9, m/z 314 \rightarrow 122, Quantitation |
| Annia | 0.1 | 5 | 80 | 6 | 4901019, 11/2 3 14 \rightarrow 122, Qualititation |
| Apple | 0.01 | 5 | 72 | 10 | Kresoxim-methyl (as 490M1), m/z |
| | 0.1 | 5 | 78 | 4 | 298 \rightarrow 222, Confirmation |
| | 0.01 | 5 | 78 | 16 | 490M2, m/z 314→102, |
| | 0.1 | 5 | 79 | 5 | Confirmation |
| | 0.01 | 5 | 78 | 7 | 490M9, m/z 314→116, |
| | 0.1 | 5 | 78 | 8 | Confirmation |
| | 0.01 | 5 | 83 | 16 | Kresoxim-methyl (as 490M1), m/z |
| | 0.1 | 5 | 79 | 16 | 298 \rightarrow 102, Quantitation |
| | 0.01 | 5 | 70 | 12 | 400M2 m/z 214 |
| | 0.1 | 5 | 77 | 9 | 490M2, m/z 314→132, Quantitation |
| | 0.01 | 5 | 89 | 12 | 400M0 m/z 214 . 122 Ouentitation |
| Cronoc | 0.1 | 5 | 80 | 10 | 490M9, m/z 314 \rightarrow 122, Quantitation |
| Grapes | 0.01 | 5 | 83 | 15 | Kresoxim-methyl (as 490M1), m/z |
| | 0.1 | 5 | 87 | 19 | 298 \rightarrow 222, Confirmation |
| | 0.01 | 5 | 78 | 19 | 490M2, m/z 314→102, |
| | 0.1 | 5 | 85 | 9 | Confirmation |
| | 0.01 | 5 | 78 | 10 | 490M9, m/z 314→116, |
| | 0.1 | 5 | 73 | 9 | Confirmation |
| | 0.01 | 5 | 97 | 14 | Kresoxim-methyl (as 490M1), m/z |
| | 0.1 | 5 | 88 | 19 | 298→102, Quantitation |
| | 0.01 | 5 | 75 | 12 | 400M2 m/z 214 122 Quantitation |
| Dried pea | 0.1 | 5 | 79 | 7 | − 490M2, m/z 314 \rightarrow 132, Quantitation |
| | 0.01 | 5 | 83 | 15 | $-$ 490M9, m/z 314 \rightarrow 122, Quantitation |
| | 0.1 | 5 | 81 | 5 | $490009, 11/2314 \rightarrow 122, Quantitation$ |
| | 0.01 | 5 | 98 | 13 | Kresoxim-methyl (as 490M1), m/z |

| Matrix | Fortification level (mg/kg) | n | Recovery, mean (%) | RSD (%) | Analyte |
|--------------|-----------------------------|---|--------------------|---------|--|
| | 0.1 | 5 | 90 | 16 | 298 \rightarrow 222, Confirmation |
| | 0.01 | 5 | 84 | 10 | 490M2, m/z 314→102, |
| | 0.1 | 5 | 86 | 10 | Confirmation |
| | 0.01 | 5 | 83 | 15 | 490M9, m/z 314→116, |
| | 0.1 | 5 | 80 | 8 | Confirmation |
| | 0.01 | 5 | 95 | 11 | Kresoxim-methyl (as 490M1), m/z |
| | 0.1 | 5 | 96 | 12 | 298 \rightarrow 102, Quantitation |
| | 0.01 | 5 | 82 | 8 | $-$ 490M2, m/z 314 \rightarrow 132, Quantitation |
| | 0.1 | 5 | 95 | 5 | 4901012, 111/2 3 14→132, Qualititation |
| | 0.01 | 5 | 94 | 7 | 400M0 m/z 214 . 122 Quantitation |
| Soubson | 0.1 | 5 | 103 | 7 | − 490M9, m/z 314 \rightarrow 122, Quantitation |
| Soy bean | 0.01 | 5 | 95 | 17 | Kresoxim-methyl (as 490M1), m/z |
| | 0.1 | 5 | 97 | 12 | 298 \rightarrow 222, Confirmation |
| | 0.01 | 5 | 90 | 9 | 490M2, m/z 314→102, |
| | 0.1 | 5 | 107 | 4 | Confirmation |
| | 0.01 | 5 | 93 | 4 | 490M9, m/z 314→116, |
| | 0.1 | 5 | 95 | 7 | Confirmation |
| | 0.01 | 5 | 86 | 5 | Kresoxim-methyl (as 490M1), m/z |
| | 0.1 | 5 | 82 | 5 | 298→102, Quantitation |
| | 0.01 | 5 | 81 | 5 | 400M2 m/= 214 122 Quantitation |
| | 0.1 | 5 | 81 | 5 | 490M2, m/z 314 \rightarrow 132, Quantitation |
| | 0.01 | 5 | 97 | 5 | 400M0 m/= 214 122 Quantitation |
| Wheet are in | 0.1 | 5 | 78 | 8 | − 490M9, m/z 314 \rightarrow 122, Quantitation |
| Wheat grain | 0.01 | 5 | 83 | 13 | Kresoxim-methyl (as 490M1), m/z |
| | 0.1 | 5 | 75 | 13 | 298→222, Confirmation |
| | 0.01 | 5 | 88 | 7 | 490M2, m/z 314→102, |
| | 0.1 | 5 | 74 | 6 | Confirmation |
| | 0.01 | 5 | 90 | 8 | 490M9, m/z 314→116, |
| | 0.1 | 5 | 79 | 9 | Confirmation |

Table 59 Recovery data for BASF method 350/4 measuring kresoxim-methyl (as 490M1) and metabolites 490M2 and 490M9 in wheat matrices (Schroth & Martin, 2009a, Kresoxim_037)

| Matrix | Fortification level (mg/kg) | n | Recovery, mean (%) | RSD (%) | Analyte, MRM transition |
|-------------------|-----------------------------|---|--------------------|---------|--|
| M/h a la vala vat | 0.01 | 3 | 87 | 3.5 | |
| Whole plant | 0.1 | 5 | 80 | 16 | |
| For | 0.01 | 1 | 71 | - | |
| Ear | 0.1 | 1 | 74 | - | Kresoxim-methyl (as 490M1), m/z: 298→102 |
| Grain | 0.01 | 3 | 86 | 5.1 | $- \qquad \qquad$ |
| Grain | 0.1 | 2 | 76 | - | Quantitation |
| Straw | 0.01 | 3 | 93 | 13 | |
| Slidw | 0.1 | 3 | 91 | 13 | |
| Whole plant | 0.01 | 3 | 79 | 13 | |
| whole plant | 0.1 | 5 | 79 | 14 | |
| Ear | 0.01 | 1 | 66 | - | |
| Edi | 0.1 | 1 | 81 | - | |
| Grain | 0.01 | 3 | 85 | 8.0 | − 490M2, m/z 314 \rightarrow 238, Quantitation |
| Gialli | 0.1 | 2 | 68 | - | |
| Straw | 0.01 | 3 | 75 | 21 | |
| Slidw | 0.1 | 3 | 77 | 21 | |
| Whole plant | 0.01 | 3 | 79 | 29 | |
| whole plant | 0.1 | 5 | 72 | 17 | |
| Ear | 0.01 | 1 | 76 | - | |
| Edi | 0.1 | 1 | 70 | - | 490M9, m/z 314→238, Quantitation |
| Grain | 0.01 | 3 | 81 | 1.9 | |
| Gialli | 0.1 | 2 | 78 | - | |
| Straw | 0.01 | 3 | 78 | 20 | |
| Suaw | 0.1 | 3 | 64 | 22 | |

760

| Matrix | Fortification level (mg/kg) | n | Recovery, mean (%) | RSD (%) | Analyte, MRM transition |
|--------------|-----------------------------|---|--------------------|---------|--|
| Whole plant | 0.01 | 3 | 77 | 14 | |
| Whole plant | 0.1 | 4 | 84 | 10 | |
| For | 0.01 | 1 | 86 | - | |
| Ear | 0.1 | 2 | 90 | - | Kresoxim-methyl (as 490M1), m/z; 298→102 |
| Grain | 0.01 | 2 | 78 | - | $- \frac{11}{2} \frac{298}{102}$ |
| Grain | 0.1 | 1 | 77 | - | Quantitation |
| Chrow | 0.01 | 3 | 87 | 15 | |
| Straw | 0.1 | 3 | 77 | 11 | |
| W/holo mlomt | 0.01 | 3 | 81 | 2.5 | |
| Whole plant | 0.1 | 4 | 83 | 5.3 | |
| For | 0.01 | 1 | 76 | - | 490M2, m/z 314→238, Quantitation |
| Ear | 0.1 | 2 | 72 | - | |
| Casia | 0.01 | 2 | 69 | - | |
| Grain | 0.1 | 1 | 63 | - | |
| Chrow | 0.01 | 3 | 74 | 5.6 | |
| Straw | 0.1 | 3 | 67 | 6.9 | |
| W/holo mlomt | 0.01 | 3 | 87 | 1.8 | |
| Whole plant | 0.1 | 4 | 76 | 5.6 | |
| For | 0.01 | 1 | 69 | - | |
| Ear | 0.1 | 2 | 77 | - | |
| Croin | 0.01 | 2 | 79 | - | 490M9, m/z 314 \rightarrow 238, Quantitation |
| Grain | 0.1 | 1 | 57 | - | 7 |
| Chrow | 0.01 | 3 | 60 | 21 | |
| Straw | 0.1 | 3 | 60 | 3.3 | |

Table 60 Recovery data for BASF method 350/4 measuring kresoxim-methyl (as 490M1) and metabolites 490M2 and 490M9 in barley matrices (Schroth & Martin, 2009b, Kresoxim_038)

Table 61 Recovery data for BASF method SOP-PA.0295 measuring kresoxim-methyl (as 490M1) and metabolites 490M2 and 490M9in mango (Silva & Alves, 2011, Kresoxim_049;Silva, 2011 Kresoxim_050)

| Matrix | Fortification level (mg/kg) | n | Recovery, mean (%) | RSD (%) | Analyte |
|-------------|-----------------------------|---|--------------------|---------|--|
| | 0.01 | 6 | 92 | 9 | Kresoxim-methyl (as 490M1), m/z |
| | 1 | 6 | 93 | 7 | 298 \rightarrow 222, Quantitation |
| | 0.01 | 6 | 96 | 7 | 490M2, m/z 314 \rightarrow 132, Quantitation |
| | 1 | 6 | 90 | 4 | 4901012, 111/2 3 14→132, Qualititation |
| | 0.01 | 6 | 91 | 7 | 490M9, m/z 314 \rightarrow 116, Quantitation |
| Mango fruit | 1 | 6 | 95 | 4 | 4901019, 111/2 3 14→1 16, Qualititation |
| Mango nun | 0.01 | 6 | 87 | 5 | Kresoxim-methyl (as 490M1), m/z |
| | 1 | 6 | 92 | 6 | 298 \rightarrow 102, Confirmation |
| | 0.01 | 6 | 97 | 7 | 490M2, m/z 314→238, |
| | 1 | 6 | 95 | 3 | Confirmation |
| | 0.01 | 6 | 86 | 12 | 490M9, m/z 314→122, |
| | 1 | 6 | 93 | 10 | Confirmation |

Method JP 2015C423 (Takahashi, 2016, Kresoxim_051)

Parent kresoxim-methyl was extracted from fruit samples by homogenization and shaking with water/acetone (20/80, v/v). After filtration of the mixture, the extract was evaporated to the aqueous remainder and purified on a porous diatomaceous earth column. The analyte was eluted with hexane. The eluate was evaporated to dryness and reconstituted in hexane prior to clean-up on a silica gel column. Kresoxim-methyl was eluted from the column with diethyl ether/hexanes (5/95, v/v), the solvent was evaporated to dryness and the remainder reconstituted in acetonitrile/water (50/50, v/v)

Final determination of kresoxim-methyl was done by LC-MS/MS in positive ionization mode using a Ascentis Express C18 column and monitoring the transition m/z 314-416. Quantitation was done with external standards in solvent.

| Matrix | Fortification level (mg/kg) | n | Recovery, mean (%) | RSD (%) | Analyte, MRM transition |
|-----------------|-----------------------------|---|--------------------|---------|----------------------------------|
| Unshu mandarin, | 0.01 | 5 | 93 | 5.7 | |
| pulp | 1.0 | 5 | 92 | 4.1 | Krocovin mothyl |
| Linghu mandarin | 0.01 | 5 | 94 | 2.2 | Kresoxim-methyl, m/z: 314→116 |
| Unshu mandarin, | 1.0 | 5 | 96 | 2.2 | 111/2: 314→116 |
| peel | 20 | 5 | 87 | 4.0 | |

Table 62 Recovery data for method JP 2015C423 measuring kresoxim-methyl in Unshu mandarin (Takahashi, 2016, Kresoxim_051)

Method Saku 6P-7-175 (Yabusaki, 1994, Kresoxim_052; Nakazawa & Gomei, 1994, Kresoxim_065)

Parent kresoxim-methyl was extracted from fruit samples by homogenization and shaking with acetone. After filtration of the mixture, the extract was evaporated to near dryness, diluted with water and purified on a porous diatomaceous earth column. The analyte was eluted with hexane. The eluate was evaporated to dryness and reconstituted in hexane hexane/ethyl ether (98:2, v/v) prior to clean-up on a silica gel column, followed by a florisil column. Kresoxim-methyl was eluted from the column with diethyl ether/hexanes (1/1, v/v), the solvent was evaporated to dryness and the remainder reconstituted in hexane.

Final determination of kresoxim-methyl was done by GC-NPD on a DB-17 column. Quantitation was done with external standards in solvent.

Table 63 Recovery data for method Saku 6P-7-175 measuring kresoxim-methyl in Unshu mandarin (Yabusaki, 1994, Kresoxim_052; Nakazawa & Gomei, 1994, Kresoxim_065)

| Matrix | Fortification level (mg/kg) | n | Recovery, mean (%) | RSD (%) | Analyte |
|-----------------|-----------------------------|---|--------------------|---------|-----------------|
| Unshu mandarin, | 0.1 | 2 | 86 | - | |
| pulp | 0.2 | 2 | 100 | - | |
| Unchu mondorin | 0.2 | 2 | 86 | - | Kresoxim-methyl |
| Unshu mandarin, | 0.4 | 2 | 92 | - | |
| peel | 4 | 2 | 98 | - | |

Animal materials

BASF method L0177/01 (Goecer & Class, 2011, Kresoxim_053)

Milk, egg, meat, fat, liver and kidney samples were homogenised with methanol. No further clean-up was performed, except for fat. Fat extracts were evaporated to dryness re-dissolved in n-hexane saturated with acetonitrile and partitioned against acetonitrile. The extracts are evaporated to dryness and are dissolved in acetonitrile/water (1/1, v/v) + 0.1% acetic acid.

Final determination of metabolite 490M1 was done by LC-MS/MS in negative ionization mode using a C18 column and monitoring the transition m/z 298 +02 and m/z 222 +02. Quantitation was done with external standards in matrix.

| Table 64 Recove | ery data for BASF 1 | method L0177/0 | l measuring met | tabolite 490M1 | in animal matrices |
|-----------------|---------------------|----------------|-----------------|----------------|--------------------|
| | | 7 7 | | 2 | n n |

| | | | | 0 | |
|----------|-----------------------------|---|--------------------|---------|---------------------------|
| Matrix | Fortification level (mg/kg) | Ν | Recovery, mean (%) | RSD (%) | Analyte, MRM transition |
| Milk | 0.01 | 5 | 92 | 12 | |
| IVIIIK | 0.10 | 5 | 95 | 5 | |
| Faa | 0.01 | 5 | 85 | 9 | |
| Egg | 0.10 | 5 | 93 | 4 | |
| Meat | 0.01 | 5 | 82 | 7 | motobalita 400M1 |
| weat | 0.10 | 5 | 90 | 10 | metabolite 490M1, |
| Kidaau | 0.01 | 5 | 88 | 12 | m/z: 298→102 Quantitation |
| Kidney | 0.10 | 5 | 83 | 10 | Quantitation |
| Liver | 0.01 | 5 | 86 | 11 | |
| Liver | 0.10 | 5 | 102 | 5 | |
| Eat | 0.01 | 5 | 99 | 10 | |
| Fat | 0.10 | 5 | 92 | 9 | |
| N 4111- | 0.01 | 5 | 90 | 13 | |
| Milk | 0.10 | 5 | 95 | 7 | |
| F | 0.01 | 5 | 87 | 11 | metabolite 490M1, |
| Egg | 0.10 | 5 | 94 | 3 | m/z: 222→102 ¹ |
| Maat | 0.01 | 5 | 83 | 5 | Confirmation |
| Meat | 0.10 | 5 | 92 | 8 | |
| Kidney | 0.01 | 5 | 81 | 11 | |

| Matrix | Fortification level (mg/kg) | Ν | Recovery, mean (%) | RSD (%) | Analyte, MRM transition |
|--------|-----------------------------|---|--------------------|---------|-------------------------|
| | 0.10 | 5 | 80 | 8 | |
| Liver | 0.01 | 5 | 89 | 9 | |
| Livei | 0.10 | 5 | 102 | 5 | |
| F-4 | 0.01 | 5 | 95 | 6 | |
| Fat | 0.10 | 5 | 88 | 9 | |

¹ m/z 222 [M-77]⁻ is generated as in-source fragment from m/z 298

BASF method 354/1 (Mackenroth & Dams, 1994)

Metabolites 490M2 and 490M9 were extracted from milk by homogenization with acetone. After centrifugation, aqueous KH_2PO_4 solution and ascorbic acid was added and the organic phase evaporated. The extract was partitioned against iso-octane, followed by dichloromethane. Additional clean-up was performed by SPE on a NH2-column. Analytes were eluted with a water/NaCl/formic acid/ascorbic acid/methanol. The eluate was once more partitioned against dichloromethane. After evaporation of the organic phase, the remainder was reconstituted in water/acetonitrile (9/1, v/v).

Final determination of metabolites 490M2 and 490M9 was done by LC-LC-UV at 270 nm using column switching with a NH₂-column and a C18-column.

| Matrix | Fortification level (mg/kg) | n | Recovery, mean (%) | RSD (%) | Analyte |
|--------|-----------------------------|---|--------------------|---------|---------|
| | 0.001 | 4 | 108 | 4.8 | 400142 |
| Milk | 0.025 | 4 | 88 | 4.9 | 490M2 |
| IVIIIK | 0.001 | 4 | 93 | 5.8 | 400140 |
| | 0.025 | 4 | 100 | 2.3 | 490M9 |

Table 65 Recovery data for BASF method 354/1 measuring metabolites 490M2 and 490M9 in milk

BASF 354/2 (Mackenroth & Krotzky, 1994c, Kresoxim_055)

Metabolites 490M1, 490M2 and 490M9 were extracted from animal tissues by homogenization with methanol. After filtration the organic phase was evaporated and the resultant aqueous phase shaken with Ca(OH)₂ and filtered. The solution was acidified with formic acid and aqueous KH₂PO₄ solution and ascorbic acid was added. The extract was partitioned against dichloromethane and further cleaned-up by SPE on a NH2-column. Analytes were eluted with a water/NaCl/formic acid/ascorbic acid/methanol. The eluate was once more partitioned against dichloromethane. After evaporation of the organic phase, the remainder was reconstituted in water/acetonitrile and further cleaned-up on a preparative C18 HPLC column.

Final determination of metabolites 490M2 and 490M9 was done by LC-LC-UV at 270 nm using column switching with a NH₂-column and a CN-column.

Table 66 Recovery data for BASF method 354/2 measuring metabolites 490M1, 490M2 and 490M9 in animal tissues

| Matrix | Fortification level (mg/kg) | n | Recovery, mean (%) | RSD (%) | Analyte |
|-------------|-----------------------------|---|--------------------|---------|---------|
| | 0.01 | 4 | 89 | 21 | 490M1 |
| Mucolo | 1.0 | 4 | 84 | 2.5 | 49001 |
| Muscle | 0.01 | 4 | 88 | 7.4 | 490M2 |
| | 1.0 | 4 | 89 | 2.8 | 4901012 |
| | 0.01 | 4 | 97 | 17 | 490M1 |
| | 1.0 | 4 | 80 | 6.2 | 49001 |
| Kidnov | 0.01 | 4 | 91 | 6.5 | 490M2 |
| Kidney | 1.0 | 4 | 80 | 4.5 | 4901012 |
| | 0.01 | 4 | 109 | 3.4 | 490M9 |
| | 1.0 | 4 | 82 | 3.9 | 4901019 |
| | 0.01 | 4 | 84 | 17 | 400141 |
| Liver | 1.0 | 4 | 77 | 2.2 | - 490M1 |
| Livei | 0.01 | 4 | 102 | 0.7 | 490M9 |
| | 1.0 | 4 | 72 | 5.5 | 490019 |
| | 0.01 | 4 | 56 | 8.2 | 490M1 |
| | 1.0 | 4 | 82 | 5.6 | 49001 |
| F ot | 0.01 | 4 | 53 | 12 | 400142 |
| Fat | 1.0 | 4 | 84 | 4.8 | - 490M2 |
| | 0.01 | 4 | 62 | 9.1 | 400140 |
| | 1.0 | 4 | 89 | 1.9 | 490M9 |

Independent laboratory validation of BASF methods 354/1 and 354/2 (Maxwell, 1994, Kresoxim_056)

The sample preparation was identical to BASF method 354/1 and 354/2. Only minor modifications were applied.

Table 67 Recovery data for the ILV of BASF methods 354/1 and 354/2 measuring metabolites 490M1, 490M2 and 490M9 in milk and animal tissues

| Matrix | Fortification level (mg/kg) | n | Recovery, mean (%) | RSD (%) | Analyte |
|--------|-----------------------------|----|--------------------|---------|---------|
| Milk | 0.001, 0,002, 0.1 | 12 | 90 | 14 | 490M1 |
| IVIIIK | 0.001, 0,002, 0.1 | 12 | 87 | 7.7 | 490M9 |
| Liver | 0.01, 1.0 | 6 | 74 | 5.3 | 490M1 |
| LIVEI | 0.01, 1.0 | 6 | 90 | 16 | 490M9 |
| | 0.01, 1.0 | 6 | 82 | 10 | 490M1 |
| Kidney | 0.01, 1.0 | 6 | 84 | 12 | 490M2 |
| | 0.01, 1.0 | 6 | 83 | 13 | 490M9 |
| Muscle | 0.01, 1.0 | 8 | 91 | 5.8 | 490M1 |
| Muscle | 0.01, 1.0 | 8 | 87 | 13 | 490M2 |
| | 0.01, 1.0 | 12 | 75 | 19 | 490M1 |
| Fat | 0.01, 1.0 | 12 | 81 | 24 | 490M2 |
| | 0.01, 1.0 | 12 | 96 | 27 | 490M9 |

Stability of pesticides in stored analytical samples

Plant matrices

The storage stability of kresoxim-methyl and metabolites 490M2 and 490M9 under frozen conditions in wheat grain (high starch), soy bean (oily) and dried pea (high protein) was investigated by Class & Senciuc (2009, Kresoxim_057).

The three analytes were dosed together to homogenised samples at a rate of 0.5 mg/kg. These fortified commodities were stored deep frozen (-20 °C) and analysed for kresoxim-methyl and metabolites 490M2 and 490M9 after 0, 1, 3, 6, 9, 12, 18 and 24 month. For each fortification level three samples were measured. All samples were analysed according to BASF method 445/0.

Table 68 Storage stability of kresoxim-methyl and metabolites 490M2 and 490M9 in wheat grain, soy bean and dried peas, fortified at 0.5 mg/kg

| Storage | Kresoxim-methyl | | 490M2 | | 490M9 | 490M9 | |
|------------|-----------------|-----------------|----------------|-----------------|----------------|-----------------|--|
| period | Mean remaining | Mean concurrent | Mean remaining | Mean concurrent | Mean remaining | Mean concurrent | |
| (month) | (%) | recovery (%) | (%) | recovery (%) | (%) | recovery (%) | |
| Wheat grai | n | | | | | | |
| 0 | 75 | 81 | 102 | 104 | 97 | 104 | |
| 1 | 83 | 93 | 93 | 102 | 91 | 101 | |
| 3 | 72 | 78 | 100 | 104 | 95 | 103 | |
| 6 | 85 | 89 | 90 | 98 | 99 | 105 | |
| 9 | 71 | 110 | 87 | 100 | 97 | 96 | |
| 12 | 75 | 96 | 95 | 109 | 86 | 97 | |
| 18 | 80 | 79 | 93 | 106 | 80 | 94 | |
| 24 | 102 | 109 | 107 | 109 | 89 | 101 | |
| Dried pea | | | | | | | |
| 0 | 84 | 90 | 102 | 106 | 98 | 100 | |
| 1 | 100 | 100 | 88 | 96 | 80 | 98 | |
| 3 | 100 | 108 | 95 | 105 | 90 | 103 | |
| 6 | 85 | 98 | 85 | 80 | 88 | 87 | |
| 9 | 85 | 102 | 83 | 86 | 82 | 79 | |
| 12 | 94 | 108 | 98 | 105 | 91 | 98 | |
| 18 | 78 | 93 | 86 | 96 | 80 | 88 | |
| 24 | 109 | 109 | 107 | 103 | 105 | 96 | |
| Soya bean | | | | | | | |
| 0 | 78 | 81 | 96 | 102 | 97 | 99 | |
| 1 | 87 | 99 | 85 | 91 | 53 | 85 | |
| 2 | - | - | - | - | 45 | 98 | |
| 3 | 82 | 95 | 97 | 105 | 36 | 98 | |
| 6 | 50 | 90 | 73 | 75 | 34 | 89 | |

| Storage | Kresoxim-methyl | | 490M2 | | 490M9 | |
|---------|-----------------|-----------------|----------------|-----------------|----------------|-----------------|
| period | Mean remaining | Mean concurrent | Mean remaining | Mean concurrent | Mean remaining | Mean concurrent |
| (month) | (%) | recovery (%) | (%) | recovery (%) | (%) | recovery (%) |
| 9 | 45 | 94 | 75 | 82 | 28 | 82 |
| 12 | 50 | 91 | 80 | 90 | 7 | 84 |
| 18 | 65 | 88 | 75 | 80 | 12 | 86 |
| 24 | 67 | 105 | 97 | 97 | 8 | 81 |

The storage stability of kresoxim-methyl and the glucosides of metabolites 490M2 and 490M9 under frozen conditions in grapes (acidic), apple (high water content) and processed commodities (apple pomace and juice) was investigated by Jordan & Riley (1999, Kresoxim_058) and Movasaghi & Riley (1998. Kresoxim_059)

Kresoxim-methyl was dosed to homogenised samples at 1 mg/kg, while the glucosides of metabolites 490M2 and 490M9 were dosed to grapes and apples at rates ranging from 0.26–0.52 mg/kg. These fortified commodities were stored deep frozen (<- 10 °C) and analysed after 2, 6, 9, 12 and 26 month. For each fortification level two samples were measured. All samples were analysed according to BASF method 350/3.

Table 69 Storage stability of kresoxim-methyl and the glucosides of metabolites 490M2 and 490M9 in grapes, apple and processed commodities

| Storage | Kresoxim-methyl | | Glucoside of 490M2 | | Glucoside of 490M | 9 |
|-------------|-----------------|-----------------|--------------------|----------------------------------|-------------------|-----------------|
| period | Mean remaining | Mean concurrent | Mean remaining | Mean remaining Mean concurrent N | | Mean concurrent |
| (month) | (%) | recovery (%) | (%) | recovery (%) | (%) | recovery (%) |
| Whole grap | e | | | | | |
| 0 | - | 84 | - | 92 | - | 113 |
| 2 | 75 | 81 | 103 | 123 | 114 | 116 |
| 6 | 86 | 89 | 74 | 99 | 113 | 107 |
| 9 | - | - | 123 | 104 | 123 | 116 |
| 12 | 90 | 79 | 83 | 68 | 99 | 79 |
| 26 | 76 | 108 | 87 | n/a | 87 | n/a |
| Whole appl | e | | | | | |
| 0 | - | 86 | - | 122 | - | 112 |
| 2 | 73 | 83 | 113 | 103 | 120 | 102 |
| 6 | 74 | 92 | 125 | 119 | 108 | 107 |
| 12 | 70 | 81 | 99 | 98 | 111 | 110 |
| Apple wet | oomace | | | | | |
| 0 | - | 83 | - | - | - | - |
| 2 | 83 | 81 | - | - | - | - |
| 6 | 84 | 100 | - | - | - | - |
| 12 | 74 | 86 | - | - | - | - |
| Apple juice | | | | | | |
| 0 | - | 95 | - | - | - | - |
| 2 | 68 | 87 | - | - | - | - |
| 6 | 87 | 83 | - | - | - | - |
| 12 | 91 | 110 | - | - | - | - |

The storage stability of kresoxim-methyl under frozen conditions in cucumber (high water content) was investigated by Abdel-Baky & Riley (1998, Kresoxim_060)

Kresoxim-methyl was dosed to homogenised cucumber samples at 1 mg/kg, stored deep frozen (<-10 °C) and analysed after 0, 2, 6 and 12 month. For each fortification level two samples were measured. All samples were analysed according to BASF method 350/3.

Table 70 Storage stability of kresoxim-methyl in cucumber

| Storage period (month) | Kresoxim-methyl | | | | | |
|------------------------|---|----|--|--|--|--|
| | Mean remaining (%) Mean concurrent recovery (%) | | | | | |
| Cucumber | | | | | | |
| 0 | 82 | 80 | | | | |
| 2 | 80 | 93 | | | | |
| 6 | 87 | 79 | | | | |
| 12 | 63 | 78 | | | | |

The storage stability of kresoxim-methyl and the glucosides of metabolites 490M2 and 490M9 under frozen conditions in pecan nuts (oily) was investigated by Thornton (1998, Kresoxim_061)

Kresoxim-methyl was dosed to homogenised samples at 1 mg/kg, while the glucosides of metabolites 490M2 and 490M9 were dosed at 0.28 mg/kg and 0.47 mg/kg, respectively. These fortified commodities were stored deep frozen (<-10 °C) and analysed after 2 and 6 month. For each fortification level two samples were measured. All samples were analysed according to BASF method D9611.

Table 71 Storage stability of kresoxim-methyl and the glucosides of metabolites 490M2 and 490M9 in pecan nuts

| Storage | Kresoxim-methyl | | Glucoside of 490M2 | | Glucoside of 490M9 | |
|-------------------|-----------------|-----------------|--------------------|-----------------|--------------------|-----------------|
| period (month) | Mean remaining | Mean concurrent | Mean remaining | Mean concurrent | Mean remaining | Mean concurrent |
| (monun) | (%) | recovery (%) | (%) | recovery (%) | (%) | recovery (%) |
| Pecan nut | | | | | | |
| 0 | - | 84 | - | 114 | - | 120 |
| 2 | 82 | 82 | - | - | - | - |
| 6 | 76 | 78 | 85 | 90 | 83 | 92 |

The storage stability of kresoxim-methyl under frozen conditions in apples (high water content) was investigated by Mackenroth & Krotzky, (1994d, Kresoxim_062)

Kresoxim-methyl was dosed to homogenised samples at 1 mg/kg, stored deep frozen (<-20 °C) and analysed after 0, 7 days, 1, 2, 6 and 10 month. For each fortification level two samples were measured. All samples were analysed according to BASF method 351/2.

| Table 72 Storage stability | of kresoxim-methyl in apple |
|----------------------------|-----------------------------|
| | |

| Storage period (month) | Kresoxim-methyl | | | | | |
|------------------------|--------------------|------------------------------|--|--|--|--|
| | Mean remaining (%) | Mean concurrent recovery (%) | | | | |
| Apple | | | | | | |
| 0 | 96 | 96 | | | | |
| 0.25 (7 days) | 89 | 86 | | | | |
| 1 | 83 | 96 | | | | |
| 2 | 87 | 88 | | | | |
| 6 | 80 | 84 | | | | |
| 10 | 89 | 90 | | | | |

The storage stability of kresoxim-methyl and metabolites 490M2 and 490M9 under frozen conditions in wheat matrices was investigated by Krotzky (1994 Kresoxim_063).

The parent kresoxim-methyl was dosed to homogenised wheat grain and green material at a rate of 1 mg/kg. Additionally, the stabilities of kresoxim-methyl (in straw) and the glucosides of metabolites 490M2 and 490M9 (in green matter and straw) were determined in incurred field samples. Samples were stored deep frozen (-20 °C), analysed after 0, 0.25 (7days) 1, 3, 6 and 7 month. For each fortification level two samples were measured. All samples were analysed according to BASF method 351/2 and 350/1.

Table 73 Storage stability of kresoxim-methyl and the glucosides of metabolites 490M2 and 490M9 in wheat matrices

| Storage | Kresoxim-methyl | | Glucoside of 490M2 | Glucoside of 490M2 | | 9 |
|------------|-----------------------|-----------------|--------------------|--------------------|----------------|-----------------|
| period | Mean remaining | Mean concurrent | Mean remaining | Mean concurrent | Mean remaining | Mean concurrent |
| (month) | (%) | recovery (%) | (%) | recovery (%) | (%) | recovery (%) |
| Wheat gree | en matter (spiked sam | ple) | | | | |
| 0 | 99 | 93 | n/d | 79 | - | 82 |
| 0.25 | 83 | 82 | n/d | 93 | - | 90 |
| 1 | 109 | 104 | n/d | - | 102 | - |
| 6 | 92 | 100 | n/d | 74 | 102 | 80 |
| Wheat grai | n (spiked sample) | | | | | |
| 0 | 81 | 77 | - | - | - | - |
| 0.25 | 84 | 76 | - | - | - | - |
| 1 | 69 | 76 | - | - | - | - |
| 3 | 71 | 72 | - | - | - | - |
| 7 | 90 | 86 | - | - | - | - |
| Wheat stra | w (incurred sample) | | | | | |
| 0 | - | 74 | - | 66 | - | 72 |

766

| Storage | Kresoxim-methyl | | Glucoside of 490M2 | | Glucoside of 490M9 | |
|---------|--------------------------------|--------------|--------------------|-----------------|--------------------|-----------------|
| period | Mean remaining Mean concurrent | | Mean remaining | Mean concurrent | Mean remaining | Mean concurrent |
| (month) | (%) | recovery (%) | (%) | recovery (%) | (%) | recovery (%) |
| 2 | 183/97 | 106 | 115/n/a | 79 | 101/20 | 81 |
| 3 | 161/98 | 95 | 149/134 | 84 | 144/155 | 75 |

Animal matrices

The storage stability of kresoxim-methyl metabolites 490M1, 490M2 and 490M9 under frozen conditions in animal tissues was investigated by Mewis (2016, Kresoxim_064).

The three analytes were individually dosed to homogenised samples at a rate of 0.1 mg/kg, stored deep frozen (<-18 °C) and analysed after 0, 6, 12 and 15 month. Two samples were measured per time point.. All samples were analysed according to BASF method L0177/01.

Table 74 Storage stability of kresoxim-methyl metabolites 490M1, 490M2 and 490M9 in animal matrices fortified at 0.1 mg/kg

| Storage | 490M1 | | 490M2 | | 490M9 | | |
|--------------|----------------|-----------------|----------------|-----------------|----------------|--|--|
| period | Mean remaining | Mean concurrent | Mean remaining | Mean concurrent | Mean remaining | Mean concurrent | |
| , (month) | (%) | recovery (%) | (%) | recovery (%) | (%) | recovery (%) | |
| Milk | | | | | 1 () | | |
| 0 | - | - | 99 | 108 | | | |
| 6 | - | - | 85 | 100 | 96 | 100 | |
| 12 | - | - | 83 | 103 | 103 | 100 | |
| 15 | - | - | 86 | 1 | 105 | | |
| Liver | | | | ÷ | | | |
| 0 | 100 | | - | - | 98 | | |
| 6 | 93 | 130 | - | - | 103 | 100 | |
| 12 | 89 | 130 | - | - | 101 | 100 | |
| 15 | 87 | | - | - | 97 | | |
| Muscle | | | | | | | |
| 0 | 93 | | 100 | 87 | - | - | |
| 6 | 89 | 95 | 98 | | - | - | |
| 12 | 95 | 90 | 100 | 87 | - | - | |
| 15 | 93 | | 87 | | - | - | |
| Kidney | | | | | | | |
| 0 | 90 | | 105 | | 105 | | |
| 6 | 97 | 92 | 103 | 89 | 99 | 86 | |
| 12 | 77 | 92 | 99 | 09 | 95 | 00 | |
| 15 | 81 | | 100 | | 90 | <u>] </u> | |
| Fat | | | | | | | |
| 0 | 101 | | 107 | | 90 | | |
| 6 | 97 | 104 | 109 | 99 | 103 | 101 | |
| 12 | 100 | 104 | 109 | 77 | 97 | 101 | |
| 15 | 97 | | 95 | | 91 | 1 | |

Stability of residues in stored analytical samples

The stability of kresoxim-methyl in different solvents was investigated by Funk & Mackenroth (2001, Kresoxim_151).

Methanol, methanol/water, iso-octane and acetonitrile were fortified with kresoxim-methyl at a concentration of 10 mg/L, followed by storage at 4 °C in the dark or RT in daylight. Samples were analysed after 0, 1, 7, 14, 30, 60 and 120 days. For each fortification level three samples were measured. All samples were analysed using HPLC-UV.

Table 75 Degradation of kresoxim-methyl in different solvents after 118 days of storage

| Compound | Methanol (%) | | Methanol/Water (%) | | Iso-octane (%) | | Acetonitrile (%) | |
|-----------------|--------------|-----------------|--------------------|-----------------|-------------------|------|------------------|-----------------|
| Compound | 4 °C1 | RT ² | 4 °C ^a | RT ^b | 4 °C ^a | RT⁵ | , í í | RT ^b |
| Kresoxim-methyl | 12.3 | 12.7 | 7.4 | 9.6 | 3.1 | 12.7 | 1.2 | 2.4 |

^a In the dark

^b Room temperature at daylight

| Compound | Methan | ol (%) | Methanol/ | Water (%) | lso-octa | ne (%) | Acetonitrile (%) | |
|-----------------|--------|--------|-----------|-----------|----------|--------|------------------|-----|
| Compound | 4 °Cª | RT⁵ | 4 °Ca | RT⁵ | 4 °Ca | RT⁵ | 4 °Ca | RT⁵ |
| Kresoxim-methyl | 85 | 77 | 199 | 127 | 484 | 68 | 2349 | 706 |

Table 76 Time interval to reach 90% of the starting concentration

^a In the dark

^b Room temperature at daylight

USE PATTERN

Kresoxim-methyl is a systemic fungicide globally registered in many countries for the control of scab and other fungal diseases. In the following table, GAP information on all crops supported with residue data is summarized in Table 77.

Table 77 List of uses of kresoxim-methyl

| | | Formulation | | Application | | | | |
|--|---------------------------------|--------------------------------|------|--------------|---------------------------|------------------|---|---------------|
| Crop/ Commodity | Country | Active substance content | Туре | Method | Rate | Water volume | No or Seasonal max. (interval) | PHI (days) |
| Citrus | Japan | 500 g/kg | WP | Foliar spray | 25 g ai/hL | - | 3 | 14 |
| Citrus | Turkey | 500 g/kg | WG | Foliar spray | 12.5 g ai/hL | - | 2-4 (12-14 days) | 35 |
| Apple | Brazil | 500 g/L | SC | Foliar spray | 10 g ai/hL | 1000 L/ha | 3 (8-12 days) | 35 |
| Apple | Japan | 500 g/kg | WP | Foliar spray | 33 g ai/hL | - | 3 | 1 |
| Pear | Japan | 500 g/kg | WP | Foliar spray | 25 g ai/hL | - | 3 | 1 |
| Small stone fruits | Japan | 500 g/kg | WP | Foliar spray | 25 g ai/hL | - | 3 | 7 |
| Peach/Nectarine | Japan | 500 g/kg | WP | Foliar spray | 25 g ai/hL | - | 3 | 1 |
| Peach | Peru | 500 g/kg | WG | Foliar spray | 150 g ai/ha 15 g ai/hL | - | 2 (14 days) | 30 |
| Plum | Japan | 500 g/kg | WP | Foliar spray | 25 g ai/hL | - | 3 | 7 |
| Strawberry | Netherlands | 500 g/kg | WG | Foliar spray | 150 g ai/ha | 600-1200 L/ha | 3 (7 days) | 7 |
| Currant | United Kingdom | 500 g/kg | WG | Foliar spray | 100 g ai/ha | 500-1600 L/ha | 3 (10-14 days) | 14 |
| Grape | Japan | 500 g/kg | WP | Foliar spray | 25 g ai/hL | - | 3 | 14 |
| Grape | USA | 500 g/kg | WG | Foliar spray | 224 g ai/ha | >94 L/ha | 4 (7-21 days) | 14 |
| Mango | Brazil | 100 g/L | SC | Foliar spray | 120 g ai/ha 12 g ai/hL | 1000 L/ha | 2 (15 days) | 7 |
| Mango | Japan | 500 g/kg | WP | Foliar spray | 17 g ai/hL | - | 3 | 1 |
| Onion | Netherlands | 500 g/L | SC | Foliar spray | 200 g ai/ha | 200-400 L/ha | 3 (7-10 days) | - |
| Garlic | Brazil | 100 g/L | SC | Foliar spray | 70 g ai/ha | 800 L/ha | 4 (10 days) | 7 |
| Shallot | Taiwan, Province of China | 440 g/L | SC | Foliar spray | 400 g ai/ha | 750-2000 L/ha | 1-2 (7 days) | 10 |
| Leek | Netherlands | 500 g/L | SC | Foliar spray | 375 g ai/ha | 200-800 L/ha | 3 (10-14 days) | 14 |
| Cucurbits (cantaloupe, cucumber, gherkin, melon, squash, pumpkin, watermelon) | USA | 500 g/kg | WG | Foliar spray | 168 g ai/ha | Not reported | 4 (7-10 days) | 0 |
| Cucurbits (except cucmber) | Taiwan, Province of China | 500 g/kg | WG | Foliar spray | 150-200 g ai/ha | 900-1200 L/ha | 1-3 (7 days) | 6 |

768

| | | Formulation | | Application | | | | |
|--|---------------------------------|--------------------------------|------|--------------|--------------------|------------------|---|---------------|
| Crop/ Commodity | Country | Active substance content | Туре | Method | Rate | Water volume | No or Seasonal max. (interval) | PHI (days) |
| Melon | Taiwan, Province of China | 500 g/kg | WG | Foliar spray | 150-200 g ai/ha | 900-1200 L/ha | 1-3 (7 days) | 6 |
| Pumpkin, courgette, melon, cucumber, watermelon | Mexico | 500 g/kg | WG | Foliar spray | 100 g ai/ha | 400 L/ha | 4 (7 days) | 3 |
| Pepper | Brazil | 100 g/L | SC | Foliar spray | 100 g ai/ha | 400 L/ha | 4 (10 days) | 3 |
| Tomato | Brazil | 500 g/L | SC | Foliar spray | 200 g ai/ha | 1000 L/ha | 2 (7 days) | 3 |
| Grape (leaves) | Turkey | 100 g/L | SC | Foliar spray | 30 g ai/ha | 500-1000 L/ha | 3 (10-14 days) | 14 |
| Beetroot | Germany | 125 g/L | SC | Foliar spray | 125 g ai/ha | 200-400 L/ha | 2 (10-14 days) | 28 |
| Sugar beet | Germany | 125 g/L | SC | Foliar spray | 125 g ai/ha | 200-400 L/ha | 1 | 28 |
| Turnip | Germany | 125 g/L | SC | Foliar spray | 125 g ai/ha | 200-400 L/ha | 1 | 28 |
| Cereals (barley, oat, wheat, triticale, rye) | Chile | 125 g/L | SC | Foliar spray | 125 g ai/ha | 150-200 I/ha | 2 | 35 |
| Cereals (barley, oat, wheat, triticale, rye) | United Kingdom | 125 g/L | SE | Foliar spray | 125 g ai/ha | 200 L/ha | 2 | - |
| Pecans | USA | 500 g/kg | WG | Foliar spray | 168 g ai/ha | 470 L/ha | 3 (14-21 days) | 45 |
| Olives (table and for oil production) | France | 500 g/kg | WG | Foliar spray | 100 g ai/ha | 700-1000 L/ha | 3 | 30 |

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

Residue levels were reported as measured. Application rates were always reported as kresoxim-methyl equivalents. When residues were not detected they are shown as below the LOQ, e.g., < 0.01 mg/kg. Application rates, spray concentrations and mean residue results have generally been rounded to the even with two significant figures. HR and STMR values from the trials conducted according to maximum GAP have been used for the estimation of maximum residue levels. These results are underlined.

Laboratory reports included method validation including batch recoveries with spiking 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 % recovery.

| Table 78 Kresoxim-methyl – supervised residue trials |
|--|
|--|

| Commodity | Indoor/Outdoor | Treatment | Countries | Table |
|---------------|----------------|--------------|---|-----------------------|
| Citrus | Indoor/outdoor | Foliar spray | Japan, South Africa | Table 5- Table 84 |
| Pome fruit | Outdoor | Foliar spray | Germany, France, Poland, Hungary, Soth Africa | Table 85- Table 86 |
| Stone fruit | Outdoor | Foliar spray | Spain, France | Table 87- Table 88 |
| Black currant | Outdoor | Foliar spray | Denmark, Sweden, United Kingdom | Table 89 |
| Strawberry | Indoor/outdoor | Foliar spray | Germany, United Kingdom, The Netherlands, Spain, Belgium, Sweden, Denmark | Table 90- Table 91 |

| Commodity | Indoor/Outdoor | Treatment | Countries | Table |
|---------------------------|----------------|--------------|--|-------------------------|
| Grape | Outdoor | Foliar spray | USA | Table 93 |
| Mango | Outdoor | Foliar spray | Brazil | Table 3 |
| Leek | Outdoor | Foliar spray | Belgium, The Netherlands, France, Germany | Table 99 |
| Onion | Outdoor | Foliar spray | Germany, The Netherlands, Brazil | Table 96- Table 97 |
| Garlic | Outdoor | Foliar spray | Brazil | Table 98 |
| Cucumber | Indoor/outdoor | Foliar spray | USA, Spain, | Table 100- Table 101 |
| Squash | Outdoor | Foliar spray | USA | Table 103 |
| Gherkin | Outdoor | Foliar spray | Germany | Table 102 |
| Melon | Indoor/outdoor | Foliar spray | France, Spain, USA | Table 104- Table 106 |
| Sweet pepper | Indoor/outdoor | Foliar spray | Brazil, Spain | Table 107- Table 108 |
| Tomato | Indoor/outdoor | Foliar spray | Brazil, Spain | Table 110- Table 111 |
| Grape leaves | Outdoor | Foliar spray | Germany, France, Italy, Spain, Greece | Table 113 |
| Sugar beet | Outdoor | Foliar spray | Germany, France | Table 114 |
| Wheat | Outdoor | Foliar spray | Spain, Italy, France, Germany, The Netherlands, Belgium, United Kingdom | Table 115 |
| Barley | Outdoor | Foliar spray | Spain, Italy, France, Greece | Table 116 |
| Pecan | Outdoor | Foliar spray | USA | Table 117 |
| Olives for oil production | Outdoor | Foliar spray | Spain | Table 94 |

Citrus

Mandarin

Three greenhouse trials were conducted on mandarin in Japan in the 2015 growing season (Takahashi, 2016, Kresoxim_051). Mandarins received spray three applications of kresoxim-methyl at nominal rates of 25 g ai/hL with a 7±1 day interval between applications. Samples of mandarins were collected 14, 21 and 28 days after the last application. Residues of kresoxim-methyl were determined using method JP 2015C423 with a limit of quantification at 0.01 mg/kg. Overall mean procedural recoveries in mandarin peel and pulp spiked with kresoxim-methyl are listed in

Table 62.

During the 1994 growing season 4 supervised field trial were conducted in Japan (Yabusaki, 1994, Kresoxim_052; Nakazawa & Gomei, 1994, Kresoxim_065) at nominal rates of three times 25 g ai/hL with a 7 day interval between applications. Samples of mandarins were collected 14, 28 and 42 days after the last application. Residues of kresoxim-methyl were determined using method Saku 6P-7-175 with a limit of quantification in pulp and peel of 0.2 mg/kg and 0.4 mg/kg, respectively. Overall mean procedural recoveries in mandarin peel and pulp spiked with kresoxim-methyl are listed in Table 63.

Table 79 Residues of kresoxim-methyl in Unshu mandarin following outdoor and indoor foliar treatment (cGAP Japan: 3×25 g ai/hL; 14 day PHI)

| Location, | Treatment method | Application rate | Growth stage at final | DALT | Crop part | Residue Found (mg/kg) | Report/Trial No., Reference |
|--------------------------------|------------------|------------------|-----------------------|----------|--------------|------------------------------|--------------------------------|
| Year (variety) | | (g ai/hL) | application | | | Kresoxim-methyl ^a | Storage period |
| Japan | Foliar spray | 3×25 | Fruit | 14 | Pulp | 0.05 | Study: 2016/8000283 |
| Wakayama- | | | maturing | 21 | | 0.04 | Trial: 1 |
| Shoku | | | stage | 28 | | 0.05 | Takahashi, 2016, |
| 2015 | | | | 14 | Peel | 5.6 | Kresoxim_051 |
| (Nichinan No. 1) | | | | 21 | | 5.5 | Max. frozen storage: 1.5 |
| Greenhouse | | | | 28 | | 5.0 | month |
| | | | | 14 | Whole | 1.2 | |
| | | | | 21 | fruit | 1.3 | |
| | | | | 28 | | 1.3 | |
| Japan | Foliar spray | 3×25 | Fruit growth | 14 | Pulp | 0.02 | Study: 2016/8000283 |
| Nichi-Shoku-Bo- | | | period – | 21 | | 0.01 | Trial: 2 |
| Kochi | | | Fruit | 28 | | <0.01 | Takahashi, 2016, |
| 2015 | | | coloring/ | 14 | Peel | 3.5 | Kresoxim_051 |
| (Nichinan No. 1) | | | maturing | 21 | | 2.3 | Max. frozen storage: 2.5 |
| Greenhouse | | | stage | 28 | | 1.9 | month |
| | | | | 14 | Whole | 0.64 | |
| | | | | 21 | fruit | 0.40 | |
| | | | | 28 | | 0.31 | |
| Japan | Foliar spray | 3×25 | Harvest | 14 | Pulp | 0.05 | Study: 2016/8000283 |
| Nichi-Shoku-Bo- | | | season | 21 | | 0.03 | Trial: 2 |
| Miyazaki | | | | 28 | | 0.04 | Takahashi, 2016, |
| 2015 | | | | 14 | Peel | 15 | Kresoxim_051 |
| (Nichinan No. 1) Greenhouse | | | | 21 | | 17 15 | Max. frozen storage: 0.5 month |
| Greenhouse | | | | 28 14 | Whole | 3.4 | month |
| | | | | | | | |
| | | | | 21 28 | fruit | 4.0 3.3 | |
| Japan | Foliar spray | 3×25 | Not | 14 | Pulp | 0.61 | Study: 1994/8000001 |
| Aichi | ruliai spiay | 3×20 | specified | 28 | Pulp | 0.46 | Trial: 1 |
| 1994 | | | specified | 42 | | 0.40 | Nakazawa & Gomei, |
| (Miyagawa early) | | | | 14 | Peel | 8.2 | 1994, Kresoxim_065 |
| Field | | | | 28 | 1 001 | 3.9 | Max. frozen storage: 4 |
| 1 Iolu | | | | 42 | | 4.5 | month |
| | | | | 14 | Whole | 1.9 | |
| | | | | 28 | fruit | 1.3 | |
| | | | | 42 | | 1.6 | |
| Japan | Foliar spray | 3×25 | Not | 14 | Pulp | 0.56 | Study: 1994/8000002 |
| Aichi | | | specified | 28 | | 0.20 | Trial: 1 |
| 1994 | | | | 42 | | 0.36 | Yabusaki, 1994, |
| (Miyagawa early) | | | | 14 | Peel | 9.9 | Kresoxim_052 |
| Field | | | | 28 | | 3.4 | Max. frozen storage: 3.6 |
| | | | | 42 | | 4.2 | month |
| | | | | 14 | Whole | 2.3 | 7 |
| | | | | 28 | fruit | 0.73 | |
| | | | | 42 | | 1.1 | |
| Japan | Foliar spray | 3×25 | Not | 14 | Pulp | 0.32 | Study: 1994/8000001 |
| Tokushima | | | specified | 28 | | 0.61 | Trial: 2 |
| 1994 | | | | 42 | | 0.48 | Nakazawa & Gomei, |
| (Nankan No. 20) | | | | 14 | Peel | 17 | 1994, Kresoxim_065 |
| Field | | | | 28 | | 12 | Max. frozen storage: 1.7 |
| | | | | 42 | | 10 | month |

| Location, | Treatment method | Application rate (g ai/hL) | Growth stage at final application | DALT | Crop part | Residue Found (mg/kg) | Report/Trial No., Reference |
|----------------------------|---------------------|----------------------------------|---|----------------|----------------|---|--|
| Year (variety) | | (g ai/nL) | аррисации | 14 28 42 | Whole fruit | Kresoxim-methyl ^a 3.7 3.8 3.1 | Storage period |
| Japan Tokushima 1994 | Foliar spray | 3×25 | Not specified | 14 28 42 | Pulp | 0.57 0.49 0.58 | Study: 1994/8000002 Trial: 2 Yabusaki, 1994, |
| (Nankan No. 20) Field | | | | 14 28 42 | Peel | 10 9.6 9.0 | Kresoxim_052 Max. frozen storage: 0.5 month |
| | | | | 14 28 42 | Whole fruit | 2.5 2.5 2.4 | |

^a Mean of duplicate analysis.

As two trials were conducted in Aichi and Tokushima at the same location and time, using the same variety, they were not considered as independent. Hence, here only the higher residues of these trials were considered.

Orange

Field trials were conducted on oranges in South Africa during the 1996 or 1997/1998 growing seasons (Viljoen & Zyl, 1997a, Kresoxim_066; Viljoen & Zyl, 1997b, Kresoxim_067; Viljoen & Zyl, 1999, Kresoxim_068, Viljoen & Zyl, 1998, Kresoxim_069). Oranges received 1–3 spray applications of kresoxim-methyl at nominal rates ranging between 10–20 g ai/hL. Samples of oranges were collected at various time points after the last application. Residues of kresoxim-methyl were determined using BASF method 351/2 with a limit of quantification at 0.01 mg/kg. Overall mean procedural recoveries for kresoxim-methyl in orange pulp spiked at 0.04–2.0 mg/kg, peel spiked at 0.04–4.0 mg/kg and whole fruit spiked at 0.04–0.4 mg/kg were 94% (n=15), 93% (n=16) and 90% (n=2), respectively

Table 80 Residues of kresoxim-methyl in orange following foliar treatment (cGAP Turkey: 4×12.5 g ai/hL; 35 day PHI)

| Location, | Treatment method | Application rate (g ai/hL) | Growth stage at final | DALT | Crop part | Residue Found (mg/kg) Kresoxim-methyl ^a | Report/Trial No., Reference |
|----------------|---------------------|----------------------------------|-----------------------------|------|--------------|--|--------------------------------|
| Year (variety) | | (g ai/iic) | application | | | Kresoxiini-meuryi | Storage period |
| South Africa | Foliar | 3×10 | Not stated | 0 | Pulp | <0.01 | Study: 1997/11323 |
| Nelspruit | spray | | | 7 | | <0.01 | Trial: |
| 1996 | | | | 14 | | <0.01 | F/96/801/ZA9/052/A |
| (Valencia) | | | | 28 | | <0.01 | Treatment 1 |
| | | | | 56 | | <0.01 | Viljoen & Zyl, 1997a, |
| | | | | 0 | Peel | 0.61 | Kresoxim_066 |
| | | | | 7 | | 0.75 | Max. frozen storage: 15 |
| | | | | 14 | | 0.71 | month |
| | | | | 28 | | 0.49 | |
| | | | | 56 | | 0.28 | |
| | | | | 0 | Whole | 0.22 | |
| | | | | 7 | fruit | 0.25 | |
| | | | | 14 | | 0.24 | |
| | | | | 28 | | 0.15 | |
| | | | | 56 | | 0.08 | |
| | Foliar | 3×20 | Not stated | 0 | Pulp | 0.03 | Study: 1997/11323 |
| | spray | | | 7 | | 0.01 | Trial: |
| | | | | 14 | | 0.01 | F/96/801/ZA9/052/A |
| | | | | 28 | | <0.01 | Treatment 2 |
| | | | | 56 | | <0.01 | Viljoen & Zyl, 1997a, |
| | | | | 0 | Peel | 2.45 | Kresoxim_066 |
| | | | | 7 | | 2.25 | Max. frozen storage: 15 |
| | | | | 14 | | 2.10 | month |
| | | | | 28 | | 1.30 | |
| | | | | 56 | | 1.15 | |

| Location, | Treatment method | Application rate (g ai/hL) | Growth stage at final | DALT | Crop part | Residue Found (mg/kg) Kresoxim-methyl ^a | Report/Trial No., Reference |
|----------------|---------------------|----------------------------------|-----------------------------|-----------|--------------|--|---|
| Year (variety) | | - | application | | | | Storage period |
| | | | | 0 | Whole | 0.88 | |
| | | | | 7 | fruit | 0.77 | |
| | | | | 14 28 | | 0.67 0.42 | |
| | | | | 28 56 | | 0.42 | |
| | Foliar | 3×20 plus | Not stated | 0 | Pulp | 0.09 | Study: 1997/11323 |
| | spray | citrex mineral | Not Stated | 7 | 1 dip | 0.05 | Trial: |
| | | oil | | 14 | | 0.06 | F/96/801/ZA9/052/A |
| | | | | 28 | | 0.03 | Treatment 3 |
| | | | | 56 | | 0.03 | Viljoen & Zyl, 1997a, |
| | | | | 0 | Peel | 4.95 | Kresoxim_066 |
| | | | | 7 | | 5.00 | Max. frozen storage: 15 |
| | | | | 14 | | 4.30 | month |
| | | | | 28 56 | | 2.45 2.35 | |
| | | | | 0 | Whole | 2.35 1.9 | |
| | | | | 7 | fruit | 1.9 | |
| | | | | 14 | nun | 1.6 | |
| | | | | 28 | | 0.77 | |
| | | | | 56 | | 0.64 | |
| South Africa | Foliar | 2×10 | Not stated | 0 | Pulp | <0.01 | Study: 1997/11299 |
| Nelspruit | spray | | | 7 | | <0.01 | Trial: F/96/801/ZA9/052 |
| 1996 | | | | 14 | | <0.01 | Treatment 1 |
| (Valencia) | | | | 28 | | < 0.01 | Viljoen & Zyl, 1997b, |
| | | | | 56 112 | | <0.01 <0.01 | Kresoxim_067 Max. frozen storage: 15 |
| | | | | 0 | Peel | 0.80 | month |
| | | | | 7 | Feel | 0.72 | month |
| | | | | 14 | | 0.51 | |
| | | | | 28 | | 0.40 | |
| | | | | 56 | | 0.21 | |
| | | | | 112 | | 0.29 | |
| | | | | 0 | Whole | 0.27 | |
| | | | | 7 | fruit | 0.28 | |
| | | | | 14 28 | | 0.19 0.13 | |
| | | | | 28 56 | | 0.13 | |
| | | | | 112 | | 0.06 | |
| | Foliar | 2×20 | Not stated | 0 | Pulp | 0.03 | Study: 1997/11299 |
| | spray | | | 7 | | 0.03 | Trial: F/96/801/ZA9/052 |
| | - | | | 14 | | 0.02 | Treatment 2 |
| | | | | 28 | | <0.01 | Viljoen & Zyl, 1997b, |
| | | | | 56 | | < 0.01 | Kresoxim_067 |
| | | | | 112 | D. I | <0.01 | Max. frozen storage: 15 |
| | | | | 0 7 | Peel | 2.20 2.05 | month |
| | | | | 14 | | 1.65 | |
| | | | | 28 | | 1.10 | |
| | | | | 56 | | 0.85 | |
| | | | | 112 | | 0.93 | |
| | | | | 0 | Whole | 0.82 | |
| | | | | 7 | fruit | 0.68 | |
| | | | | 14 | | 0.55 | |
| | | | | 28 | | 0.34 | |
| | | | | 56 | | 0.26 | |
| | | | | 112 | | 0.23 | |

| Location, | Treatment method | Application rate (g ai/hL) | Growth stage at final | DALT | Crop part | Residue Found (mg/kg) | Report/Trial No., Reference |
|-------------------------------------|---------------------|------------------------------------|-----------------------------|---------------------------------|----------------|--|---|
| Year (variety) | | (g al/nL) | application | | | Kresoxim-methyl ^a | Storage period |
| | Foliar spray | 2×20 plus citrex mineral oil | Not stated | 0 7 14 28 56 112 | Pulp | 0.03 0.02 0.01 <0.01 <0.01 <0.01 <0.01 | Study: 1997/11299 Trial: F/96/801/ZA9/052 Treatment 3 Viljoen & Zyl, 1997b, Kresoxim_067 Max. frozen storage: 15 |
| | | | | 0 7 14 28 56 112 | Peel | 2.95 3.10 2.10 1.75 1.05 1.10 | month |
| | | | | 0 7 14 28 56 112 | Whole fruit | 1.1 1.1 0.80 0.57 0.32 0.30 | |
| South Africa Malelane 1997/98 | Foliar spray | 2×10 | Not stated | 0 84 0 | Pulp Peel | 0.01 <0.01 1.80 | Study: 1999/10181 Treatment 2 Viljoen & Zyl, 1999, |
| (Valencia) | | | | 84 0 84 | Whole fruit | 0.45 0.69 0.21 | Kresoxim_068 Max. frozen storage: 12 month |
| | Foliar spray | 2×20 | Not stated | 0 84 0 | Pulp Peel | 0.24 0.03 3.20 | Study: 1999/10181 Treatment 3 Viljoen & Zyl, 1999, |
| | | | | 84 0 84 | Whole fruit | 0.74 1.40 0.35 | Kresoxim_068 Max. frozen storage: 12 month |
| | Foliar spray | 1×10 | Not stated | 0 28 56 105 | Pulp | 0.28 0.05 0.04 <0.01 | Study: 1999/10181 Treatment 4 Viljoen & Zyl, 1999, Kresoxim_068 |
| | | | | 0 28 56 105 | Peel | 3.60 0.74 0.43 0.32 | Max. frozen storage: 12 month |
| | | | | 0 28 56 105 | Whole fruit | 2.2 0.39 0.21 0.15 | |
| | Foliar spray | 1×20 | Not stated | 0 28 56 105 0 | Pulp | 0.76 0.04 0.04 <0.01 3.60 | Study: 1999/10181 Treatment 6 Viljoen & Zyl, 1999, Kresoxim_068 Max. frozen storage: 12 |
| | | | | 28 56 105 0 | | 0.91 0.61 0.40 | month |
| | | | | 0 28 56 105 | Whole fruit | 2.0 0.43 0.26 0.14 | |
| | Foliar spray | 1×10 | Not stated | 0 27 48 90 | Pulp | 0.11 <0.01 <0.01 <0.01 | Study: 1999/10181 Treatment 8 Viljoen & Zyl, 1999, Kresoxim_068 |

| Location, | Treatment method | Application rate | Growth stage at | DALT | Crop part | Residue Found (mg/kg) | Report/Trial No., |
|-------------------------------|---------------------|------------------|-------------------|-----------|--------------|------------------------------|--------------------------------------|
| Year (variety) | inotitou | (g ai/hL) | final application | | part | Kresoxim-methyl ^a | Reference Storage period |
| | | | | 0 | Peel | 1.60 | Max. frozen storage: 12 |
| | | | | 27 48 | | <0.01 <0.01 | month |
| | | | | 48 90 | | <0.01 | |
| | | | | 0 | Whole | 0.74 | |
| | | | | 27 | fruit | <0.01 | |
| | | | | 48 | | <0.01 | |
| | | | | 90 | | <0.01 | |
| | Foliar | 1×20 | Not stated | 0 | Pulp | 0.18 | Study: 1999/10181 |
| | spray | | | 27 | | < 0.01 | Treatment 9 |
| | | | | 48 90 | | <0.01 <0.01 | Viljoen & Zyl, 1999, Kresoxim_068 |
| | | | | 0 | Peel | 2.70 | Max. frozen storage: 12 |
| | | | | 27 | | <0.01 | month |
| | | | | 48 | | <0.01 | |
| | | | | 90 | | <0.01 | |
| | | | | 0 | Whole | 1.1 | |
| | | | | 27 | fruit | < 0.01 | |
| | | | | 48 90 | | <0.01 <0.01 | |
| South Africa | Foliar | 2×10 | Not stated | 90 54 | Pulp | <0.01 | Study: 1998/11401 |
| Letsitele | spray | 2~10 | Not stated | 85 | rup | <0.01 | Trial: SAF-98-00824 |
| 1997/98 | | | | 54 | Peel | 0.67 | Treatment 2 |
| (Valencia) | | | | 85 | | 0.65 | Viljoen & Zyl, 1998, |
| | | | | 0 | Whole | 0.57 | Kresoxim_069 |
| | | | | 28 | fruit | 0.26 | Max. frozen storage: 7 |
| | | | | 54 85 | | 0.19 | month |
| | Foliar | 1×10 | Not stated | 80 118 | Pulp | 0.16 <0.01 | Study: 1998/11401 |
| | spray | 1~10 | Not stated | 118 | Peel | 0.33 | Trial: SAF-98-00824 |
| | -17 | | | 24 | Whole | 0.41 | Treatment 3 |
| | | | | 64 | fruit | 0.25 | Viljoen & Zyl, 1998, |
| | | | | 92 | | 0.08 | Kresoxim_069 |
| | | | | 118 | | 0.11 | Max. frozen storage: 7 |
| | Foliar | 1×10 | Not stated | 54 | Pulp | <0.01 | month Study: 1998/11401 |
| | spray | 1×10 | Not stated | 54 | Peel | 0.45 | Trial: SAF-98-00824 |
| | opray | | | 0 | Whole | 0.60 | Treatment 5 |
| | | | | 28 | fruit | 0.15 | Viljoen & Zyl, 1998, |
| | | | | 54 | | 0.13 | Kresoxim_069 |
| | | | | | | | Max. frozen storage: 7 |
| | Foliar | 1×20 | Not stated | 92 | Pulp | <0.01 | month Study: 1998/11401 |
| | spray | 1^20 | NOT STATED | 92 118 | i uip | <0.01 | Trial: SAF-98-00824 |
| | | | | 92 | Peel | 0.44 | Treatment 6 |
| | | | | 118 | | 0.54 | Viljoen & Zyl, 1998, |
| | | | | 24 | Whole | 0.89 | Kresoxim_069 |
| | | | | 64 | fruit | 0.28 | Max. frozen storage: 7 |
| | | | | 92 118 | | 0.17 0.20 | month |
| | Foliar | 1×20 | Not stated | 54 | Pulp | <0.01 | Study: 1998/11401 |
| | spray | 1^20 | NUT STATED | 54 54 | Peel | 1.1 | Trial: SAF-98-00824 |
| | | | | 0 | Whole | 1.3 | Treatment 8 |
| | | | | 28 | fruit | 0.60 | Viljoen & Zyl, 1998, |
| | | | | 54 | | 0.34 | Kresoxim_069 |
| | | | | | | | Max. frozen storage: 7 |
| ^a Mean of duplicat | <u> </u> | | | | <u> </u> | | month |

^a Mean of duplicate analysis.

Grapefruit

Field trials were conducted on grapefruit in South Africa during the 1997/1998 growing season (Viljoen & Zyl, 1998b, Kresoxim_070). Grapefruits received 1 or 2 spray applications of kresoxim-methyl at nominal rates ranging between 10–20 g ai/hL. Samples were collected at various time points after the last application. Residues of kresoxim-methyl were determined using BASF method 351/2 with a limit of quantification at 0.01 mg/kg. Overall mean procedural recoveries for kresoxim-methyl in orange pulp spiked at 0.04–2.0 mg/kg and peel spiked at 0.04–2.0 mg/kg were 100% (n=4) and 96% (n=4), respectively

Table 81 Residues of kresoxim-methyl in grapefruit following foliar treatment (cGAP Turkey: 4×12.5 g ai/hL; 35 day PHI)

| | Treatment | Application | Growth | DALT | Crop | Residue Found | |
|----------------|-----------|-------------|-------------|-----------|--------------|------------------------------|--|
| Location, | method | rate | stage at | | part | (mg/kg) | Report/Trial No., |
| Veer(veriety) | | (g ai/hL) | final | | | Kresoxim-methyl ^a | Reference |
| Year (variety) | | | application | | | - | Storage period |
| South Africa | Foliar | 2×10 | Not stated | 0 | Pulp | 0.01 | Study: 1998/11303 |
| Letsitele | spray | | | 14 | | <0.01 | Trial: SAF-98-00823 |
| 1997/98 | | | | 0 | Peel | 1.1 | Treatment 2 |
| (Marsh) | | | | 7 | | 0.60 | Viljoen & Zyl, 1998b, |
| | | | | 14 | | 0.67 | Kresoxim_070 |
| | | | | 28 | | 0.52 | Max. frozen storage: 3 |
| | | | | 56 | | 0.42 | month |
| | | | | 0 | Whole | 0.45 | |
| | | | | 7 | fruit | 0.23 | |
| | | | | 14 | | 0.22 | |
| | | | | 28 | | 0.18 0.14 | |
| | Foliar | 1×10 | Not stated | 56 64 | Dulp | <0.01 | Study: 1000/11202 |
| | | 1×10 | NUL STATED | 64 64 | Pulp Peel | 0.27 | Study: 1998/11303 Trial: SAF-98-00823 |
| | spray | | | 64 78 | Peel | 0.27 | Treatment 3 |
| | | | | 82 | | 0.27 | Viljoen & Zyl, 1998b, |
| | | | | 108 | | 0.16 | Kresoxim_070 |
| | | | | 128 | | 0.21 | Max. frozen storage: 3 |
| | | | | 64 | Whole | 0.11 | month |
| | | | | 78 | fruit | 0.10 | |
| | | | | 82 | | 0.09 | |
| | | | | 108 | | 0.06 | |
| | | | | 128 | | 0.07 | |
| | Foliar | 1×10 | Not stated | 0 | Pulp | 0.05 | Study: 1998/11303 |
| | spray | | | 14 | | <0.01 | Trial: SAF-98-00823 |
| | | | | 0 | Peel | 0.61 | Treatment 5 |
| | | | | 14 | | 0.21 | Viljoen & Zyl, 1998b, |
| | | | | 28 | | 0.17 | Kresoxim_070 |
| | | | | 44 | | 0.18 | Max. frozen storage: 3 |
| | | | | 63 | | 0.20 | month |
| | | | | 0 | Whole | 0.30 | |
| | | | | 14 | fruit | 0.08 | |
| | | | | 28 | | 0.06 | |
| | | | | 44 | | 0.06 | |
| | | 4.00 | | 63 | D.I. | 0.06 | 01 1 4000/44000 |
| | Foliar | 1×20 | Not stated | 64 70 | Pulp | <0.01 | Study: 1998/11303 |
| | spray | | | 78 92 | | <0.01 | Trial: SAF-98-00823 Treatment 6 |
| | | | | 92 108 | | <0.01 <0.01 | Viljoen & Zyl, 1998b, |
| | | | | 128 | | <0.01 | Kresoxim_070 |
| | | | | 64 | Peel | 0.39 | Max. frozen storage: 3 |
| | | | | 78 | 1 661 | 0.39 | month |
| | | | | 92 | | 0.34 | |
| | | | | 108 | | 0.41 | |
| | | | | 128 | | 0.31 | |
| | | | | 64 | Whole | 0.16 | t |
| | | | | 78 | fruit | 0.15 | |
| | | | | 92 | | 0.11 | |
| | | | | 108 | | 0.12 | |
| | | | | 128 | | 0.09 | |

| Location, Year (variety) | Treatment method | Application rate (g ai/hL) | Growth stage at final application | DALT | Crop part | Residue Found (mg/kg) Kresoxim-methylª | Report/Trial No., Reference Storage period |
|-----------------------------|---------------------|----------------------------------|--|--|----------------|---|---|
| | Foliar spray | 1×20 | Not stated | 0 14 28 44 63 0 14 28 44 63 | Pulp Peel | 0.11 1.60 0.89 <0.01 <0.01 1.5 0.04 0.03 0.58 0.67 | Study: 1998/11303 Trial: SAF-98-00823 Treatment 8 Viljoen & Zyl, 1998b, Kresoxim_070 Max. frozen storage: 3 month |
| | | | | 0 14 28 44 63 | Whole fruit | 0.59 0.60 0.34 0.17 0.18 | |

^a Mean of duplicate analysis.

Natsumikan (Citrus natsudaidai)

Field trials were conducted on Natsumikan in Japan during the 1994 growing season (Komatsu & Yabusaki, 1994, Kresoxim_071; Gomyo, 1994, Kresoxim_072). Plants received three spray applications of kresoxim-methyl at nominal rates of 25 g ai/hL. Samples were collected at 14/15, 28 and 45 days after the last application. Residues of kresoxim-methyl were determined using the same method, or a modified method of Saku 6P-7-175. Overall mean procedural recoveries for kresoxim-methyl in Natsumikan spiked at 0.2 mg/kg ranged between 90–95%.

Table 82 Residues of kresoxim-methyl in Natsumikan following foliar treatment (cGAP Japan: 3x25 g ai/hL; 14 day PHI)

| Location, Year (variety) | Treatment method | Application rate (g ai/hL) | Growth stage at final application | DALT | Crop part | Residue Found (mg/kg) Kresoxim-methyl ^a | Report/Trial No., Reference Storage period |
|---|---------------------|----------------------------------|-----------------------------------|----------------|----------------|--|---|
| Japan Chiba Prefectural 1994 (Kawano natsudaidai) | Foliar spray | 3×25 | Not stated | 15 28 45 | Whole fruit | 0.59 0.55 0.60 | Study: 1994/1006429 Trial: 1 Komatsu & Yabusaki, 1994, Kresoxim_071 Max. frozen storage: 6 month |
| | Foliar spray | 3×25 | Not stated | 15 28 45 | Whole fruit | 0.92 0.77 0.87 | Study: 1994/1006431 Trial: 1 Gomyo, 1994, Kresoxim_072 Max. frozen storage: 2 month |
| Japan Oita Prefectural 1994 (Amanatsu) | Foliar spray | 3×25 | Not stated | 14 28 45 | Whole fruit | 1.5 0.83 0.53 | Study: 1994/1006429 Trial: 2 Komatsu & Yabusaki, 1994, Kresoxim_071 Max. frozen storage: 6 month |
| | Foliar spray | 3×25 | Not stated | 14 28 45 | Whole fruit | 1.8 1.2 1.0 | Study: 1994/1006431 Trial: 2 Gomyo, 1994, Kresoxim_072 Max. frozen storage: 2 month |

^a Mean of duplicate analysis.

As two trials were conducted in at the same location and time, using the same variety, they were not considered as independent. Hence, here only the higher residues of these trials were considered.

Kabosu (Citrus sphaerocarpa)

Field trials were conducted on Kabosu in Japan during the 1994 growing season (Tokieda & Gomyo, 1994, Kresoxim_073). Plants received three spray applications of kresoxim-methyl at nominal rates of 25 g ai/hL. Samples were collected at 14, 30/31 and 45 days after the last application. Residues of kresoxim-methyl were determined using the same method, or a modified method of Saku 6P-7-175. Overall mean procedural recoveries for kresoxim-methyl in Kabosu spiked at 0.1 mg/kg were at 87%.

Table 83 Residues of kresoxim-methyl in Kabosu following foliar treatment (cGAP Japan: 3×25 g ai/hL; 14 day PHI)

| Location, Year (variety) | Treatment method | Application rate (g ai/hL) | Growth stage at final application | DALT | Crop part | Residue Found (mg/kg) Kresoxim-methyl ^a | Report/Trial No., Reference Storage period |
|--|---------------------|----------------------------------|--|----------------|----------------|--|--|
| Japan Hiroshima Prefectural 1994 (local variety) | Foliar spray | 3×25 | Not stated | 14 31 45 | Whole fruit | 4.6 4.2 4.0 | Study: 1994/1006435 Trial: 1 Tokieda & Gomyo, 1994, Kresoxim_073 Max. frozen storage: 1 month |
| Japan Oita Prefectural 1994 (local variety) | Foliar spray | 3×25 | Not stated | 14 30 45 | Whole fruit | 1.2 1.5 0.84 | Study: 1994/1006435 Trial: 2 Tokieda & Gomyo, 1994, Kresoxim_073 Max. frozen storage: 1 month |

^a Mean of duplicate analysis.

Sudachi (Citrus sudachi)

One field trial was conducted on Sudachi in Japan during the 2004 growing season (Kobayashi & Tokieda, 2005, Kresoxim_074). Plants received three spray applications of kresoxim-methyl at nominal rates of 25 g ai/hL. Samples were collected at 14, 30 and 45 days after the last application. Residues of kresoxim-methyl were determined using the same method, or a modified method of Saku 6P-7-175. Overall mean procedural recoveries for kresoxim-methyl in Sudachi spiked at 0.1 mg/kg and 2.0 mg/kg were at 94% and 98% respectively.

Table 84 Residues of kresoxim-methyl in Sudachi following foliar treatment (cGAP Japan: 3×25 g ai/hL; 14 day PHI)

| Location, Year (variety) | Treatment method | Application rate (g ai/hL) | Growth stage at final application | DALT | Crop part | Residue Found (mg/kg) Kresoxim-methylª | Report/Trial No., Reference Storage period |
|---|---------------------|----------------------------------|--|----------------|----------------|--|--|
| Japan Tokushima 2004 (local variety) | Foliar spray | 3×25 | Not stated | 14 30 45 | Pulp + peel | <0.1 <0.1 <0.1 | Study: 2005/1045480 Trial: 1 Kobayashi & Tokieda, 2005, Kresoxim_074 Max. frozen storage: 4 month |

^a Mean of duplicate analysis.

Pome fruit

Apple

Four field trials were conducted with apple in Germany in the 2010 growing season (Plier, 2011, Kresoxim_075). Apples received 4 spray applications of kresoxim-methyl at nominal rates of 100 g ai/hL (2×500 g ai/ha) followed by 208 g ai/hL (2×650 g ai/ha) with a 7 day interval between applications. Samples of apples were collected at 0 and 35±1 days after the last application. Residues of kresoxim-methyl (as 490M1) and metabolites 490M2 and 490M9 were determined using method BASF method 350/4 with a limit of quantification at 0.01 mg/kg. Overall mean procedural recoveries for kresoxim-methyl (as 490M1) and metabolites 490M2 and 490M9 in apple spiked at 0.01–10 mg/kg were 89% (n=30), 87% (n=30) and 92% (n=30), respectively.

| Location, | Treatment | Application | Growth | DALT | Crop | Residue Fou | und (mg/kg) | | | Report/Trial |
|--|-----------------|-------------------|----------------------------------|---------|-------|---------------------|----------------|--------------|----------------------------|--|
| Year (variety) | method | rate (g ai/hL) | stage at final application | | part | Kresoxim- methyl | 490M2 | 490M9 | Sum as parent equiv. | No., Reference Storage period |
| Germany Pirna 2010 (Jonica) | Foliar spray | 2×100 2×208 | BBCH 81 | 0 34 | Fruit | 1.30 0.10 | <0.01 <0.01 | 0.01 0.01 | 1.32 0.12 | Study: 2011/1109264 Trial: L100273 Plier, 2011, Kresoxim_075 Max. frozen storage: 5 month |
| Germany Sornzig 2010 (Gala) | Foliar spray | 2×100 2×208 | BBCH 77 | 0 35 | Fruit | 0.94 0.07 | <0.01 0.01 | 0.01 0.02 | 0.96 0.10 | Study: 2011/1109264 Trial: L100274 Plier, 2011, Kresoxim_075 Max. frozen storage: 6 month |
| Germany Leisnig 2010 (Jonagored) | Foliar spray | 2×100 2×208 | BBCH 81 | 0 35 | Fruit | 0.89 0.12 | <0.01 <0.01 | 0.01 | 0.91 0.14 | Study: 2011/1109264 Trial: L100275 Plier, 2011, Kresoxim_075 Max. frozen storage: 5 month |
| Germany Werneuchten 2010 (Jonica) | Foliar spray | 2×100 2×208 | BBCH 78 | 0 35 | Fruit | 0.88 0.36 | 0.01 0.02 | 0.02 0.04 | 0.91 0.42 | Study: 2011/1109264 Trial: L100276 Plier, 2011, Kresoxim_075 Max. frozen storage: 5 month |

Table 85 Residues of kresoxim-methyl (as 490M1) and metabolites 490M2 and 490M9 in apple following foliar treatment

Four field trials were conducted with apple in Germany (two trials) and France (two trials) in the 1997 growing season (Meumann & Schulz, 1998, Kresoxim_076). Apples received 12 spray applications of kresoxim-methyl at nominal rates of 10×102 g ai/ha followed by 2×128 g ai/ha. Samples of apples were collected at 0, 21/22, 28, 34/35 and 40/42 days after the last application. Residues of kresoxim-methyl were determined using method BASF method 351/1 with a limit of quantification at 0.05 mg/kg. Overall mean procedural recoveries for kresoxim-methyl in apple spiked at 0.05 and 5 mg/kg were 85% (n=5).

One field trial was conducted with apple in Poland in the 1997 growing season (Nowacka, 1997, Kresoxim_077). Apples received 8 spray applications of kresoxim-methyl at nominal rates of 200 g ai/ha with a 7 day interval between applications. Samples of apples were collected at 1, 27, 33 and 40 days after the last application. Residues of kresoxim-methyl were determined using method BASF method 351/1 with a limit of quantification at 0.05 mg/kg. Overall mean procedural recoveries for kresoxim-methyl in apple spiked at 0.05 and 0.5 mg/kg were 108% (n=6).

One field trial was conducted with apple in Hungary in the 1994 growing season (Korsos, 1994, Kresoxim_078). Apples received 14 spray applications of kresoxim-methyl at nominal rates of 400 g ai/ha with a 7-11 day interval between applications. Samples of apples were collected at 0, 3, 7, 10, 14, 21, 28 and 33 days after the last application. Residues of kresoxim-methyl were determined using method BASF method 351/1 with a limit of quantification at 0.05 mg/kg. Overall mean procedural recoveries for kresoxim-methyl in apple spiked at 0.04 and 0.2 mg/kg were 63% and 67%, respectively.

Three field trials were conducted with apple in South Africa in the 1994/1995 growing season (Viljoen, 1995a, Kresoxim_079; Viljoen, 1995b, Kresoxim_080; Viljoen & Zyl, 1996, Kresoxim_081). Apples received 10–12 spray applications of kresoxim-methyl at nominal rates of 125 or 250 g ai/ha. Samples of apples were collected at 0, 12, 24, 35 and 35 days after the last application. Residues of kresoxim-methyl were determined using method BASF method 351/2 with a limit of quantification at 0.05 mg/kg. Overall mean procedural recoveries for kresoxim-methyl in apple spiked at 0.08–4.0 mg/kg were 97% (n=7).

| | r | . | [| | 1 | r | |
|----------------------------|---------------|-------------|-------------|------|-------|-----------|------------------------------|
| | Treatment | Application | Growth | DALT | Crop | Residue | |
| Location, | method | rate | stage at | | part | Found | Report/Trial No., Reference |
| | | (g ai/ha) | final | | | (mg/kg) | Storage period |
| Year (variety) | | | application | | | Kresoxim- | Storage period |
| | | | | | | methyl | |
| Germany | Foliar spray | 10×102 | BBCH 89 | 0 | Fruit | 0.23 | Study: 1998/10187 |
| Nordrhein-Westfalen | | 2×128 | | 21 | | 0.13 | Trial: AGR/16/97 |
| 1997 | | | | 28 | | 0.09 | Meumann & Schulz, 1998, |
| (Elstar) | | | | 35 | | 0.07 | Kresoxim_076 |
| | | | | 42 | | 0.07 | Max. frozen storage: 3 month |
| Germany | Foliar spray | 10×102 | BBCH 87/88 | 0 | Fruit | 0.18 | Study: 1998/10187 |
| Schleswig-Holstein | 1 5 | 2×128 | | 21 | | < 0.05 | Trial: D05/04/97 |
| 1997 | | | | 28 | | < 0.05 | Meumann & Schulz, 1998, |
| (Jonica) | | | | 35 | | < 0.05 | Kresoxim_076 |
| | | | | 42 | | < 0.05 | Max. frozen storage: 3 month |
| France | Foliar spray | 10×102 | BBCH 89 | 0 | Fruit | 0.22 | Study: 1998/10187 |
| Gard | | 2×128 | | 21 | | 0.07 | Trial: FR3/02/97 |
| 1997 | | | | 28 | | 0.08 | Meumann & Schulz, 1998, |
| (Golden Delicious) | | | | 35 | | 0.07 | Kresoxim_076 |
| (control policiouc) | | | | 42 | | < 0.05 | Max. frozen storage: 3 month |
| France | Foliar spray | 10×102 | BBCH 87 | 0 | Fruit | 0.28 | Study: 1998/10187 |
| Haute Garonne | , oner spruy | 2×128 | 5501107 | 21 | | 0.12 | Trial: FR8/04/97 |
| 1997 | | 20120 | | 28 | | 0.12 | Meumann & Schulz, 1998, |
| (Golden Delicious) | | | | 35 | | 0.08 | Kresoxim_076 |
| (oolden benelous) | | | | 42 | | < 0.05 | Max. frozen storage: 3 month |
| Poland | Foliar spray | 8×200 | Not stated | 1 | Fruit | 0.15 | Study: 1997/11558 |
| Skierniewice | i ullai spray | 0~200 | NUL SIALEU | 27 | Truit | < 0.05 | Nowacka, 1997, Kresoxim_077 |
| 1997 | | | | 33 | | < 0.05 | Max. frozen storage: not |
| (Jereymac) | | | | 40 | | < 0.05 | stated |
| Hungary | Foliar spray | 14×400 | Not stated | 0 | Fruit | 0.09 | Study: 1994/11727 |
| Nagykálló | Fullal Splay | 14×400 | NUL SIALEU | 3 | FIUIL | 0.09 | Korsos, 1994, Kresoxim_078 |
| 1997 | | | | 7 | | 0.07 | Max. frozen storage: 4 month |
| (Jonathane) | | | | 10 | | 0.07 | Max. Hozen storage: 4 month |
| (Juliatilalie) | | | | 14 | | 0.05 | |
| | | | | 21 | | 0.03 | |
| | | | | 21 | | 0.04 | |
| | | | | 33 | | 0.02 | |
| South Africa | Foliar spray | 12×125 | Not stated | 0 | Fruit | 0.01 | Study: 1995/10948 |
| | Fullal Splay | 12×125 | NUL SIALEU | 12 | FIUIL | 0.27 | Viljoen, 1995a, Kresoxim_079 |
| Villiersdorp 1995 | | | | 24 | | | Max. frozen storage: not |
| | | | | | | 0.13 | Ű |
| (Grany Smith) | | | | 35 | | 0.09 | stated |
| | | 10.050 | | 42 | E 11 | n.d. | - |
| | Foliar spray | 12×250 | Not stated | 0 | Fruit | 0.63 | |
| | | | | 12 | | 0.40 | 1 |
| | | | | 24 | | 0.18 | |
| | | | | 35 | | 0.18 | |
| Couth Afric - | Falles | 10 105 | Netetat | 42 | En 14 | 0.09 | Churchus 1005/10040 |
| South Africa | Foliar spray | 12×125 | Not stated | 0 | Fruit | 0.15 | Study: 1995/10949 |
| Vyeboom | | | | 12 | | 0.13 | Viljoen, 1995b, Kresoxim_080 |
| 1995 (Calden Daliaiaua) | | | | 24 | | n.d. | Max. frozen storage: not |
| (Golden Delicious) | | | | 35 | | n.d. | stated |
| | | 40.075 | | 42 | | n.d. | 4 |
| | Foliar spray | 12×250 | Not stated | 0 | Fruit | 0.38 | |
| | | | | 12 | | 0.19 | 1 |
| | | | | 24 | | 0.11 | |
| | | | | 35 | | 0.14 | |
| | | | | 42 | + | 0.06 | |
| South Africa | Foliar spray | 12×125 | Not stated | 0 | Fruit | 0.21 | Study: 1996/10831 |
| Tiefkasfontein, | | | | 12 | | 0.09 | Viljoen & Zyl, 1996, |
| Villiersdorp | | | | 24 | | 0.04 | Kresoxim_081 |
| 1995 | | | | 35 | | 0.02 | Max. frozen storage: not |

Table 86 Residues of kresoxim-methyl in apple following foliar treatment

| Location, Year (variety) | Treatment method | Application rate (g ai/ha) | Growth stage at final application | DALT | Crop part | Residue Found (mg/kg) Kresoxim- methyl | Report/Trial No., Reference Storage period |
|-----------------------------|---------------------|----------------------------------|--|---------------------|--------------|--|---|
| (Top Red) | Foliar spray | 12×250 | Not stated | 0 12 24 35 | Fruit | 0.48 0.31 0.13 0.11 | stated |

^a Mean of duplicate analysis.

Stone fruit

Peach

A total of eight field trials were conducted with peaches in Spain in the 1997 (Meumann, 1999a, Kresoxim_082) and 1998 (Meumann, 1999b, Kresoxim_083) growing season. Peaches received 4 spray applications of kresoxim-methyl at nominal rates of 20 g ai/hL (200 g ai/ha) with a 10 day interval between applications. Samples of peaches were collected at 0, 3/4, 9/11 and 14/15 days after the last application. Residues of kresoxim-methyl (as 490M1) and metabolites 490M2 and 490M9 were determined using method BASF method 350/3 with a limit of quantification of 0.05 mg/kg. Overall mean procedural recoveries for kresoxim-methyl (as 490M1) and metabolites 490M2 and 490M2 and 490M9 in peaches spiked at 0.05–5 mg/kg were 93% (n=14), 96% (n=14) and 95% (n=14), respectively.

Two field trials were conducted with peaches in France in the 1999 (Meumann & Benz, 2000, Kresoxim_084) growing season. Peaches received 4 spray applications of kresoxim-methyl at nominal rates of 30 g ai/hL (150 g ai/ha) with an 8-14 day interval between applications. Samples of peaches were collected at 0, 3, 7 and 14 days after the last application. Residues of kresoxim-methyl (as 490M1) and metabolites 490M2 and 490M9 were determined using method BASF method 350/3 with a limit of quantification of 0.05 mg/kg. Overall mean procedural recoveries for kresoxim-methyl (as 490M1) and metabolites 490M2 and 490M9 in peaches spiked at 0.05–5 mg/kg were 97% (n=4), 96% (n=4) and 93% (n=4), respectively.

| Table 87 Residues of kresoxim-methyl (as 490M1) and metabolites 490M2 and 490M9 in peaches following foliar treatment (cGAI | 2 |
|---|---|
| Japan: 3×25 g ai/hL; 1 day PHI) | |

| Location, | Treatment | Application | Growth | DALT | Crop | Residue Fou | nd (mg/kg) | | | Deport/Trial |
|--|-----------------|-------------------|----------------------------------|--------------------|-------|--------------------------------|----------------------------------|------------------------------|------------------------------|--|
| Year (variety) | method | rate (g ai/hL) | stage at final application | | part | Kresoxim- methyl | 490M2 | 490M9 | Sum of parent + 490M9 | Report/Trial No., Reference Storage period |
| Spain Huelva 1997 (Spring crest) | Foliar spray | 4×20 | BBCH 87/88 | 0 4 11 15 | Fruit | 0.41 0.38 0.14 0.19 | <0.05 <0.05 <0.05 <0.05 | 0.10 0.09 0.11 0.16 | 0.51 0.47 0.25 0.35 | Study: 1999/10097 Trial: ALO/01/97 Meumann, 1999a, Kresoxim_082 Max. frozen storage: 14 month |
| Spain Sevilla 1997 (Katerina) | Foliar spray | 4×20 | BBCH 87 | 0 4 10 15 | Fruit | 0.21 0.09 <0.05 <0.05 | <0.05 <0.05 <0.05 <0.05 | 0.06 0.05 0.07 0.10 | 0.27 0.14 0.12 0.15 | Study: 1999/10097 Trial: ALO/01/97 Meumann, 1999a, Kresoxim_082 Max. frozen storage: 14 month |

| Location, | Treatment | Application | Growth | DALT | Crop | Residue Fou | nd (ma/ka) | | | |
|---|-----------------|-------------------|----------------------------------|--------------------|-------|--------------------------------|----------------------------------|----------------------------------|--------------------------------|--|
| Year (variety) | method | rate (g ai/hL) | stage at final application | Driz I | part | Kresoxim- methyl | 490M2 | 490M9 | Sum of parent + 490M9 | Report/Trial No., Reference Storage period |
| Spain Huelva 1997 (May crest) | Foliar spray | 4×20 | BBCH 88/89 | 0 4 11 15 | Fruit | 0.17 0.34 0.06 0.11 | <0.05 <0.05 <0.05 <0.05 | 0.07 0.07 0.06 0.11 | 0.24 0.41 0.12 0.22 | Study: 1999/10097 Trial: ALO/01/97 Meumann, 1999a, Kresoxim_082 Max. frozen storage: 14 month |
| Spain Sevilla 1997 (Spring crest) | Foliar spray | 4×20 | BBCH 88/89 | 0 4 10 14 | Fruit | 0.34 0.24 0.09 <0.05 | <0.05 <0.05 <0.05 <0.05 | 0.08 0.07 0.07 <0.05 | 0.42 0.31 0.16 <0.10 | Study: 1999/10097 Trial: ALO/01/97 Meumann, 1999a, Kresoxim_082 Max. frozen storage: 14 month |
| Spain Huelva 1998 (May crest) | Foliar spray | 4×20 | BBCH 78- 82 | 0 5 11 14 | Fruit | 0.59 0.17 0.15 0.06 | <0.05 <0.05 <0.05 <0.05 | 0.07 0.09 0.09 0.06 | 0.66 0.26 0.24 0.12 | Study: 1999/10107 Trial: ALO/14/98 Meumann, 1999b, Kresoxim_083 Max. frozen storage: 6 month |
| Spain Huelva 1998 (Spring crest) | Foliar spray | 4×20 | BBCH 79- 82 | 0 5 11 14 | Fruit | 0.66 0.15 0.11 0.05 | <0.05 <0.05 <0.05 <0.05 | 0.09 0.08 0.09 0.08 | 0.75 0.23 0.20 0.13 | Study: 1999/10107 Trial: ALO/15/98 Meumann, 1999b, Kresoxim_083 Max. frozen storage: 6 month |
| Spain Sevilla 1998 (Babygold 6) | Foliar spray | 4×20 | BBCH 80- 85 | 0 4 9 14 | Fruit | 0.08 0.13 <0.05 <0.05 | <0.05 <0.05 <0.05 <0.05 | <0.05 <0.05 <0.05 <0.05 | 0.13 0.18 <0.10 <0.10 | Study: 1999/10107 Trial: ALO/16/98 Meumann, 1999b, Kresoxim_083 Max. frozen storage: 5 month |
| Spain Sevilla 1998 (Babygold 6) | Foliar spray | 4×20 | BBCH 85- 89 | 0 5 9 15 | Fruit | 0.07 0.08 <0.05 <0.05 | <0.05 <0.05 <0.05 <0.05 | <0.05 <0.05 <0.05 0.06 | 0.12 0.13 <0.10 0.11 | Study: 1999/10107 Trial: ALO/17/98 Meumann, 1999b, Kresoxim_083 Max. frozen storage: 5 month |

| Location, | Treatment | Application | Growth | DALT | Crop | Residue Fou | nd (mg/kg) | | | Dement/Trial |
|-------------------|-----------|-------------|----------------------|------|-------|-------------|------------|-------|-------------------|--------------------------------|
| | method | rate | stage at | | part | Kresoxim- | 490M2 | 490M9 | Sum of | Report/Trial No., Reference |
| Year (variety) | | (g ai/hL) | final application | | | methyl | | | parent + 490M9 | Storage period |
| France | Foliar | 4×30 | BBCH 87 | 0 | Fruit | 0.25 | <0.05 | 0.065 | 0.32 | Study: |
| Gard | spray | | | 3 | | 0.16 | <0.05 | 0.073 | 0.23 | 2000/1000241 |
| 1999 | | | | 7 | | 0.11 | <0.05 | 0.099 | 0.21 | Trial: |
| (Elegant | | | | 14 | | 0.07 | <0.05 | 0.12 | 0.19 | FR3/01/99 |
| lady) | | | | | | | | | | Meumann & |
| | | | | | | | | | | Benz, 2000, |
| | | | | | | | | | | Kresoxim_084 |
| | | | | | | | | | | Max. frozen |
| | | | | | | | | | | storage: 6 |
| | | | | | | | | | | month |
| France | Foliar | 4×30 | BBCH 89 | 0 | Fruit | 0.14 | <0.05 | 0.064 | 0.20 | Study: |
| Haute | spray | | | 3 | | 0.092 | <0.05 | 0.11 | 0.20 | 2000/1000241 |
| Garonne | | | | 7 | | 0.061 | <0.05 | 0.13 | 0.19 | Trial: |
| 1999 | | | | 14 | | <0.05 | <0.05 | 0.13 | 0.18 | FR8/01/99 |
| (GF-305) | | | | | | | | | | Meumann & |
| | | | | | | | | | | Benz, 2000, |
| | | | | | | | | | | Kresoxim_084 |
| | | | | | | | | | | Max. frozen |
| | | | | | | | | | | storage: 5 |
| | | | | | | | | | | month |

A total of nine field trials were conducted with peaches in Spain during the 2001 (Beck, 2002, Kresoxim_085) and 2000 (Schroth, 2001, Kresoxim_086) growing seasons. Peaches received 3 spray applications of kresoxim-methyl at nominal rates of 7.5 g ai/ha) with a 10 day interval between applications. Samples of peaches were collected at 0, 7/8, 13/14/15 and 20/21/22 days after the last application. Residues of kresoxim-methyl were determined using method BASF method 445/0 with a limit of quantification of 0.05 mg/kg. Overall mean procedural recoveries for kresoxim-methyl in peaches spiked at 0.05–5 mg/kg were 82% (n=6).

| Table 88 Residues of kresoxim-meth | yl in | peaches following | g foliar treatment (| (cGAP Peru: 2×150 | g ai/ha; 30 da | y PHI) |
|------------------------------------|-------|-------------------|----------------------|-------------------|----------------|--------|
| | | | | | | |

| Location, | Treatment | Application | Growth | DALT | Crop | Residue Foun | d (mg/kg) | _ | Report/Trial No., |
|-------------|-----------|-------------|-------------|------|-------|--------------|-----------|-------|-------------------|
| | method | rate | stage at | | part | Kresoxim- | 490M2 | 490M9 | Reference |
| Year | | (g ai/hL) | final | | | methyl | | | Storage period |
| (variety) | | | application | | | | | | otorage period |
| Spain | Foliar | 3×7.5 | BBCH 89 | 0 | Fruit | <0.05 | n/a | n/a | Study: |
| Santi Ponce | spray | | | 7 | | <0.05 | | | 2001/1015033 |
| Sevilla | | | | 13 | | <0.05 | | | Trial: AYE/01/01 |
| 2001 | | | | 20 | | <0.05 | | | Beck, 2002, |
| (Hermoine) | | | | | | | | | Kresoxim_085 |
| | | | | | | | | | Max. frozen |
| | | | | | | | | | storage: 7 month |
| Spain | Foliar | 3×7.5 | BBCH 89 | 0 | Fruit | <0.05 | n/a | n/a | Study: |
| Alcala del | spray | | | 7 | | <0.05 | | | 2001/1015033 |
| Rio | | | | 13 | | <0.05 | | | Trial: AYE/02/01 |
| Sevilla | | | | 20 | | <0.05 | | | Beck, 2002, |
| 2001 | | | | | | | | | Kresoxim_085 |
| (A-477) | | | | | | | | | Max. frozen |
| | | | | | | | | | storage: 7 month |
| Spain | Foliar | 3×7.5 | BBCH 89 | 0 | Fruit | <0.05 | n/a | n/a | Study: |
| Cordoba | spray | | | 8 | | <0.05 | | | 2001/1015033 |
| 2001 | | | | 15 | | <0.05 | | | Trial: AYE/03/01 |
| (Loadel) | | | | 22 | | <0.05 | | | Beck, 2002, |
| | | | | | | | | | Kresoxim_085 |
| | | | | | | | | | Max. frozen |
| | | | | | | | | | storage: 6 month |

| Location, | Treatment | Application | Growth | DALT | Crop | Residue Four | d (mg/kg) | | D 1/7 1 1 1 |
|--|-----------------|-------------------|----------------------------------|--------------------|-------|----------------------------------|-----------|-------|---|
| Year (variety) | method | rate (g ai/hL) | stage at final application | | part | Kresoxim- methyl | 490M2 | 490M9 | Report/Trial No., Reference Storage period |
| Spain Murcia 2001 (Catherina) | Foliar spray | 3×7.5 | BBCH 89 | 0 7 14 21 | Fruit | <0.05 <0.05 <0.05 <0.05 | n/a | n/a | Study: 2001/1015033 Trial: ENA/01/01 Beck, 2002, Kresoxim_085 Max. frozen storage: 6 month |
| Spain Valenciana 2001 (Catherina) | Foliar spray | 3×7.5 | BBCH 89 | 0 7 14 21 | Fruit | <0.05 <0.05 <0.05 <0.05 | n/a | n/a | Study: 2001/1015033 Trial: ENA/02/01 Beck, 2002, Kresoxim_085 Max. frozen storage: 6 month |
| Spain Munebrega Zaragoza 2000 (Babygold 6) | Foliar spray | 3×7.5 | BBCH 81 | 0 7 14 22 | Fruit | <0.05 <0.05 <0.05 <0.05 | n/a | n/a | Study: 2001/1009085 Trial: 00S018R Schroth, 2001, Kresoxim_086 Max. frozen storage: 10 month |
| Spain Ateca Zaragoza 2000 (Babygold 6) | Foliar spray | 3×7.5 | BBCH 85 | 0 7 14 22 | Fruit | <0.05 <0.05 <0.05 <0.05 | n/a | n/a | Study: 2001/1009085 Trial: 00S019R Schroth, 2001, Kresoxim_086 Max. frozen storage: 10 month |
| Spain Maluenda Zaragoza 2000 (Babygold 6) | Foliar spray | 3×7.5 | BBCH 85 | 0 7 14 22 | Fruit | <0.05 <0.05 <0.05 <0.05 | n/a | n/a | Study: 2001/1009085 Trial: 00S020R Schroth, 2001, Kresoxim_086 Max. frozen storage: 10 month |
| Spain La Gironda Sevilla 2000 (Babygold 6) | Foliar spray | 3×7.5 | BBCH 89 | 0 7 14 21 | Fruit | <0.05 <0.05 <0.05 <0.05 | n/a | n/a | Study: 2001/1009085 Trial: 00S021R Schroth, 2001, Kresoxim_086 Max. frozen storage: 11 month |

Berries & other small fruits

Black currant

A total of five field trials were conducted with black currant in Denmark, Great Britain, and Sweden in the 1999 (Beck & Benz, 2000, Kresoxim_087) and 1998 (Beck et al, 1999a, Kresoxim_088) growing season. Plants received 3 spray applications of kresoxim-methyl at nominal rates of 100 g ai/ha with a 14 day interval between applications. Samples of black currant were collected at 0, 2–4, 6–8, 13–14 and 21 days after the last application. Residues of kresoxim-methyl (as 490M1) and metabolites 490M2 and 490M9 were determined using method BASF method 350/3 with a limit of quantification of 0.05 mg/kg. Overall mean procedural recoveries for kresoxim-methyl (as 490M1) and metabolites 490M2 and 490M9 in black currant spiked at 0.05–5 mg/kg were 95% (n=18), 91% (n=18) and 90% (n=18), respectively.

| | Treatment | Application | Growth stage | DALT | Crop | Residue Four | nd (mg/kg) | | | |
|---|--------------|-------------------|----------------------|-------------------------|-------|--------------------------------------|---|---|--------------------------------------|--|
| Location, Year (variety) | method | rate (g ai/ha) | at final application | | part | Kresoxim- methyl | 490M2 | 490M9 | Sum of parent + 490M9 | Report/Trial No., Reference Storage period |
| Denmark Middelfart 1999 (Ben Lomond) | Foliar spray | 3×100 | BBCH 92 | 0 2 7 14 21 | Fruit | 0.37 0.18 0.22 0.18 0.09 | <0.05 <0.05 <0.05 <0.05 <0.05 | <0.05 <0.05 <0.05 <0.05 0.05 | 0.42 0.23 0.27 0.23 0.14 | Study: 2000/1000222 Trial: ALB/15/99 Beck & Benz, 2000, Kresoxim_087 Max. frozen storage: 6 month |
| Sweden Bjärred 1999 (Öjeby) | Foliar spray | 3×100 | BBCH 93 | 0 4 7 14 21 | Fruit | 0.39 0.23 0.19 0.13 0.06 | <0.05 <0.05 <0.05 <0.05 <0.05 | <0.05 <0.05 <0.05 <0.05 0.08 | 0.44 0.28 0.24 0.18 0.14 | Study: 2000/1000222 Trial: HUS/09/99 Beck & Benz, 2000, Kresoxim_087 Max. frozen storage: 6 month |
| Great Britain Launton 1999 (Ben Lowman) | Foliar spray | 3×100 | BBCH 89 | 0 4 8 14 21 | Fruit | 0.53 0.58 0.36 0.16 0.13 | <0.05 <0.05 <0.05 <0.05 <0.05 | <0.05 <0.05 <0.05 <0.05 <0.05 | 0.58 0.63 0.41 0.21 0.18 | Study: 2000/1000222 Trial: OAT/01/99 Beck & Benz, 2000, Kresoxim_087 Max. frozen storage: 6 month |
| Denmark Middelfart 1998 (Ben Lomond) | Foliar spray | 3×100 | BBCH 89 | 0 2 7 14 21 | Fruit | 1.02 0.84 0.43 0.50 0.20 | <0.05 <0.05 <0.05 <0.05 <0.05 | <0.05 <0.05 <0.05 0.063 0.061 | 1.1 0.89 0.48 0.57 0.26 | Study: 1999/11508 Trial: ABL/01/98 Beck et al, 1999, Kresoxim_088 Max. frozen storage: 10 month |
| Great Britain Madley 1998 (Ben Lowman) | Foliar spray | 3×100 | BBCH 89 | 0 3 6 13 21 | Fruit | 0.72 0.31 0.10 0.22 0.12 | <0.05 <0.05 <0.05 <0.05 <0.05 | <0.05 <0.05 <0.05 <0.05 <0.05 | 0.77 0.36 0.15 0.18 0.17 | Study: 1999/11508 Trial: OAT/15/98 Beck et al, 1999, Kresoxim_088 Max. frozen storage: 10 month |

Table 89 Residues of kresoxim-methyl in black currant following foliar treatment (cGAP United Kingdom: 3×100 g ai/ha; 14 day PHI)

Strawberry

Four field trials were conducted with strawberry under greenhouse conditions in Germany, Great Britain, and The Netherlands during the 2013 growing season (Oxspring, 2013, Kresoxim_089). Plants received 3 spray applications of kresoxim-methyl at nominal rates of 150 g ai/ha with a 7±1 day interval between applications. Samples of strawberries were collected at 0, 2–4, 7, 14–15 and 21–22 days after the last application. Residues of kresoxim-methyl (as 490M1) and metabolites 490M2 and 490M9 were determined using method BASF method 350/4 with a limit of quantification of 0.01 mg/kg. Overall mean procedural recoveries for kresoxim-methyl (as 490M1) and metabolites 490M2 and 490M2 and 490M9 in strawberry spiked at 0.01 and 10 mg/kg were 100% (n=6), 95% (n=6) and 89% (n=6), respectively.

A total of 8 trials were conducted with strawberry under plastic tunnels during the 1999 (Beck et al, 1999b, Kresoxim_090) and 1998 (Beck & Benz, 1999, Kresoxim_091) growing seasons in Spain. Plants received 3 spray applications of kresoxim-methyl at nominal rates of 150 g ai/ha with a 7±1 day interval between applications. Samples of strawberries were collected at various time points. Residues of kresoxim-methyl (as 490M1) and metabolites 490M2 and 490M9 were determined using method BASF method 350/3 with a limit of quantification of 0.05 mg/kg. Overall mean procedural recoveries for kresoxim-methyl (as 490M1) and

metabolites 490M2 and 490M9 in strawberry spiked at 0.05 and 5 mg/kg were 99% (n=14), 103% (n=14) and 92% (n=14), respectively.

Table 90 Residues of kresoxim-methyl in strawberry following foliar treatment (cGAP Netherlands: 3×150 g ai/ha; 7 day PHI) under protected conditions

| | Treatment | Application | Growth | DALT | Crop | Residue For | und (mg/kg | 1) | | |
|--|-----------------|-------------------|----------------------------------|-------------------------|-------|--|---|---|--|---|
| Location, Year (variety) | method | rate (g ai/ha) | stage at final application | | part | Kresoxim- methyl | 490M2 | 490M9 | Sum as parent equiv. | Report/Trial No., Reference Storage period |
| Germany Rhineland- Palatinate 2013 (Darselect) | Foliar spray | 3×150 | BBCH 81 | 0 3 7 14 22 | Fruit | 0.54 0.34 0.18 0.19 0.049 | <0.01 <0.01 <0.01 <0.01 <0.01 | <0.01 <0.01 <0.01 <0.01 <0.01 | 0.56 0.36 0.20 0.21 0.069 | Study: 2013/1343454 Trial: L130348 Oxspring, 2013, Kresoxim_089 Max. frozen storage: 3 month |
| Great Britain Nottinghamshire 2013 (Elsanta) | Foliar spray | 3×150 | BBCH 65- 85 | 0 4 7 14 21 | Fruit | 0.39 0.44 0.31 0.25 0.20 | <0.01 <0.01 <0.01 0.013 0.010 | <0.01 <0.01 <0.01 0.023 0.021 | 0.41 0.46 0.33 0.29 0.23 | Study: 2013/1343454 Trial: L130349 Oxspring, 2013, Kresoxim_089 Max. frozen storage: 2 month |
| Great Britain Staffordshire 2013 (Sonata) | Foliar spray | 3×150 | BBCH 81 | 0 4 7 14 21 | Fruit | 0.28 0.33 0.30 0.22 0.10 | <0.01 <0.01 <0.01 <0.01 <0.01 | <0.01 <0.01 <0.01 <0.01 <0.01 | 0.30 0.35 0.32 0.24 0.12 | Study: 2013/1343454 Trial: L130350 Oxspring, 2013, Kresoxim_089 Max. frozen storage: 2 month |
| The Netherland Doornenburg 2013 (Elsanta) | Foliar spray | 3×150 | BBCH 60- 87 | 0 2 7 15 21 | Fruit | 0.25 0.22 0.17 0.066 0.049 | <0.01 <0.01 <0.01 <0.01 <0.01 | <0.01 <0.01 <0.01 <0.01 <0.01 | 0.27 0.24 0.19 0.086 0.069 | Study: 2013/1343454 Trial: L130351 Oxspring, 2013, Kresoxim_089 Max. frozen storage: 3 month |
| Spain Huelva Bonares 1999 (Camarosa) | Foliar spray | 3×150 | BBCH 85 | 0 7 14 | Fruit | 0.98 0.56 0.41 | <0.05 <0.05 <0.05 | <0.05 0.09 0.11 | 1.08 0.70 0.57 | Study: 1999/11992 Trial: AC/01/99 Beck et al, 1999b, Kresoxim_090 Max. frozen storage: 8 month |

786

| | Treatment | Application | Growth | DALT | Crop | Residue For | und (mg/kg | I) | | |
|--|-----------------|-------------------|----------------------------------|--|---|-------------------------------------|--|--|-------------------------------------|---|
| Location, Year (variety) | method | rate (g ai/ha) | stage at final application | | part | Kresoxim- methyl | 490M2 | 490M9 | Sum as parent equiv. | Report/Trial No., Reference Storage period |
| Spain Huelva Moguer 1999 (Camarosa) | Foliar spray | 3×150 | BBCH 85 | 0 7 15 | Fruit | 1.37 0.62 0.35 | <0.05 <0.05 <0.05 | <0.05 <0.05 <0.05 | 1.47 0.72 0.45 | Study: 1999/11992 Trial: AC/02/99 Beck et al, 1999b, Kresoxim_090 Max. frozen storage: 8 month |
| Spain Huelva Cartaya 1999 (Camarosa) | Foliar spray | 3×150 | BBCH 85 | 0 7 14 | Fruit | 1.16 0.64 0.33 | <0.05 <0.05 <0.05 | <0.05 <0.05 <0.05 | 1.26 0.74 0.43 | Study: 1999/11992 Trial: AC/03/99 Beck et al, 1999b, Kresoxim_090 Max. frozen storage: 8 month |
| Spain Huelva Lepe 1999 (Camarosa) | Foliar spray | 3×150 | BBCH 85 | 0 7 14 | Fruit | 0.51 0.48 0.31 | <0.05 <0.05 <0.05 | <0.05 <0.05 <0.05 | 0.61 0.58 0.41 | Study: 1999/11992 Trial: AC/04/99 Beck et al, 1999b, Kresoxim_090 Max. frozen storage: 8 month |
| Spain Huelva Bonares 1998 (Camarosa) | Foliar spray | 3×150 | BBCH 87- 89 | 7 ^a 8 ^b 6 13 0 | Fruit Whole plant w/o roots | 0.14 0.30 0.26 0.16 9.2 | <0.05 <0.05 <0.05 <0.05 <0.05 | <0.05 <0.05 <0.05 <0.05 0.37 | 0.24 0.40 0.36 0.26 9.7 | Study: 1999/11996 Trial: AC/01/98 Beck & Benz, 1999, Kresoxim_091 Max. frozen storage: 7 month |
| Spain Huelva Bollullos 1998 (Oso Grande) | Foliar spray | 3×150 | BBCH 87- 89 | 7 ^a 8 ^b 6 13 0 | Fruit Whole plant w/o roots | 0.17 0.06 0.27 0.07 4.1 | <0.05 <0.05 <0.05 <0.05 <0.05 <0.05 | <0.05 <0.05 <0.05 <0.05 0.13 | 0.27 0.16 0.37 0.17 4.3 | Study: 1999/11996 Trial: AC/02/98 Beck & Benz, 1999, Kresoxim_091 Max. frozen storage: 7 month |
| Spain Huelva Moguer 1998 (Camarosa) | Foliar spray | 3×150 | BBCH 87- 89 | 6 ^a 7 ^b 7 13 0 | Fruit Whole plant w/o roots | 0.23 0.37 0.30 0.13 15 | <0.05 <0.05 <0.05 <0.05 <0.05 | <0.05 <0.05 <0.05 <0.05 0.11 | 0.43 0.47 0.40 0.24 16 | Study: 1999/11996 Trial: AC/03/98 Beck & Benz, 1999, Kresoxim_091 Max. frozen storage: 7 month |

| | Treatment | Application | Growth | DALT | Crop | Residue Fou | und (mg/kg | I) | | |
|----------------|-----------|-------------|-------------|-----------------------|-------|-------------|------------|-------|--------|----------------|
| Location, | method | rate | stage at | | part | Kresoxim- | 490M2 | 490M9 | Sum | Report/Trial |
| | | (g ai/ha) | final | | | methyl | | | as | No., Reference |
| Year (variety) | | | application | | | | | | parent | Storage period |
| | | | | | | | | | equiv. | |
| Spain | Foliar | 3×150 | BBCH 89 | 6 ^a | Fruit | 0.20 | <0.05 | <0.05 | 0.30 | Study: |
| Huelva | spray | | | 6 ^b | | 0.34 | <0.05 | <0.05 | 0.44 | 1999/11996 |
| Moguer | | | | 8 | | 0.13 | <0.05 | <0.05 | 0.23 | Trial: |
| 1998 | | | | 14 | | 0.06 | <0.05 | <0.05 | 0.16 | AC/04/98 |
| (Camarosa) | | | | 0 | Whole | 10 | <0.05 | 0.12 | 10 | Beck & Benz, |
| | | | | | plant | | | | | 1999, |
| | | | | | w/o | | | | | Kresoxim_091 |
| | | | | | roots | | | | | Max. frozen |
| | | | | | | | | | | storage: 7 |
| | | | | | | | | | | month |

^a after the first application

^b after the second application

A total of nine trials were conducted with strawberry under field conditions during the 1998 (Raunft et al., 1999, Kresoxim_092) and 1999 (Raunft & Benz, 1999, Kresoxim_093) growing seasons in Belgium, Denmark, Germany, the Netherlands and Sweden. Plants received 5 spray applications of kresoxim-methyl at nominal rates of 150 g ai/ha with a 10 day interval between applications. Samples of strawberries were collected at 0, 6–8, 13–15 and 19–22 days after the last application. Residues of kresoxim-methyl (as 490M1) and metabolites 490M2 and 490M9 were determined using method BASF method 350/3 with a limit of quantification of 0.05 mg/kg. Overall mean procedural recoveries for kresoxim-methyl (as 490M1) and metabolites 490M2 and 490M9 in strawberry spiked at 0.05–5 mg/kg were 98% (n=17), 99% (n=17) and 85% (n=18), respectively.

Table 91 Residues of kresoxim-methyl in strawberry following foliar treatment (cGAP Netherlands: 3×150 g ai/ha; 7 day PHI) under field conditions

| Leveller | Treatment | Applicatio | Growth | DALT | Crop | Residue I | ound (mg/ | kg) | | Dement/TeleINIe |
|--|-----------------|------------------------|----------------------------------|---------------|--------------------------------|-------------------------|-------------------------|-------------------------|----------------------------|--|
| Location, Year (variety) | method | n rate (g ai/ha) | stage at final application | | part | Kresoxi m- methyl | 490M2 | 490M9 | Sum as parent equiv. | Report/Trial No., Reference Storage period |
| Belgium Kortenaken 1998 (Elsanta) | Foliar spray | 5×150 | BBCH 85 | 7 14 21 | Fruit | <0.05 <0.05 <0.05 | <0.05 <0.05 <0.05 | <0.05 <0.05 <0.05 | <0.15 <0.15 <0.15 | Study: 1999/12004 Trial: AGR/01/98 Raunft et al., |
| | | | | 0 | Whole plant w/o roots | 3.6 | <0.05 | 0.23 | 3.8 | 1999, Kresoxim_092 Max. frozen storage: 16 month |
| Germany Vehlefanz 1998 | Foliar spray | 5×150 | BBCH 73- 81 | 6 13 20 | Fruit | <0.05 <0.05 <0.05 | <0.05 <0.05 <0.05 | <0.05 <0.05 <0.05 | <0.15 <0.15 <0.15 | Study: 1999/12004 Trial: ACK/01/98 |
| (Elsanta) | | | | 0 | Whole plant w/o roots | 6.3 | <0.05 | 0.34 | 6.7 | Raunft et al., 1999, Kresoxim_092 Max. frozen storage: 16 month |
| Germany Bad Sassendorf 1998 (Tenira) | Foliar spray | 5×150 | BBCH 73 | 13 19 | Fruit | 0.07 <0.05 | <0.05 <0.05 | <0.05 <0.05 | 0.17 <0.15 | Study: 1999/12004 Trial: D08/01/98 Raunft et al., 1999, Kresoxim_092 Max. frozen storage: 16 month |

| | Treatment | Applicatio | Growth | DALT | Crop | Residue I | Found (mg/ | /ka) | | |
|--------------------------------------|-----------------|------------------------|----------------------------------|--------------------|--------------------------------|---------------------------------|----------------------------------|---------------------------------|--------------------------------|--|
| Location, Year (variety) | method | n rate (g ai/ha) | stage at final application | | part | Kresoxi m- methyl | 490M2 | 490M9 | Sum as parent equiv. | Report/Trial No., Reference Storage period |
| Germany Ahlen 1998 (Polka) | Foliar spray | 5×150 | BBCH 73 | 14 20 | Fruit | 0.13 0.10 | <0.05 <0.05 | <0.05 <0.05 | 0.23 0.20 | Study: 1999/12004 Trial: D08/03/98 Raunft et al., 1999, Kresoxim_092 Max. frozen storage: 16 month |
| The Netherlands Siebengewald | Foliar spray | 5×150 | BBCH 85 | 7 14 21 | Fruit | <0.05 <0.05 <0.05 | <0.05 <0.05 <0.05 | <0.05 <0.05 <0.05 | <0.15 <0.15 <0.15 | Study: 1999/12004 Trial: AGR/02/98 |
| 1998 (Korona) | | | | 0 | Whole plant w/o roots | 5.1 | <0.05 | 0.11 | 5.3 | Raunft et al., 1999, Kresoxim_092 Max. frozen storage: 16 month |
| Sweden Bjärred 1998 | Foliar spray | 5×150 | BBCH 81 | 8 15 22 | Fruit | <0.05 <0.05 <0.05 | <0.05 <0.05 <0.05 | <0.05 <0.05 <0.05 | <0.15 <0.15 <0.15 | Study: 1999/12004 Trial: HUS/01/98 |
| (Honeyone) | | | | 0 | Whole plant w/o roots | 4.8 | <0.05 | 0.20 | 5.1 | Raunft et al., 1999, Kresoxim_092 Max. frozen storage: 16 month |
| Germany Brandenburg 1999 | Foliar spray | 5×150 | BBCH 73- 81 | 7 14 21 | Fruit | <0.05 <0.05 <0.05 | <0.05 <0.05 <0.05 | <0.05 <0.05 <0.05 | <0.15 <0.15 <0.15 | Study: 1999/12005 Trial: ACK/08/99 |
| (Elsanta) | | | | 0 | Whole plant w/o roots | 3.6 | <0.05 | 0.29 | 3.9 | Raunft & Benz, 1999, Kresoxim_093 Max. frozen storage: 4 month |
| Denmark Fuenen Jutland 1999 | Foliar spray | 5×150 | BBCH 68- 71 | 8 14 21 0 | Fruit Whole | <0.05 <0.05 <0.05 3.4 | <0.05 <0.05 <0.05 <0.05 | <0.05 <0.05 <0.05 0.29 | <0.15 <0.15 <0.15 3.7 | Study: 1999/12005 Trial: ALB/16/99 Raunft & Benz, |
| (Sengana) | | | | | plant w/o roots | | | | | 1999, Kresoxim_093 Max. frozen storage: 4 month |
| Sweden Malmoe Skane 1999 | Foliar spray | 5×150 | BBCH 75 | 8 15 22 0 | Fruit Whole | <0.05 <0.05 <0.05 2.09 | <0.05 <0.05 <0.05 <0.05 | <0.05 <0.05 <0.05 0.36 | <0.15 <0.15 <0.15 2.5 | Study: 1999/12005 Trial: HUS/10/99 Raunft & Benz, |
| (Honeyone) | | | | | plant w/o roots | 2.07 | | 5.00 | 2.0 | 1999, Kresoxim_093 Max. frozen storage: 4 month |

Grape

Eight field trials were conducted with grapes in the United States of America during the 1997 growing season (Wofford & Riley, 1998, Kresoxim_094). Plants received 4 spray applications of kresoxim-methyl at nominal rates of 224 g ai/ha with a 10±1 day interval between applications (per site two plots were treated with different spay volumes). Samples of grapes were collected at 14±1 days after the last application. In addition, at one site samples were collected at 0, 7 and 21 DALT. Residues of kresoxim-methyl (as 490M1) and metabolites 490M2 and 490M9 were determined using method BASF method 350/3 with a limit of

quantification of 0.05 mg/kg. Overall mean procedural recoveries for kresoxim-methyl (as 490M1) and metabolites 490M2 and 490M9 in grapes spiked at 0.05 and 0.5 mg/kg were 85% (n=6), 101% (n=6) and 97% (n=6), respectively.

| | Treatment | Application | Growth | DALT | Crop | Residue Fou | und (mg/ka | 1) | | Report/Trial |
|--|-----------------|-------------------------|----------------------------------|----------|-------|---------------------------|-----------------------------|-----------------------------|-----------------------------|--|
| Location, Year (variety) | method | rate (g ai/ha) | stage at final application | | part | Kresoxim- methyl | 490M2 | 490M9 | Sum of parent + 490M9 | No., Reference Storage period |
| USA Yates | Foliar spray | 4×224 (467 L/ha) | 5 ft tall vines at verasion | 14 | Fruit | 0.30 ^a | 0.33ª | 0.06 ^a | 0.36 | Study: 1998/5134 |
| County, NY 1997 (Aurora) | Foliar spray | 4×224 (2333 L/ha) | | 14 | Fruit | 0.31ª | 0.54 ^a | 0.07 ^a | 0.38 | Trial: 97065 Wofford & Riley, 1998, Kresoxim_094 Max. frozen storage: 9 month |
| USA Kern | Foliar spray | 4×224 (467 L/ha) | 6 ft tall vines at maturity | 0 7 | Fruit | 0.24 0.18 ^b | <0.05 <0.05 ^b | <0.05 <0.05 ^b | 0.29 0.23 | Study: 1998/5134 |
| County, CA | | , , | 5 | 14 | | 0.18 | <0.05 | <0.05 | 0.23 | Trial: 97066 |
| 1997 | | | | 21 | | 0.10 | <0.05 | <0.05 | 0.15 | Wofford & |
| (Emperor) | Foliar | 4×224 | | 0 | Fruit | 0.43 | <0.05 | <0.05 | 0.48 | Riley, 1998, |
| | spray | (2333 | | 7 | | 0.31 ^b | <0.05 ^b | <0.05 ^b | 0.36 | Kresoxim_094 |
| | | L/ha) | | 14 21 | | 0.31 0.21 | <0.05 <0.05 | <0.05 <0.05 | 0.36 0.26 | Max. frozen storage: 6 |
| | | | | 21 | | 0.21 | <0.05 | <0.05 | 0.20 | month |
| USA Tulare | Foliar spray | 4×224 (467 L/ha) | 6 ft tall vines at maturity | 14 | Fruit | 0.18 | <0.05 | <0.05 | 0.23 | Study: 1998/5134 |
| County, CA 1997 (Emperor) | Foliar spray | 4×224 (2333 L/ha) | | 14 | Fruit | 0.27 | <0.05 | <0.05 | 0.32 | Trial: 97067 Wofford & Riley, 1998, Kresoxim_094 Max. frozen storage: 6 month |
| USA Glenn | Foliar spray | 4×224 (467 L/ha) | 6.5 ft tall vines at fruit | 14 | Fruit | 0.09 | 0.22 | 0.17 | 0.26 | Study: 1998/5134 |
| County, CA 1997 (Zinfandel) | Foliar spray | 4×224 (2333 L/ha) | development | 14 | Fruit | 0.12 | 0.30 | 0.25 | 0.37 | Trial: 97068 Wofford & Riley, 1998, Kresoxim_094 Max. frozen storage: 10 month |
| USA Fresno | Foliar spray | 4×224 (467 L/ha) | 6-8 ft tall vines with | 14 | Fruit | 0.07 | 0.07 | <0.05 | 0.12 | Study: 1998/5134 |
| County, CA Kerman 1997 (Thompson Seedless) | Foliar spray | 4×224 (2333 L/ha) | maturing grapes | 14 | Fruit | 0.05 | 0.05 | 0.10 | 0.15 | Trial: 97069 Wofford & Riley, 1998, Kresoxim_094 Max. frozen storage: 10 month |
| USA Fresno | Foliar spray | 4×224 (467 L/ha) | 6-8 ft tall vines with | 14 | Fruit | 0.11 | 0.10 | 0.05 | 0.16 | Study: 1998/5134 |
| County, CA Fresno 1997 (Thompson Seedless) | Foliar spray | 4×224 (2333 L/ha) | maturing grapes | 14 | Fruit | 0.09 | 0.14 | 0.07 | 0.16 | Trial: 97070 Wofford & Riley, 1998, Kresoxim_094 Max. frozen storage: 10 month |

Table 92 Residues of kresoxim-methyl in grapes following foliar treatment (cGAP USA: 4×224 g ai/ha; 14 day PHI)

| Location, | Treatment | Application | Growth | DALT | Crop | Residue Fou | und (mg/kg | g) | | Report/Trial |
|---|-----------------|-------------------------|----------------------------------|------|-------|---------------------|------------|-------|-----------------------------|---|
| Year (variety) | method | rate (g ai/ha) | stage at final application | | part | Kresoxim- methyl | 490M2 | 490M9 | Sum of parent + 490M9 | No., Reference Storage period |
| USA Washington | Foliar spray | 4×224 (467 L/ha) | 6.5 ft tall plants with | 14 | Fruit | 0.25 | <0.05 | <0.05 | 0.30 | Study: 1998/5134 |
| County, ID 1997 (Concord) | Foliar spray | 4×224 (2333 L/ha) | fruits near maturity | 14 | Fruit | 0.30 | 0.05 | <0.05 | 0.35 | Trial: 97071 Wofford & Riley, 1998, Kresoxim_094 Max. frozen storage: 9 month |
| USA Linn | Foliar spray | 4×224 (467 L/ha) | 6.5 ft tall plants | 14 | Fruit | 0.91 | <0.05 | 0.05 | 0.96 | Study: 1998/5134 |
| County, OR 1997 (White Riesling) | | 4×224 (2333 L/ha) | | 14 | Fruit | 0.58 | <0.05 | <0.05 | 0.63 | Trial: 97279 Wofford & Riley, 1998, Kresoxim_094 Max. frozen storage: 8 month |

^a mean of triplicate analyses

^b mean of duplicate analyses

The meeting noted that trials 97069 and 97070 were conducted at the same location and time. Hence, here only the higher residues of these trials were considered.

In the 1994 growing season a total of twelve field trials with grapes were conducted in the United States of America (Jackson, 1996, Kresoxim_095). Plants received 4 spray applications of kresoxim-methyl at nominal rates of 224 g ai/ha with a 10 day interval between applications. Samples of grapes were collected at 14, 30, 75, 90 and 105 days after the last application. However, only the 14 and 30 day PHI data is reported here. Residues of kresoxim-methyl (as 490M1) and metabolites 490M2 and 490M9 were determined using method BASF method 350/3 with a limit of quantification of 0.05 mg/kg. Overall mean procedural recoveries for kresoxim-methyl (as 490M1) and metabolites 490M2 and 490M9 in grapes spiked at 0.05–1 mg/kg were 89% (n=15), 88% (n=17) and 87% (n=17), respectively.

| Table 93 Residues of kresoxim-meth | yl in grapes following | g foliar treatment (cGAP USA: 4×224 d | ai/ha; 14 day PHI) |
|------------------------------------|------------------------|---------------------------------------|--------------------|
| | | | |

| Location, | Treatment | Application | Growth stage | DALT | Crop | Residue Fou | und (mg/kg | j) ^a | | Doport/Trial No |
|---|--------------|-------------|-------------------------|----------|-------|-----------------------------|----------------------------|-----------------------------|-----------------------------|--|
| Year (variety) | method | | at final application | | part | Kresoxim- methyl | 490M2 | 490M9 | Sum of parent + 490M9 | Report/Trial No., Reference Storage period |
| USA Fresno County, CA 1994 (Thompson seedless) | Foliar spray | 4×224 | Not stated | 14 30 | Fruit | 0.071 ^b <0.05 | <0.05 ^b 0.07 | <0.05 ^b <0.05 | | Study: 1996/5219 Trial: 94001 Jackson, 1996, Kresoxim_095 Max. frozen storage: 24 month |
| USA Tulare County, GA 1994 (Crimson Seedless) | Foliar spray | 4×224 | Not stated | 14 30 | Fruit | 0.12 <0.05 | <0.05 <0.05 | <0.05 <0.05 | | Study: 1996/5219 Trial: 94002 Jackson, 1996, Kresoxim_095 Max. frozen storage: 24 month |

| | Treatment | Application | Growth stage | DALT | Crop | Residue Fo | und (ma/k | a) ^a | | |
|---|--------------|-------------------|----------------------|----------|-------|--|--|--|-----------------------------|--|
| Location, Year (variety) | method | rate (g ai/ha) | at final application | | part | Kresoxim- methyl | 490M2 | 490M9 | Sum of parent + 490M9 | Report/Trial No., Reference Storage period |
| USA Kern County, CA 1994 (Red Globes) | Foliar spray | 4×224 | Not stated | 14 30 | Fruit | <0.05 <0.05 | <0.05 <0.05 | <0.05 <0.05 | <0.10 <0.10 | Study: 1996/5219 Trial: 94003 Jackson, 1996, Kresoxim_095 Max. frozen storage: 24 month |
| USA Monterey County, CA 1994 (Cabernet) | Foliar spray | 4×224 | Not stated | 14 30 | Fruit | 0.063 <0.05 | <0.05 <0.05 | <0.05 <0.05 | 0.11 <0.10 | Study: 1996/5219 Trial: 94004 Jackson, 1996, Kresoxim_095 Max. frozen storage: 24 month |
| USA Grant County, WA 1994 (White Riesling) | Foliar spray | 4×224 | Not stated | 14 30 | Fruit | 0.25 0.18 | <0.05 <0.05 | <0.05 <0.05 | 0.30 0.23 | Study: 1996/5219 Trial: 94005 Jackson, 1996, Kresoxim_095 Max. frozen storage: 24 month |
| USA Grant County, WA 1994 (Concord) | Foliar spray | 4×224 | Not stated | 14 30 | Fruit | 0.14 0.088 | <0.05 <0.05 | <0.05 <0.05 | 0.19 0.14 | Study: 1996/5219 Trial: 94006 Jackson, 1996, Kresoxim_095 Max. frozen storage: 24 month |
| USA Ontario County, NY 1994 (Catawba) | Foliar spray | 4×224 | Not stated | 14 30 | Fruit | 0.69 ^b 0.26 ^b | <0.05 ^b <0.05 ^b | <0.05 ^b <0.05 ^b | 0.74 0.31 | Study: 1996/5219 Trial: 94007 Jackson, 1996, Kresoxim_095 Max. frozen storage: 24 month |
| USA Yates County, CA 1994 (Concords) | Foliar spray | 4×224 | Not stated | 14 30 | Fruit | 0.42 0.18 ^b | <0.05 <0.05 ^b | <0.05 <0.05 ^b | 0.47 0.22 | Study: 1996/5219 Trial: 94008 Jackson, 1996, Kresoxim_095 Max. frozen storage: not stated |
| USA Northhampton County, PA 1994 (Cayuga) | Foliar spray | 4×224 | Not stated | 14 30 | Fruit | 0.61 0.49 ^b | 0.05 0.089 ^b | <0.05 0.051 ^b | 0.66 0.58 | Study: 1996/5219 Trial: 94009 Jackson, 1996, Kresoxim_095 Max. frozen storage: 24 month |

| Leastian | Treatment | Application | Growth stage | DALT | Crop | Residue Fou | und (mg/kg |)) ^a | | Dement /Trial No. |
|--|--------------|-------------------|-------------------------|----------|-------|---------------------------|-----------------------------|-----------------------------|-----------------------------|--|
| Location, Year (variety) | method | rate (g ai/ha) | at final application | | part | Kresoxim- methyl | 490M2 | 490M9 | Sum of parent + 490M9 | Report/Trial No., Reference Storage period |
| USA Kent County, MI 1994 (Concord) | Foliar spray | 4×224 | Not stated | 14 30 | Fruit | 0.42 ^b 0.43 | 0.053 ^b 0.098 | <0.05 ^b 0.074 | 0.47 0.50 | Study: 1996/5219 Trial: 94010 Jackson, 1996, Kresoxim_095 Max. frozen storage: 24 month |
| USA Sampson County, NC 1994 (Magnolia) | Foliar spray | 4×224 | Not stated | 14 30 | Fruit | 0.36 0.13 | <0.05 0.065 | <0.05 <0.05 | 0.41 0.18 | Study: 1996/5219 Trial: 94011 Jackson, 1996, Kresoxim_095 Max. frozen storage: 24 month |
| USA Johnston County, NC 1994 (Carlos) | Foliar spray | 4×224 | Not stated | 14 30 | Fruit | 0.35 0.11 | 0.055 0.11 | <0.05 <0.05 | 0.40 0.16 | Study: 1996/5219 Trial: 94012 Jackson, 1996, Kresoxim_095 Max. frozen storage: 24 month |

^a mean of duplicate analysis if not stated otherwise

^b mean of quadruplicate analyses

The meeting noted that trials 94005 and 94006 were conducted at the same location and time. Hence, here only the higher residues of these trials were considered.

Assorted tropical and sub-tropical fruits –edible peel

Olives for oil production

A total of four field trials were conducted with olives in Spain in the 1998 growing season (Meumann & Rabe, 1999, Kresoxim_136). Olives received 1 spray applications of kresoxim-methyl at a nominal rate of 100 g ai/ha. Samples of olives were collected at 30±1 days after the application. Residues of kresoxim-methyl (as 490M1) and metabolites 490M2 and 490M9 were determined using method BASF method 350/3 with a limit of quantification of 0.05 mg/kg. Overall mean procedural recoveries for kresoxim-methyl (as 490M1) and metabolites 490M2 and 490M2 and 490M9 in olive fruit spiked at 0.05–5 mg/kg were 87% (n=4), 93% (n=4) and 99% (n=4), respectively.

Additionally, a total of four field trials were conducted with olives in Spain in the 1997 growing season (Rabe, 1999, Kresoxim_137). Olives received 1 spray applications of kresoxim-methyl at a nominal rate of 100 g ai/ha. Samples of olives were collected at 0, 15, 30 and 43 days after the application. Residues of kresoxim-methyl (as 490M1) and metabolites 490M2 and 490M9 were determined using method BASF method 350/3 with a limit of quantification of 0.05 mg/kg. Overall mean procedural recoveries for kresoxim-methyl (as 490M1) and metabolites 490M2 and 490M9 in olive fruits spiked at 0.05–5 mg/kg were 75% (n=16), 104% (n=16) and 101% (n=16), respectively.

| | Treatment | Application | Growth stage | DALT | Crop | Residue Four | nd (ma/ka) | | | |
|--|-----------------|-------------------|----------------------|---------------------|-------|---------------------------------|----------------------------------|----------------------------------|---------------------------------|--|
| Location, Year (variety) | method | rate (g ai/ha) | at final application | | part | Kresoxim- methyl | 490M2 | 490M9 | Sum of parent + 490M9 | Report/Trial No., Reference Storage period |
| Spain Andalusia Anduja 1998 (Picual) | Foliar spray | 1×100 | BBCH 79-85 | 29 | Fruit | <0.05 | <0.05 | <0.05 | <0.10 | Study: 1999/10705 Trial: AC/22/98 Meumann & Rabe, 1999, Kresoxim_136 Max. frozen storage: 4 month |
| Spain Andalusia La Higuera 1998 (Picual) | Foliar spray | 1×100 | BBCH 79-85 | 29 | Fruit | <0.05 | <0.05 | <0.05 | <0.10 | Study: 1999/10705 Trial: AC/23/98 Meumann & Rabe, 1999, Kresoxim_136 Max. frozen storage: 4 month |
| Spain Andalusia Moraleda de Zafayona 1998 (Martena) | Foliar spray | 1×100 | BBCH 79-85 | 30 | Fruit | <0.05 | <0.05 | <0.05 | <0.10 | Study: 1999/10705 Trial: AC/24/98 Meumann & Rabe, 1999, Kresoxim_136 Max. frozen storage: 4 month |
| Spain 1998 Andalusia Moraleda de Zafayona (Hoj. blanca) | Foliar spray | 1×100 | BBCH 79-80 | 30 | Fruit | 0.11 | <0.05 | <0.05 | 0.16 | Study: 1999/10705 Trial: AC/25/98 Meumann & Rabe, 1999, Kresoxim_136 Max. frozen storage: 4 month |
| Spain 1997 (not stated) | Foliar spray | 1×100 | Not stated | 0 15 30 43 | Fruit | 0.23 0.11 0.09 <0.05 | <0.05 <0.05 0.05 0.06 | <0.05 <0.05 <0.05 <0.05 | 0.28 0.16 0.14 0.10 | Study: 1999/10706 Trial: OL/97/01 Rabe, 1999, Kresoxim_137 Max. frozen storage: not stated |
| Spain 1997 (not stated) | Foliar spray | 1×100 | Not stated | 0 15 30 43 | Fruit | 0.12 0.05 <0.05 <0.05 | <0.05 <0.05 <0.05 0.06 | <0.05 <0.05 <0.05 <0.05 | 0.17 0.10 <0.10 0.10 | Study: 1999/10706 Trial: OL/97/02 Rabe, 1999, Kresoxim_137 Max. frozen storage: not stated |
| Spain 1997 (not stated) | Foliar spray | 1×100 | Not stated | 0 15 30 43 | Fruit | 0.09 <0.05 <0.05 <0.05 | <0.05 <0.05 <0.05 <0.05 | <0.05 <0.05 <0.05 <0.05 | 0.14 <0.10 <0.10 <0.10 | Study: 1999/10706 Trial: OL/97/03 Rabe, 1999, Kresoxim_137 Max. frozen storage: not stated |

Table 94 Residues of kresoxim-methyl in olive roots following foliar treatment (cGAP: 3 × 100 g ai/ha; 30 days PHI)

| Leastien | Treatment | Application | Growth stage | DALT | Crop | Residue Foun | d (mg/kg) | | | Doment/Trial No. |
|-----------------------------|-----------|-------------------|-------------------------|------|-------|---------------------|-----------|-------|-----------------------------|--|
| Location, Year (variety) | method | rate (g ai/ha) | at final application | | part | Kresoxim- methyl | 490M2 | 490M9 | Sum of parent + 490M9 | Report/Trial No., Reference Storage period |
| Spain | Foliar | 1×100 | Not stated | 0 | Fruit | 0.13 | <0.05 | <0.05 | 0.18 | Study: |
| 1997 | spray | | | 15 | | 0.09 | <0.05 | <0.05 | 0.14 | 1999/10706 |
| (not stated) | | | | 30 | | <0.05 | <0.05 | <0.05 | <0.10 | Trial: 0L/97/04 |
| | | | | 43 | | <0.05 | 0.05 | <0.05 | 0.10 | Rabe, 1999, |
| | | | | | | | | | | Kresoxim_137 |
| | | | | | | | | | | Max. frozen |
| | | | | | | | | | | storage: not |
| | | | | | | | | | | stated |

The meeting noted that for trials OL/97/01, OL/97/02, OL/97/03, OL/97/04 no information on the location of the trials was given. Hence, the trials were not considered independent and only the highest residue of these trials was considered.

Assorted tropical and sub-tropical fruits – inedible peel

Mango

A total of nine field trials were conducted with mango in Brazil in the 2010–11 and 2014–15 growing seasons (Silva & Alves, 2011a, Kresoxim_049; Silva, 2011 Kresoxim_050; Silva, 2015a, Kresoxim_096; Silva, 2015b, Kresoxim_077; Silva & Alves, 2011b, Kresoxim_098). Plants received either 2 spray applications of kresoxim-methyl at nominal rates of 120 g ai/ha with a 15 day interval between applications, or 2 spray application at nominal rates of 300 g ai/ha with a 14 day interval between applications. Samples of mango were collected at 0, 7 and 14 days after the last application. Residues of kresoxim-methyl (as 490M1) and metabolites 490M2 and 490M9 were determined using method BASF method 350/4 (identical to method L0095/01 and SOP-PA.0295) with a limit of quantification of 0.01 mg/kg. Overall mean procedural recoveries for kresoxim-methyl (as 490M1) and metabolites 490M2 and 490M9 in mango spiked at 0.01–1 mg/kg were 78% (n=2), 97% (n=2) and 103% (n=2), respectively.

Table 3: Residues of kresoxim-methyl in mango following foliar treatment (cGAP Brazil: 2×120 g ai/ha; 7 day PHI)

| | Treatment | Application | Growth stage at | DALT | Crop | Residue Found | | | | |
|--|--------------|-------------|-------------------|--------------|-------|-------------------------|-------------------------|-------------------------|-----------------------------|--|
| Location, Year (variety) | method | | final application | | | Kresoxim- methyl | 490M2 | 490M9 | Sum of parent + 490M9 | Report/Trial No., Reference Storage period |
| Brazil Rolândia, PR 2014 (Tommy Atkins) | Foliar spray | 2×120 | BBCH 85-89 | 0 7 14 | Fruit | <0.01 <0.01 <0.01 | <0.01 <0.01 <0.01 | <0.01 <0.01 <0.01 | <0.02 <0.02 <0.02 | Study: 2015/3002561, 2015/3002961 Trial: G140014 Silva, 2015a, Kresoxim_096; Silva, 2015b, Kresoxim_097 Max. frozen storage: 11 month |
| Brazil Petrolina, PE 2014 ^a (Palmer) | Foliar spray | 2×120 | BBCH 88 | 7 | Fruit | 0.014 | <0.01 | <0.01 | 0.024 | Study: 2015/3002561, 2015/3002961 Trial: G140015 Silva, 2015a, Kresoxim_096; Silva, 2015b, Kresoxim_097 Max. frozen storage: 11 month |

| | Treatment | Application | Growth stage at | DALT | Crop | Residue Fou | nd (mg/kg) | | | |
|---|--------------|-------------------|-------------------|--------------|-------|-----------------------------------|-------------------------|-------------------------|-----------------------------|--|
| Location, Year (variety) | method | rate (g ai/ha) | final application | | part | Kresoxim- methyl | 490M2 | 490M9 | Sum of parent + 490M9 | Report/Trial No., Reference Storage period |
| Brazil Mogi Mirirm, SP 2014 (Choc Anao) | Foliar spray | 2×120 | BBCH 83 | 7 | Fruit | 0.041 | 0.038 | 0.014 | 0.055 | Study: 2015/3002561, 2015/3002961 Trial: G140022 Silva, 2015a, Kresoxim_096; Silva, 2015b, Kresoxim_097 Max. frozen storage: 12 month |
| Brazil Petrolina, PE 2014 ^a (Tommy) | Foliar spray | 2×120 | BBCH 78-85 | 0 7 14 | Fruit | 0.11 <u>0.049</u> <0.01 | 0.021 0.015 0.01 | <0.01 <0.01 <0.01 | 0.12 0.059 0.02 | Study: 2015/3002561, 2015/3002961 Trial: G140111 Silva, 2015a, Kresoxim_096; Silva, 2015b, Kresoxim_097 Max. frozen storage: 7 month |
| Brazil Uraí, PR 2014 (Palmer) | Foliar spray | 2×120 | BBCH 85 | 0 7 14 | Fruit | 0.011 <u><0.01</u> <0.01 | <0.01 <0.01 <0.01 | <0.01 <0.01 <0.01 | 0.021 <0.02 <0.02 | Study: 2015/3002561, 2015/3002961 Trial: G140257 Silva, 2015a, Kresoxim_096; Silva, 2015b, Kresoxim_097 Max. frozen storage: 2 month |
| Brazil Londrina, PR 2010-11 (Tommy Atkins) | Foliar spray | 2×300 | BBCH 81 | 0 7 14 | Fruit | 0.09 0.06 <0.01 | <0.01 0.02 <0.01 | <0.01 0.01 <0.01 | 0.10 0.07 <0.02 | Study: 2011/3008004, 2011/1226624 Trial: G100443 Silva & Alves, 2011, Kresoxim_049; Silva & Alves, 2011b, Kresoxim_098 Max. frozen storage: 8 month |
| Brazil Anápolis, GO 2010-11 (Tommy) | Foliar spray | 2×300 | BBCH 85 | 0 7 14 | Fruit | 0.24 0.14 0.06 | 0.02 0.03 0.03 | 0.01 0.02 0.02 | 0.25 0.16 0.08 | Study: 2011/3008004, 2011/1226624 Trial: G100444 Silva & Alves, 2011, Kresoxim_049; Silva & Alves, 2011b, Kresoxim_098 Max. frozen storage: 8 month |

| | Treatment | Application | Growth stage at final application | | part | Residue Found | d (mg/kg) | | | Dement/Trial Na |
|---|--------------|-------------------|-----------------------------------|---|-------|---------------------|-----------|-------|-----------------------------|--|
| Location, Year (variety) | method | rate (g ai/ha) | | | | Kresoxim- methyl | 490M2 | 490M9 | Sum of parent + 490M9 | Report/Trial No., Reference Storage period |
| Brazil Sto. Antônio de Posse, SP 2010-11 (Palmer) | Foliar spray | 2×300 | BBCH 87 | 7 | Fruit | 0.03 | <0.01 | <0.01 | 0.04 | Study: 2011/3008004, 2011/1226624 Trial: G100445 Silva & Alves, 2011, Kresoxim_049; Silva & Alves, 2011b, Kresoxim_098 Max. frozen storage: 8 month |
| Brazil Uraí, PR 2014 (Palmer) | Foliar spray | 2×300 | BBCH 85 | 7 | Fruit | 0.10 | 0.03 | 0.02 | 0.12 | Study: 2011/3008004, 2011/1226624 Trial: G100446 Silva & Alves, 2011, Kresoxim_049; Silva & Alves, 2011b, Kresoxim_098 Max. frozen storage: 7 month |

^a Trials can be considered independent since actual treatment days differed by about 4 months.

Bulb vegetables

Bulb onion

A total of 12 field trials were conducted with onion in Germany and the Netherlands in the 1995, 1996 and 1997 growing seasons (Raunft & Schulz, 1998, Kresoxim_103; Fuchs & Rabe, 1997, Kresoxim_104; Raunft & Rabe, 1997, Kresoxim_105). Plants received 7 spray applications of kresoxim-methyl at nominal rates of 200 g ai/ha with an 8 day interval between applications. Samples of onion were collected at 0, 5, 7, 13–14 and 21 days after the last application. Residues of kresoxim-methyl (as 490M1) and metabolites 490M2 and 490M9 were determined using method BASF method 350/3 with a limit of quantification of 0.05 mg/kg. Overall mean procedural recoveries for kresoxim-methyl (as 490M1) and metabolites 490M2 and 490M9 in onion spiked at 0.05 and 5 mg/kg were 91% (n=27), 89% (n=27) and 90% (n=27), respectively.

Table 96 Residues of kresoxim-methyl in onion following foliar treatment in Germany and the Netherlands

| Location, | Treatment | Application | Growth stage | DALT | Crop | Residue Found | l (mg/kg) | I | Report/Trial No., |
|-----------------|--------------|-------------|--------------|------|-------|---------------|-----------|----------|-------------------|
| | method | rate | at final | | part | Kresoxim- | 490M2 | 490M9 | Reference |
| Year (variety) | | (g ai/ha) | application | | | methyl | | | Storage period |
| Germany | Foliar spray | 7×200 | BBCH 47 | 0 | Bulbs | 0.06 | <0.05 | <0.05 | Study: |
| Nordrhein- | | | | 5 | | 0.10 | <0.05 | <0.05 | 1998/10791 |
| Westfalen | | | | 7 | | <0.05 | <0.05 | <0.05 | Trial: AGR/01/97 |
| Üdermerbruch | | | | 14 | | <0.05 | <0.05 | <0.05 | Raunft & Schulz, |
| 1997 | | | | 21 | | <0.05 | <0.05 | <0.05 | 1998, |
| (Hysam) | Foliar spray | 7×204 | BBCH 47 | 0 | Bulbs | 0.08 | <0.05 | <0.05 | Kresoxim_103 |
| | | | | 5 | | 0.09 | <0.05 | <0.05 | Max. frozen |
| | | | | 7 | | 0.19 | <0.05 | <0.05 | storage: 10 month |
| | | | | 14 | | <0.05 | <0.05 | <0.05 | |
| | | | | 21 | | <0.05 | <0.05 | <0.05 | |
| The Netherlands | Foliar spray | 7×200 | BBCH 47 | 0 | Bulbs | 0.13 | <0.05 | <0.05 | Study: |
| Limburg | | | | 5 | | <0.05 | <0.05 | <0.05 | 1998/10791 |
| Ottersum | | | | 7 | | <0.05 | <0.05 | <0.05 | Trial: AGR/02/97 |
| 1997 | | | | 14 | | <0.05 | <0.05 | <0.05 | Raunft & Schulz, |
| (Sturon) | | | | 21 | | <0.05 | <0.05 | <0.05 | 1998, |

| Location, | Treatment | Application | Growth stage | DALT | Crop | Residue Fou | nd (mg/kg) | | Report/Trial No., |
|--------------------|---------------------|-------------|--------------|---------|---------|---------------|----------------|----------------|-----------------------------------|
| | method | rate | at final | | part | Kresoxim- | 490M2 | 490M9 | Reference |
| Year (variety) | | (g ai/ha) | application | | | methyl | | | Storage period |
| | Foliar spray | 7×204 | BBCH 47 | 0 | Bulbs | 0.15 | < 0.05 | < 0.05 | Kresoxim_103 |
| | | | | 5 | | < 0.05 | < 0.05 | <0.05 | Max. frozen |
| | | | | 7 | | 0.06 | < 0.05 | <0.05 | storage: 10 month |
| | | | | 14 | | < 0.05 | <0.05 | < 0.05 | |
| | | | | 21 | | < 0.05 | <0.05 | <0.05 | |
| The Netherlands | Foliar spray | 7×200 | BBCH 47 | 0 | Bulbs | 0.15 | <0.05 | <0.05 | Study: |
| Gelderland | | | | 5 | | 0.09 | < 0.05 | <0.05 | 1998/10791 |
| Slijk-Ewijk | | | | 7 | | < 0.05 | <0.05 | < 0.05 | Trial: AGR/03/97 |
| 1997 | | | | 14 | | < 0.05 | < 0.05 | < 0.05 | Raunft & Schulz, |
| (Salder) | | | | 21 | | <0.05 | < 0.05 | < 0.05 | 1998, |
| | Foliar spray | 7×204 | BBCH 47 | 0 | Bulbs | 0.21 | < 0.05 | < 0.05 | Kresoxim_103 |
| | | | | 5 | | 0.15 | <0.05 | < 0.05 | Max. frozen |
| | | | | 7 | | <0.05 | < 0.05 | < 0.05 | storage: 10 month |
| | | | | 14 | | <0.05 | < 0.05 | < 0.05 | |
| | | | | 21 | | <0.05 | < 0.05 | < 0.05 | |
| The Netherlands | Foliar spray | 7×200 | BBCH 47 | 0 | Bulbs | 0.12 | < 0.05 | < 0.05 | Study: |
| Gelderland | | | | 5 | | 0.06 | <0.05 | <0.05 | 1998/10791 |
| Millingen | | | | 7 | | <0.05 | <0.05 | <0.05 | Trial: AGR/04/97 |
| 1997 | | | | 14 | | <0.05 | < 0.05 | < 0.05 | Raunft & Schulz, |
| (Hyfiela) | | | | 21 | | <0.05 | < 0.05 | <0.05 | 1998, |
| | Foliar spray | 7×204 | BBCH 47 | 0 | Bulbs | 0.11 | < 0.05 | < 0.05 | Kresoxim_103 |
| | | | | 5 | | <0.05 | < 0.05 | < 0.05 | Max. frozen |
| | | | | 7 | | 0.05 | <0.05 | <0.05 | storage: 10 month |
| | | | | 14 | | <0.05 | <0.05 | <0.05 | |
| | | | _ | 21 | | <0.05 | <0.05 | <0.05 | _ |
| Germany | Foliar spray | 7×200 | BBCH 48 | 0 | Bulbs | <0.05 | <0.05 | 0.10 | Study: |
| Nordrhein- | | | | 5 | | <0.05 | <0.05 | <0.05 | 1997/10280 |
| Westfalen | | | | 7 | | <0.05 | <0.05 | <0.05 | Trial: AGR/06/95 |
| Üdermerbruch | | | | 14 | | < 0.05 | < 0.05 | < 0.05 | Fuchs & Rabe, |
| 1995 | | | | 21 | | <0.05 | <0.05 | <0.05 | 1997, |
| (Hysam) | | | | | | | | | Kresoxim_104 |
| | | | | | | | | | Max. frozen |
| The Nietherslevele | F - U | 7 000 | DDOLL 40 | 0 | Duiller | 0.05 | 0.05 | 0.05 | storage: 10 month |
| The Netherlands | Foliar spray | 7×200 | BBCH 48 | 0 | Bulbs | < 0.05 | < 0.05 | < 0.05 | Study: 1997/10280 |
| Limburg | | | | 5 7 | | < 0.05 | <0.05 <0.05 | <0.05 <0.05 | |
| Ottersum 1995 | | | | 7 14 | | <0.05 0.06 | <0.05 <0.05 | <0.05 <0.05 | Trial: AGR/07/95 Fuchs & Rabe, |
| (Sturon) | | | | 21 | | <0.05 | < 0.05 | < 0.05 | 1997, |
| (Staron) | | | | 21 | | <0.05 | <0.05 | <0.05 | Kresoxim 104 |
| | | | | | | | | | Max. frozen |
| | | | | | | | | | storage: 10 month |
| The Netherlands | Foliar sprav | 7×200 | BBCH 48 | 0 | Bulbs | <0.05 | <0.05 | <0.05 | Study: |
| Limburg | . onai spruy | | 550110 | 5 | 20103 | <0.05 | < 0.05 | <0.05 | 1997/10280 |
| Siebengewald | | | | 7 | | <0.05 | < 0.05 | <0.05 | Trial: AGR/08/95 |
| 1995 | | | | 14 | | <0.05 | < 0.05 | < 0.05 | Fuchs & Rabe, |
| (Stuttgarter | | | | 21 | | < 0.05 | < 0.05 | < 0.05 | 1997, |
| Riesen) | | | | 1 | | | | | Kresoxim_104 |
| , | | | | | | | | | Max. frozen |
| | | | | | | | | | storage: 10 month |
| The Netherlands | Foliar spray | 7×200 | BBCH 48 | 0 | Bulbs | <0.05 | < 0.05 | <0.05 | Study: |
| Nord-Brabant | . , | | | 5 | | <0.05 | < 0.05 | < 0.05 | 1997/10280 |
| Haps | | | | 7 | | <0.05 | <0.05 | <0.05 | Trial: AGR/09/95 |
| 1995 | | | | 13 | | <0.05 | <0.05 | <0.05 | Fuchs & Rabe, |
| (Stuttgarter | | | | 21 | | <0.05 | < 0.05 | < 0.05 | 1997, |
| Riesen) | | | | 1 | | | | | Kresoxim_104 |
| | | | | | | | | | Max. frozen |
| | | | | 1 | | | | | storage: 10 month |

| Location, | Treatment | Application | Growth stage | DALT | Crop | Residue Fou | nd (mg/kg) | | Report/Trial No., |
|-----------------|--------------|-------------|--------------|------|-------|-------------|------------|--------|-------------------|
| | method | rate | at final | | part | Kresoxim- | 490M2 | 490M9 | Reference |
| Year (variety) | | (g ai/ha) | application | | | methyl | | | Storage period |
| Germany | Foliar spray | 7×200 | BBCH 48 | 0 | Bulbs | <0.05 | <0.05 | <0.05 | Study: |
| Nordrhein- | | | | 5 | | <0.05 | < 0.05 | <0.05 | 1997/10281 |
| Westfalen | | | | 7 | | <0.05 | < 0.05 | <0.05 | Trial: AGR/02/96 |
| Üdermerbruch | | | | 14 | | < 0.05 | < 0.05 | <0.05 | Raunft & Rabe, |
| 1996 | | | | 21 | | < 0.05 | < 0.05 | <0.05 | 1997, |
| (Hysam) | | | | | | | | | Kresoxim_105 |
| | | | | | | | | | Max. frozen |
| | | | | | | | | | storage: 21 month |
| The Netherlands | Foliar spray | 7×200 | BBCH 48 | 0 | Bulbs | <0.05 | <0.05 | <0.05 | Study: |
| Gelderland | | | | 5 | | <0.05 | < 0.05 | < 0.05 | 1997/10281 |
| Slijk-Ewijk | | | | 7 | | <0.05 | < 0.05 | < 0.05 | Trial: AGR/03/96 |
| 1995 | | | | 14 | | <0.05 | < 0.05 | < 0.05 | Raunft & Rabe, |
| (Jumbo) | | | | 21 | | <0.05 | < 0.05 | < 0.05 | 1997, |
| | | | | | | | | | Kresoxim_105 |
| | | | | | | | | | Max. frozen |
| | | | | | | | | | storage: 21 month |
| The Netherlands | Foliar spray | 7×200 | BBCH 47 | 0 | Bulbs | <0.05 | <0.05 | <0.05 | Study: |
| Gelderland | | | | 5 | | <0.05 | < 0.05 | < 0.05 | 1997/10281 |
| Millingen | | | | 7 | | <0.05 | < 0.05 | < 0.05 | Trial: AGR/04/96 |
| 1995 | | | | 14 | | <0.05 | < 0.05 | < 0.05 | Raunft & Rabe, |
| (Heyfast) | | | | 21 | | <0.05 | < 0.05 | < 0.05 | 1997, |
| | | | | | | | | | Kresoxim_104 |
| | | | | | | | | | Max. frozen |
| | | | | | | | | | storage: 21 month |
| The Netherlands | Foliar spray | 7×200 | BBCH 48 | 0 | Bulbs | <0.05 | <0.05 | <0.05 | Study: |
| Limburg | | | | 5 | | <0.05 | < 0.05 | < 0.05 | 1997/10281 |
| Ottersum | | | | 7 | | <0.05 | < 0.05 | <0.05 | Trial: AGR/05/96 |
| 1995 | | | | 14 | | <0.05 | < 0.05 | < 0.05 | Raunft & Rabe, |
| (Sturon F1) | | | | 21 | | <0.05 | < 0.05 | < 0.05 | 1997, |
| | | | | | | | | | Kresoxim_104 |
| | | | | | | | | | Max. frozen |
| | 1 | | | | | | | | storage: 21 month |

Additionally total of four field trials were conducted with onion in Brazil in the 2010 growing season (Silva & Cardoso, 2011a, Kresoxim_106; Silva, 2011a Kresoxim_107). Plants received 4 spray applications of kresoxim-methyl at nominal rates of 70 g ai/ha with a 7 day interval between applications. Samples of mango were collected at 0, 7 and 14 days after the last application. Residues of kresoxim-methyl (as 490M1) and metabolites 490M2 and 490M9 were determined using method BASF method 350/4 (identical to method L0095/01 and SOP-PA.0295) with a limit of quantification of 0.01 mg/kg. Overall mean procedural recoveries for kresoxim-methyl (as 490M1) and metabolites 490M2 and 490M9 in onion spiked at 0.01 and 1 mg/kg were 91% (n=12), 96% (n=12) and 84% (n=12), respectively.

Table 97 Residues of kresoxim-methyl in onion following foliar treatment in Brazil

| Location. | Treatment | Application | Growth | DALT | Crop | Residue Foun | d (mg/kg) | | Report/Trial No., |
|--|--------------|-------------------|----------------------------------|------|-------|---------------------|-----------|-------|--|
| Year (variety) | method | rate (g ai/ha) | stage at final application | | part | Kresoxim- methyl | 490M2 | 490M9 | Reference Storage period |
| Brazil Senador Canedo/GO 2010 (Cadillac H) | Foliar spray | 4×70 | BBCH 48 | 7 | Bulbs | <0.01 | <0.01 | | Study: 2011/1226626, 2011/1266275 Trial: G100306 Silva & Cardoso, 2011a, Kresoxim_106; Silva, 2011a Kresoxim_107 Max. frozen storage: 10 month |

| Leastion | Treatment | Application | Growth | DALT | Crop | Residue Four | nd (mg/kg) | | Depert/Trial No |
|---|--------------|-------------------|----------------------------------|--------------|-------|-------------------------|-------------------------|-------------------------|--|
| Location, Year (variety) | method | rate (g ai/ha) | stage at final application | | part | Kresoxim- methyl | 490M2 | 490M9 | -Report/Trial No., Reference Storage period |
| Brazil Jaboticabal/SP 2010 (Bela Vista) | Foliar spray | 4×70 | BBCH 47 | 7 | Bulbs | <0.01 | <0.01 | <0.01 | Study: 2011/1226626, 2011/1266275 Trial: G100307 Silva & Cardoso, 2011a, Kresoxim_106; Silva, 2011a Kresoxim_107 Max. frozen storage: 10 month |
| Brazil Santo Antonio de Posse/SP 2010 (Criolla) | Foliar spray | 4×70 | BBCH 49 | 0 7 14 | Bulbs | <0.01 <0.01 <0.01 | <0.01 <0.01 <0.01 | <0.01 <0.01 <0.01 | Study: 2011/1226626, 2011/1266275 Trial: G100308 Silva & Cardoso, 2011a, Kresoxim_106; Silva, 2011a Kresoxim_107 Max. frozen storage: 10 month |
| Brazil Ponta Grossa/ PR 2010 (Criolla) | Foliar spray | 4×70 | BBCH 46 | 0 7 14 | Bulbs | <0.01 <0.01 <0.01 | <0.01 <0.01 <0.01 | <0.01 <0.01 <0.01 | Study: 2011/1226626, 2011/1266275 Trial: G100309 Silva & Cardoso, 2011a, Kresoxim_106; Silva, 2011a Kresoxim_107 Max. frozen storage: 9 month |

Garlic

A total of four field trials were conducted with garlic in Brazil in the 2010/11 growing season (Silva & Cardoso, 2011b, Kresoxim_108; Silva, 2011b Kresoxim_109). Plants received 4 spray applications of kresoxim-methyl at nominal rates of 70 g ai/ha with a 7 day interval between applications. Samples of garlic were collected at 0, 7 and 14 days after the last application. Residues of kresoxim-methyl (as 490M1) and metabolites 490M2 and 490M9 were determined using method BASF method 350/4 (identical to method L0095/01 and SOP-PA.0295) with a limit of quantification of 0.01 mg/kg. Overall mean procedural recoveries for kresoxim-methyl (as 490M1) and metabolites 490M2 and 490M9 in garlic spiked at 0.01 and 1 mg/kg were 100% (n=12), 102% (n=12) and 95% (n=12), respectively.

Table 98 Residues of kresoxim-methyl in garlic following foliar treatment (cGAP: 4×70 g ai/ha; 7 days PHI) in Brazil

| Location, Year (variety) | Treatment method | rate | Growth stage at final application | DALT | | Residue Foun Kresoxim- methyl | d (mg/kg) 490M2 | 490M9 | Sum of parent + 490M9 | Report/Trial No., Reference Storage period |
|---|---------------------|------|---|--------------|-------|-------------------------------------|-------------------------|-------------------------|-----------------------------|--|
| Brazil Sto Antônio de Posse / SP 2010/11 (Pérola de cacador) | | 4×70 | BBCH 49 | 0 7 14 | Bulbs | <0.01 <0.01 <0.01 | <0.01 <0.01 <0.01 | <0.01 <0.01 <0.01 | <0.02 <0.02 <0.02 | Study: 2016/3004184, 2016/3004185 Trial: G100266 Silva & Cardoso, 2011b, Kresoxim_108; Silva, 2011b Kresoxim_109 Max. frozen storage: 11 month |

| | Treatment | Application | Growth stage | DALT | Crop | Residue Four | nd (mg/kg) | | | Dement/TeleLNIe |
|--|--------------|-------------------|-------------------------|--------------|-------|-------------------------|-------------------------|-------------------------|-----------------------------|--|
| Location, Year (variety) | method | rate (g ai/ha) | at final application | | part | Kresoxim- methyl | 490M2 | 490M9 | Sum of parent + 490M9 | Report/Trial No., Reference Storage period |
| Brazil Ponta Grossa / PR 2010/11 (Roxo) | Foliar spray | 4×70 | BBCH 48 | 0 7 14 | Bulbs | <0.01 <0.01 <0.01 | <0.01 <0.01 <0.01 | <0.01 <0.01 <0.01 | <0.02 <0.02 <0.02 | Study: 2016/3004184, 2016/3004185 Trial: G100267 Silva & Cardoso, 2011b, Kresoxim_108; Silva, 2011b Kresoxim_109 Max. frozen storage: 8 month |
| Brazil Londrina / PR 2010/11 (Lavinia rosa) | Foliar spray | 4×70 | BBCH 47 | 7 | Bulbs | <0.01 | <0.01 | <0.01 | <0.02 | Study: 2016/3004184, 2016/3004185 Trial: G100268 Silva & Cardoso, 2011b, Kresoxim_108; Silva, 2011b Kresoxim_109 Max. frozen storage: 11 month |
| Brazil Elias Fausto / SP 2010/11 (Ito) | Foliar spray | 4×70 | BBCH 47 | 7 | Bulbs | <0.01 | <0.01 | <0.01 | <0.02 | Study: 2016/3004184, 2016/3004185 Trial: G100710 Silva & Cardoso, 2011b, Kresoxim_108; Silva, 2011b Kresoxim_109 Max. frozen storage: <1 month |

Leek

A total of eight field trials were conducted with leek in Belgium, the Netherlands and northern France in the 2012 and 2013 growing seasons (Plier, 2013a, Kresoxim_099; Plier, 2013b, Kresoxim_100). Plants received 3 spray applications of kresoxim-methyl at nominal rates of 375 g ai/ha with a 9-11 day interval between applications. Samples of leek were collected at 0, 7, 13–14 and 20 days after the last application. Residues of kresoxim-methyl (as 490M1) and metabolites 490M2 and 490M9 were determined using method BASF method 350/4 (identical to method L0095/01 and SOP-PA.0295) with a limit of quantification of 0.01 mg/kg. Overall mean procedural recoveries for kresoxim-methyl (as 490M1) and metabolites 490M2 and 490M9 in leek spiked at 0.01–10 mg/kg were 92% (n=18), 91% (n=18) and 85% (n=18), respectively.

During the 1996/97 and 1997/98 growing season, a total of eight field trials were conducted with leek in Belgium, the Netherlands and Germany (Raunft, 1998a, Kresoxim_101; Raunft, 1998b, Kresoxim_102). Plants received 6 spray applications of kresoxim-methyl at nominal rates of 375 g ai/ha with a 14 day interval between applications. Samples of leek were collected at 0, 6–7, 13–17 and 20–22 days after the last application. Residues of kresoxim-methyl (as 490M1) and metabolites 490M2 and 490M9 were determined using method BASF method 350/3 with a limit of quantification of 0.05 mg/kg. Overall mean procedural recoveries for kresoxim-methyl (as 490M1) and metabolites 490M2 and 490M9 in leek spiked at 0.05 and 5 mg/kg were 81% (n=26), 85% (n=26) and 89% (n=26), respectively.

| | Treatment | Application | Growth stage | DALT | Crop | Residue Fou | nd (ma/ka) | | | I |
|-------------------|--------------|-------------|--------------|---------|---------------|-------------|----------------|----------------|-------------------|---------------------------------|
| Location, | method | rate | at final | DALI | part | Kresoxim- | 490M2 | 490M9 | Sum of | -Report/Trial No., Reference |
| Year (variety) | | (g ai/ha) | application | | | methyl | | | parent + 490M9 | Storage period |
| Belgium | Foliar spray | 3×375 | BBCH 45-47 | 0 | Whole | 4.2 | 0.011 | 0.024 | 4.2 | Study: |
| Limburg | | | | 7 | plant | 3.1 | 0.012 | 0.026 | 3.1 | 2013/1037960 |
| Halen | | | | 14 | (w/o | 2.7 | 0.023 | 0.044 | 2.7 | Trial: L120661 |
| 2012 | | | | 20 | root) | 2.4 | 0.026 | 0.055 | 2.5 | Plier, 2013a, |
| (Benton) | | | | | | | | | | Kresoxim_099 |
| | | | | | | | | | | Max. frozen |
| | | | | | | | | | | storage: 7 month |
| The Netherlands | Foliar spray | 3×375 | BBCH 45-47 | 0 | Whole | 5.0 | <0.01 | 0.014 | 5.0 | Study: |
| Limburg | | | | 7 | plant | 5.0 | < 0.01 | 0.020 | 5.0 | 2013/1037960 |
| Tienray | | | | 14 | (w/o | 4.5 | 0.015 | 0.038 | 4.5 | Trial: L120662 |
| 2012 | | | | 20 | root) | 3.9 | 0.018 | 0.044 | 3.9 | Plier, 2013a, |
| (Poulton) | | | | | | | | | | Kresoxim_099 |
| | | | | | | | | | | Max. frozen |
| The Netherlands | Eolior coray | 3×375 | BBCH 45-47 | 0 | Whole | 4.0 | 0.012 | 0.026 | 4.0 | storage: 7 month Study: |
| Limburg | rullal splay | 3×375 | DDCH 43-47 | 7 | plant | 4.0 3.1 | 0.012 | 0.028 | 4.0 3.1 | 2013/1037960 |
| Castenray | | | | , 14 | (w/o | 3.3 | 0.013 | 0.034 | 3.3 | Trial: L120663 |
| 2012 | | | | 20 | root) | 2.5 | 0.017 | 0.035 | 2.6 | Plier, 2013a, |
| (Kenton) | | | | 20 | 1000 | 2.5 | 0.020 | 0.045 | 2.0 | Kresoxim 099 |
| (| | | | | | | | | | Max. frozen |
| | | | | | | | | | | storage: 7 month |
| France (North) | Foliar spray | 3×375 | BBCH 47 | 0 | Whole | 5.5 | <0.01 | 0.021 | 5.5 | Study: |
| Limburg | | | | 7 | plant | 3.9 | 0.012 | 0.034 | 3.9 | 2013/1037960 |
| Chouzé sur | | | | 13 | (w/o | 3.1 | 0.013 | 0.031 | 3.1 | Trial: L120664 |
| Loire | | | | 20 | root) | 2.2 | 0.015 | 0.033 | 2.2 | Plier, 2013a, |
| 2012 | | | | | | | | | | Kresoxim_099 |
| (Bluebell) | | | | | | | | | | Max. frozen |
| | | | | | _ | | _ | | | storage: 7 month |
| Belgium | Foliar spray | 3×375 | BBCH 47 | 0 | Whole | 5.4 | 0.017 | 0.043 | 5.4 | Study: |
| Kortenaken | | | | 7 | plant | 3.9 | 0.020 | 0.041 | 3.9 | 2013/1292272 |
| Waanrode | | | | 14 | (w/o | 3.4 | 0.021 | 0.031 | 3.4 | Trial: L130058 |
| 2013 (Harston) | | | | 20 | root) | 1.8 | 0.033 | 0.061 | 1.9 | Plier, 2013b, |
| (Harston) | | | | | | | | | | Kresoxim_100 Max. frozen |
| | | | | | | | | | | storage: 3 month |
| The Netherlands | Foliar sprav | 3×375 | BBCH 47 | 0 | Whole | 6.7 | 0.014 | 0.032 | 6.7 | Study: |
| Limburg | i onu spruy | 0.070 | bbon n | 5 7 | plant | 4.4 | 0.020 | 0.044 | 4.4 | 2013/1292272 |
| Castenray | | | | , 14 | (w/o | 3.1 | 0.022 | 0.041 | 3.1 | Trial: L130059 |
| 2013 | | | | 20 | root) | 2.1 | 0.031 | 0.044 | 2.1 | Plier, 2013b, |
| (Vitaton) | | | | | , | | | | | Kresoxim_100 |
| | | | | | | | | | | Max. frozen |
| | | | | | | | | | | storage: 3 month |
| The Netherlands | Foliar spray | 3×375 | BBCH 47 | 0 | Whole | 5.3 | 0.020 | 0.038 | 5.3 | Study: |
| Limburg | | | | 7 | plant | 4.2 | 0.022 | 0.034 | 4.2 | 2013/1292272 |
| Meterik | | | | 14 | (w/o | 2.8 | 0.025 | 0.038 | 2.8 | Trial: L130060 |
| 2013 | | | 1 | 20 | root) | 1.5 | 0.028 | 0.065 | 1.6 | Plier, 2013b, |
| (Vitaton) | | | | | | | 1 | | | Kresoxim_100 |
| | | | | | | | 1 | | | Max. frozen storage: 3 month |
| Polaium | Eolior oprov | 2275 | | 0 | Whalo | F 1 | 0.013 | 0.024 | F 1 | |
| Belgium | Foliar spray | 3×375 | BBCH 47 | 0 | Whole | 5.1 4 9 | | 0.036 | 5.1 | Study: |
| Limburg Halen | | | | / 13 | plant (w/o | 4.8 3.3 | 0.022 0.025 | 0.052 0.032 | 4.9 3.3 | 2013/1292272 Trial: L130061 |
| 2013 | | | | 20 | (w/o root) | 3.3 2.0 | 0.025 | 0.032 | 3.3 2.1 | Plier, 2013b, |
| (Harston) | | | | 20 | 1001) | 2.0 | 0.045 | 0.005 | 2.1 | Kresoxim_100 |
| | | | | | | | 1 | | | Max. frozen |
| | 1 | | 1 | 1 | 1 | 1 | 1 | 1 | 1 | storage: 3 month |

Table 99 Residues of kresoxim-methyl in leek following foliar treatment (cGAP: 3×375 g ai/ha; 14 day PHI)

| | Treatment | Application | Growth stage | DALT | Crop | Residue Fou | ind (mg/kg |) | | |
|--|--------------|-------------------|----------------------|--------------------|---------------------------------|---------------------------|------------------------------------|--------------------------------|-----------------------------|--|
| Location, Year (variety) | method | rate (g ai/ha) | at final application | | part | Kresoxim- methyl | 490M2 | 490M9 | Sum of parent + 490M9 | Report/Trial No., Reference Storage period |
| The Netherlands Limburg Ottersum 1996/97 (Arkansas) | Foliar spray | 6×375 | BBCH 49 | 0 7 14 22 | Whole plant (w/o root) | 3.3 1.7 2.5 2.0 | < 0.05 < 0.05 0.06 0.05 | 0.13 0.11 0.15 0.09 | 3.4 1.8 2.7 2.1 | Study: 1998/10500 Trial: AGR/19/96 Raunft, E., et al., 1998a, Kresoxim_101 Max. frozen storage: 11 month |
| The Netherlands Limburg Siebengewald 1996/97 (Porinto) | Foliar spray | 6×375 | BBCH 47 | 0 7 14 22 | Whole plant (w/o root) | 2.8 2.1 2.4 2.6 | < 0.05 < 0.05 0.07 0.07 | 0.07 0.07 0.15 0.14 | 2.9 2.2 2.6 2.7 | Study: 1998/10500 Trial: AGR/20/96 Raunft, E., et al., 1998a, Kresoxim_101 Max. frozen storage: 11 month |
| Belgium Brabant Kortenaken 1996/97 (Arkansas) | Foliar spray | 6×375 | BBCH 45 | 0 6 13 20 | Whole plant (w/o root) | 2.3 1.7 1.6 1.7 | 0.05 0.07 0.08 0.07 | 0.12 0.31 0.17 0.13 | 2.4 2.0 1.8 1.8 | Study: 1998/10500 Trial: AGR/21/96 Raunft, E., et al., 1998a, Kresoxim_101 Max. frozen storage: 11 month |
| Germany Hochdorf- Assenheim 1996/97 (Alaska) | Foliar spray | 6×375 | BBCH 49 | 0 7 17 21 | Whole plant (w/o root) | 6.2 2.3 2.8 2.4 | 0.06 < 0.05 < 0.05 < 0.05 | 0.13 0.10 0.09 0.08 | 6.3 2.4 2.9 2.5 | Study: 1998/10500 Trial: DU2/15/96 Raunft, E., et al., 1998a, Kresoxim_101 Max. frozen storage: 11 month |
| Belgium Brabant Kortenaken 1997/98 (Vrizo) | Foliar spray | 6×375 | BBCH 45 | 0 7 13 20 | Whole plant (w/o root) | 4.6 2.0 1.5 0.67 | 0.05 < 0.05 < 0.05 < 0.05 | 0.10 0.09 0.10 0.06 | 4.7 2.1 1.8 0.7 | Study: 1998/10501 Trial: AGR/05/97 Raunft, E., et al., 1998b, Kresoxim_102 Max. frozen storage: 2 month |
| The Netherlands Limburg Ottersum 1997/98 (Suprina) | Foliar spray | 6×375 | BBCH 45 | 0 7 13 20 | Whole plant (w/o root) | 5.7 0.57 2.8 2.7 | 0.06 < 0.05 0.05 0.09 | 0.16 < 0.05 0.11 0.16 | 5.9 0.62 2.9 2.9 | Study: 1998/10501 Trial: AGR/06/97 Raunft, E., et al., 1998b, Kresoxim_102 Max. frozen storage: 2 month |
| The Netherlands Limburg Siebengewald 1997/98 (Porenta) | Foliar spray | 6×375 | BBCH 45 | 0 7 13 20 | Whole plant (w/o root) | 0.94 1.4 2.3 1.4 | < 0.05 < 0.05 0.05 0.06 | < 0.05 0.06 0.12 0.12 | 0.99 1.5 2.4 1.5 | Study: 1998/10501 Trial: AGR/07/97 Raunft, E., et al., 1998b, Kresoxim_102 Max. frozen storage: 2 month |

| | Treatment | Application | Growth stage | DALT | Crop | Residue Fou | nd (mg/kg) | | | Report/Trial No., |
|--|--------------|-------------------|-------------------------|--------------------|---------------------------------|--------------------------|------------------------------|------------------------------|-----------------------------|--|
| Location, Year (variety) | method | rate (g ai/ha) | at final application | | part | Kresoxim- methyl | 490M2 | 490M9 | Sum of parent + 490M9 | Reference Storage period |
| Germany Hochdorf 1997/98 (Alaska) | Foliar spray | 6×375 | BBCH 49 | 0 7 14 21 | Whole plant (w/o root) | 7.0 5.3 2.6 2.7 | 0.05 0.06 0.06 0.09 | 0.11 0.12 0.11 0.14 | 7.1 5.4 2.7 2.8 | Study: 1998/10501 Trial: DU2/01/97 Raunft, E., et al., 1998b, Kresoxim_102 Max. frozen storage: 2 month |

Fruiting vegetables, Cucurbits – Cucumber and Summer Squashes

Cucumber

A total of eight field trials were conducted with cucumber in the United States of America in the 1997/98 growing season (Wofford & Riley, 1998, Kresoxim_110). Plants received 6 spray applications of kresoxim-methyl at nominal rates of 196 g ai/ha with a 7±1 day interval between applications. Exceptions occurred in trial RCN 97026, where applications 1 and 2 were made at 168 g ai/ha, application 3 at 252 g ai/ha and the remaining applications at 196 kg ai/ha. Samples of cucumber were collected at 0, 3 and 7 days after the last application. Residues of kresoxim-methyl (as 490M1) and metabolites 490M2 and 490M9 were determined using method BASF method 350/3 with a limit of quantification of 0.05 mg/kg. Overall mean procedural recoveries for kresoxim-methyl (as 490M1) and metabolites 490M2 and 490M9 in cucumber spiked at 0.05 and 1 mg/kg were 87% (n=19), 90% (n=19) and 81% (n=19), respectively.

| Table 100 Residues of kresoxim-meth | vl in cucumber following foliar treatme | nt (cGAP: 4×168 g ai/ha; 0 days PHI) in the USA |
|-------------------------------------|---|---|
| | | |

| Location | Treatment | Application | Growth | DALT | Crop | Residue Foun | d (mg/kg) ^a | | | Depert/Trial Na |
|--|--------------|-------------------|--|-------------|-------|----------------------------|----------------------------|----------------------------|-----------------------------|---|
| Location, Year (variety) | method | rate (g ai/ha) | stage at final application | | part | Kresoxim- methyl | 490M2 | 490M9 | Sum of parent + 490M9 | Report/Trial No., Reference Storage period |
| United States Georgia Mason County 1997/98 (Straight Eight) | Foliar spray | 6×196 | 12-16 inch tall plants with fruit | 0 3 7 | Fruit | < 0.05 < 0.05 < 0.05 | < 0.05 < 0.05 < 0.05 | < 0.05 < 0.05 < 0.05 | <0.10 <0.10 <0.10 | Study: 1998/5121 Trial: 97021 Wofford & Riley, 1998, Kresoxim_110 Max. frozen storage: 10 month |
| United States South Carolina Barnwell County 1997/98 (Pointsett 76) | Foliar spray | 6×196 | 12-14 inch tall plants at bloom/ mature fruit | 0 | Fruit | 0.06 | < 0.05 | < 0.05 | 0.11 | Study: 1998/5121 Trial: 97022 Wofford & Riley, 1998, Kresoxim_110 Max. frozen storage: 10 month |
| United States Georgia Tift County 1997/98 (Straight Eight) | Foliar spray | 6×196 | 13 inch tall plants with mature fruit | 3 | Fruit | < 0.05 < 0.05 < 0.05 | < 0.05 < 0.05 < 0.05 | < 0.05 < 0.05 < 0.05 | <0.10 <0.10 <0.10 | Study: 1998/5121 Trial: 97023 Wofford & Riley, 1998, Kresoxim_110 Max. frozen storage: 10 month |

| | Treatment | Application | Growth | DALT | Crop | Residue Fou | nd (mg/kg) | а | | |
|---|--------------|-------------------------|---|-------------------------|-------|--|--|--|---------------------------------------|---|
| Location, Year (variety) | method | rate (g ai/ha) | stage at final application | | part | Kresoxim- methyl | 490M2 | 490M9 | Sum of parent + 490M9 | Report/Trial No., Reference Storage period |
| United States Florida Seminole County 1997/98 (Pointsett 76) | Foliar spray | 2×168 1×252 3×196 | 12 inch tall mature plants | 0 3 7 | Fruit | < 0.05 < 0.05 < 0.05 | < 0.05 < 0.05 < 0.05 | < 0.05 < 0.05 < 0.05 | <0.10 <0.10 <0.10 | Study: 1998/5121 Trial: 97026 Wofford & Riley, 1998, Kresoxim_110 Max. frozen storage: 10 month |
| United States Michigan Ottawa County 1997/98 (Marketmore) | | 6×196 | 12-15 inch tall plants with mature fruit | 0 3 7 | Fruit | < 0.05 < 0.05 < 0.05 | < 0.05 < 0.05 < 0.05 | < 0.05 < 0.05 < 0.05 | <0.10 <0.10 <0.10 | Study: 1998/5121 Trial: 97028 Wofford & Riley, 1998, Kresoxim_110 Max. frozen storage: 10 month |
| United States Wisconsin Pepin County 1997/98 (Lucky Strike) | Foliar spray | 6×196 | 15-20 inch tall plants fruit formation | 0 | Fruit | 0.06 | < 0.05 | < 0.05 | 0.11 | Study: 1998/5121 Trial: 97029 Wofford & Riley, 1998, Kresoxim_110 Max. frozen storage: 10 month |
| United States Texas Uvalde County 1997/98 (SMR 58) | Foliar spray | 6×196 | 12 inch tall with mature/ past mature fruit | 0 | Fruit | 0.06 | < 0.05 | < 0.05 | 0.11 | Study: 1998/5121 Trial: 97032 Wofford & Riley, 1998, Kresoxim_110 Max. frozen storage: 10 month |
| United States California Tulare County 1997/98 (Pointsett 76) | Foliar spray | 6×196 | 4 inch tall plants at fruit set/ maturity | 0 3 7 10 15 | Fruit | 0.11 0.11 0.08 < 0.05 < 0.05 | < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 | < 0.05 0.06 0.07 < 0.05 0.05 | 0.16 0.17 0.15 <0.10 0.10 | Study: 1998/5121 Trial: 97395 Wofford & Riley, 1998, Kresoxim_110 Max. frozen storage: 10 month |

^a Mean of duplicate analysis.

During the 1995 and 1996 growing season, a total of eight field trials were conducted with cucumber in Spain under greenhouse conditions (Fuchs & Rabe, 1997, Kresoxim_111). Plants received 4 spray applications of kresoxim-methyl at nominal rates of 100 g ai/ha or 150 g ai/ha with about 10 day intervals between applications. Samples of cucumber were collected at 0, 3–5, 7 and 13–14 days after the last application. Residues of kresoxim-methyl (as 490M1) and metabolites 490M2 and 490M9 were determined using method BASF method 350/3 with a limit of quantification of 0.05 mg/kg. Overall mean procedural recoveries for kresoxim-methyl (as 490M1) and metabolites 490M2 and 490M9 in cucumber spiked at 0.05 and 5 mg/kg were 93% (n=10), 91% (n=10) and 76% (n=10), respectively.

| | Treatment | Application | Growth | DALT | Crop | Residue Four | nd (ma/ka) | | | |
|---|-----------------|-------------------|----------------------------------|-------------------|-------|--------------------------------------|--------------------------------------|--------------------------------------|--------------------------------------|--|
| Location, Year (variety)ª | method | rate (g ai/ha) | stage at final application | DALI | part | Kresoxim- methyl | 490M2 | 490M9 | Sum of parent + 490M9 | Report/Trial No., Reference Storage period |
| Spain Cadiz La Algaida 1995 (PS) | Foliar spray | 4×100 | BBCH69-87 | 0 5 7 13 | Fruit | < 0.05 < 0.05 < 0.05 < 0.05 | < 0.05 < 0.05 < 0.05 < 0.05 | < 0.05 < 0.05 < 0.05 < 0.05 | < 0.10 < 0.10 < 0.10 < 0.10 | Study: 1997/10152 Trial: ALO/13/95 Fuchs & Rabe, 1997, Kresoxim_111 Max. frozen storage: 13 month |
| Spain Cadiz La Algaida 1995 (Dasher II) | Foliar spray | 4×100 | BBCH69-85 | 0 5 7 13 | Fruit | < 0.05 < 0.05 < 0.05 < 0.05 | < 0.05 < 0.05 < 0.05 < 0.05 | < 0.05 < 0.05 < 0.05 < 0.05 | < 0.10 < 0.10 < 0.10 < 0.10 | Study: 1997/10152 Trial: ALO/14/95 Fuchs & Rabe, 1997, Kresoxim_111 Max. frozen storage: 13 month |
| Spain Cadiz La Algaida 1995 (PS) | Foliar spray | 4×100 | BBCH69-88 | 0 4 7 14 | Fruit | < 0.05 < 0.05 < 0.05 < 0.05 | < 0.05 < 0.05 < 0.05 < 0.05 | < 0.05 < 0.05 < 0.05 < 0.05 | < 0.10 < 0.10 < 0.10 < 0.10 | Study: 1997/10152 Trial: ALO/15/95 Fuchs & Rabe, 1997, Kresoxim_111 Max. frozen storage: 13 month |
| Spain Cadiz La Algaida 1995 (Dasher II) | Foliar spray | 4×100 | BBCH69-86 | 0 4 7 14 | Fruit | 0.46 < 0.05 < 0.05 < 0.05 | < 0.05 < 0.05 < 0.05 < 0.05 | < 0.05 < 0.05 < 0.05 < 0.05 | 0.51 < 0.10 < 0.10 < 0.10 | Study: 1997/10152 Trial: ALO/16/95 Fuchs & Rabe, 1997, Kresoxim_111 Max. frozen storage: 13 month |
| Spain Sevilla Alcala de Guadiara 1996 (Darina) | Foliar spray | 4×150 | BBCH69-75 | 0 3 7 14 | Fruit | < 0.05 < 0.05 < 0.05 < 0.05 | < 0.05 < 0.05 < 0.05 < 0.05 | < 0.05 < 0.05 < 0.05 < 0.05 | < 0.10 < 0.10 < 0.10 < 0.10 | Study: 1997/10152 Trial: ALO/17/95 Fuchs & Rabe, 1997, Kresoxim_111 Max. frozen storage: 13 month |

Table 101 Residues of kresoxim-methyl in cucumber following foliar treatment (cGAP: 4×168 g ai/ha; 0 days PHI) in Spain under greenhouse conditions

806

| 1 | Treatment | Application | Growth | DALT | Crop | Residue Fou | nd (mg/kg) | | | |
|---|-----------------|-------------------|----------------------------------|-------------------|-------|--------------------------------------|--------------------------------------|--------------------------------------|--------------------------------------|--|
| Location, Year (variety)ª | method | rate (g ai/ha) | stage at final application | | part | Kresoxim- methyl | 490M2 | 490M9 | Sum of parent + 490M9 | Report/Trial No., Reference Storage period |
| Spain Sevilla Alcala de Guadiara 1996 (Darina) | Foliar spray | 4×150 | BBCH69-76 | 0 4 7 14 | Fruit | < 0.05 < 0.05 < 0.05 < 0.05 | < 0.05 < 0.05 < 0.05 < 0.05 | < 0.05 < 0.05 < 0.05 < 0.05 | < 0.10 < 0.10 < 0.10 < 0.10 | Study: 1997/10152 Trial: ALO/18/95 Fuchs & Rabe, 1997, Kresoxim_111 Max. frozen storage: 13 month |
| Spain Sevilla Palacios 1996 (Darina) | Foliar spray | 4×150 | BBCH69-75 | 0 3 7 14 | Fruit | < 0.05 < 0.05 < 0.05 < 0.05 | < 0.05 < 0.05 < 0.05 < 0.05 | < 0.05 < 0.05 < 0.05 < 0.05 | < 0.10 < 0.10 < 0.10 < 0.10 | Study: 1997/10152 Trial: ALO/19/95 Fuchs & Rabe, 1997, Kresoxim_111 Max. frozen storage: 13 month |
| Spain Sevilla Palacios 1996 (Dasher II) | Foliar spray | 4×150 | BBCH69-75 | 0 3 7 14 | Fruit | < 0.05 < 0.05 < 0.05 < 0.05 | < 0.05 < 0.05 < 0.05 < 0.05 | < 0.05 < 0.05 < 0.05 < 0.05 | < 0.10 < 0.10 < 0.10 < 0.10 | Study: 1997/10152 Trial: ALO/20/95 Fuchs & Rabe, 1997, Kresoxim_111 Max. frozen storage: 13 month |

Gherkin

A total of four field trials were conducted with gherkin in the Germany in the 2000 growing season (Scharm, 2001, Kresoxim_112). Plants received 4 spray applications of kresoxim-methyl at nominal rates of 150 g ai/ha with 6–7 day intervals between applications. Samples of gherkin were collected at 0 and 3 days after the last application. Residues of kresoxim-methyl (as 490M1) were determined using method BASF method 445/0 with a limit of quantification of 0.05 mg/kg. Overall mean procedural recoveries for kresoxim-methyl (as 490M1) in gherkin spiked at 0.05 and 0.5 mg/kg was 88% (n=10).

Table 102 Residues of kresoxim-methyl in gherkins following foliar treatment (cGAP: 4×168 g ai/ha; 0 days PHI)

| Location, Year (variety) | Treatment method | Application rate (g ai/ha) | Growth stage at final application | DALT | Crop part | Residue Fou Kresoxim- methyl | ind (mg/kg) ^a 490M2 | 490M9 | Report/Trial No., Reference Storage period |
|--|---------------------|----------------------------------|--|--------|--------------|------------------------------------|-----------------------------------|-------|--|
| Germany Mühlhausen 2000 (Melody) | Foliar spray | 4×150 | BBCH 82 | 0 3 | Fruit | 0.16 < 0.05 | - | - | Study: 2001/1000942 Trial: AT-00/024-1 Scharm, 2001, Kresoxim_112 Max. frozen storage: 9 month |
| Germany Riedstadt- Crumstadt 2000 (Pontomac F1) | Foliar spray | 4×150 | BBCH 85 | 03 | Fruit | < 0.05 < 0.05 | - | - | Study: 2001/1000942 Trial: AT-00/024-2 Scharm, 2001, Kresoxim_112 Max. frozen storage: 9 month |

| Location, Year (variety) | Treatment method | Application rate (g ai/ha) | Growth stage at final application | DALT | Crop part | Residue Fou Kresoxim- methyl | ind (mg/kg) ^a 490M2 | 490M9 | Report/Trial No., Reference Storage period |
|--|---------------------|----------------------------------|--|--------|--------------|------------------------------------|-----------------------------------|-------|--|
| Germany Gernsheim 2000 (Musica) | Foliar spray | 4×150 | BBCH 87 | 0 3 | Fruit | 0.059 < 0.05 | - | - | Study: 2001/1000942 Trial: AT-00/024-3 Scharm, 2001, Kresoxim_112 Max. frozen storage: 9 month |
| Germany Eich 2000 (Mauvin) | Foliar spray | 4×150 | BBCH 86 | 0 3 | Fruit | < 0.05 < 0.05 | - | - | Study: 2001/1000942 Trial: AT-00/024-4 Scharm, 2001, Kresoxim_112 Max. frozen storage: 9 month |

Summer squash

A total of five field trials were conducted with summer squash in the United States in the 1997/98 growing season (Wofford & Riley, 1998, Kresoxim_110). Plants received 6 spray applications of kresoxim-methyl at nominal rates of 196 g ai/ha with a 7±1 day interval between applications. Exceptions occurred in trial RCN 97027, where applications 1 and 2 were made at 168 g ai/ha, application 3 at 252 g ai/ha and the remaining three applications at 196 kg ai/ha. Samples of summer squash were collected at 0, 3 and 7 days after the last application. Residues of kresoxim-methyl (as 490M1) and metabolites 490M2 and 490M9 were determined using method BASF method 350/3 with a limit of quantification of 0.05 mg/kg. Overall mean procedural recoveries for kresoxim-methyl (as 490M1) and metabolites 490M2 and 490M9 in summer squash spiked at 0.05 and 1 mg/kg were 86% (n=6), 88% (n=8) and 82% (n=8), respectively.

| | Treatment | Application | Growth stage | DALT | Crop | Residue Fou | nd (mg/kg) | | | |
|--|--------------|-------------------------|---|-------------|-------|----------------------------|----------------------------|----------------------------|-----------------------------|--|
| Location, Year (variety) | method | rate (g ai/ha) | at final application | | part | Kresoxim- methyl | 490M2 | 490M9 | Sum of parent + 490M9 | Report/Trial No., Reference Storage period |
| United States Pennsylvania Lehigh County 1997/98 (Seneca Supreme F1) | Foliar spray | 6×196 | 24-30 inch tall plants at fruiting/ bloom | 0 | Fruit | <0.05 | < 0.05 | < 0.05 | <0.10 | Study: 1998/5121 Trial: 97020 Wofford & Riley, 1998, Kresoxim_110 Max. frozen storage: 10 month |
| United States North Carolina Sampson County 1997/98 (Straightneck Early) | Foliar spray | 6×196 | 24 inch tall plants with fruit | 0 3 7 | Fruit | < 0.05 < 0.05 < 0.05 | < 0.05 < 0.05 < 0.05 | < 0.05 < 0.05 < 0.05 | <0.10 <0.10 <0.10 | Study: 1998/5121 Trial: 97025 Wofford & Riley, 1998, Kresoxim_110 Max. frozen storage: 10 month |
| United States Florida Seminole County 1997/98 (Crookneck) | Foliar spray | 2×168 1×252 3×196 | 2 foot tall mature plants | 0 3 7 | Fruit | < 0.05 < 0.05 < 0.05 | < 0.05 < 0.05 < 0.05 | < 0.05 < 0.05 < 0.05 | <0.10 <0.10 <0.10 | Study: 1998/5121 Trial: 97027 Wofford & Riley, 1998, Kresoxim_110 Max. frozen storage: 10 month |
| United States Wisconsin Pepin County 1997/98 (Monet) | Foliar spray | 6×196 | 20-30 inch tall plants at bloom/ fruit formation | 0 | Fruit | <0.05 | < 0.05 | < 0.05 | <0.10 | Study: 1998/5121 Trial: 97031 Wofford & Riley, 1998, Kresoxim_110 Max. frozen storage: 10 month |

Table 103 Residues of kresoxim-methyl in summer squash following foliar treatment (cGAP: 4×168 g ai/ha; 0 days PHI)

| Legation | Treatment | Application | Growth stage | DALT | Crop | Residue Found | d (mg/kg) | | | Dement/Trial No. |
|-----------------------------|--------------|-------------|-------------------------|------|-------|---------------------|-----------|--------|--------------------|--|
| Location, Year (variety) | | | at final application | | part | Kresoxim- methyl | 490M2 | 490M9 | Sum of parent + | Report/Trial No., Reference Storage period |
| United States | Foliar spray | 6×196 | 25 inch tall | 0 | Fruit | 0.22 | < 0.05 | < 0.05 | 0.27 | Study: 1998/5121 |
| California | | | plants with | | | | | | | Trial: 97038 |
| Fresno County | | | mature fruit | | | | | | | Wofford & Riley, |
| 1997/98 | | | | | | | | | | 1998, Kresoxim_110 |
| (Crookneck, | | | | | | | | | | Max. frozen |
| Sipersett F1) | | | | | | | | | | storage: 10 month |

Fruiting vegetables, Cucurbits – Melons, Pumpkins and Winter Squashes

Melon

A total of six field trials were conducted with melon in Northern France and Spain in the 1996 (Schulz, 1998b, Kresoxim_114) and 1997 (Schulz, 1998a, Kresoxim_113; Rabe, 1998, Kresoxim_116) growing seasons. Plants received 4 spray applications of kresoxim-methyl at nominal rates of 150 g ai/ha with interval between applications of about 10 days. Samples of melon were collected at 0, 4–5, 6–9 and 13–15 days after the last application. Residues of kresoxim-methyl (as 490M1) and metabolites 490M2 and 490M9 were determined using method BASF method 350/3 with a limit of quantification of 0.05 mg/kg. Overall mean procedural recoveries for kresoxim-methyl (as 490M1) and metabolites 490M2 and 490M9 in melon spiked at 0.05 and 5 mg/kg were 90% (n=8), 99% (n=8) and 97% (n=8), respectively.

| Table 104 Residues of kresoxim-methy | I in melon following foliar treatment | (cGAP: 4×168 g ai/ha: 0 days PHI) |
|--------------------------------------|---------------------------------------|-----------------------------------|
| | | |

| | Treatment | Application | Growth stage | DALT | Crop | Residue Fou | nd (mg/kg) |) | | Depert/Trial No. |
|---|--------------|-------------------|-------------------------|-------------------|-------|----------------------------------|--------------------------------------|--------------------------------------|---------------------------------|--|
| Location, Year (variety)ª | method | rate (g ai/ha) | at final application | | part | Kresoxim- methyl | 490M2 | 490M9 | Sum of parent + 490M9 | Report/Trial No., Reference Storage period |
| Northern France Esvres/Indre 1996 (Manago) | Foliar spray | 4×150 | BBCH 81 | 0 4 7 14 | Fruit | 0.06 0.06 < 0.05 < 0.05 | < 0.05 < 0.05 < 0.05 < 0.05 | < 0.05 < 0.05 < 0.05 < 0.05 | 0.11 0.11 <0.10 <0.10 | Study: 1998/10394 Trial: X 96 62 51 Schulz, 1998b, Kresoxim_114 Max. frozen storage: 16 month |
| Southern France La Reole 1996 (Manto) | Foliar spray | 4×150 | BBCH 85 | 0 4 6 13 | Fruit | 0.19 0.08 0.09 0.06 | < 0.05 < 0.05 < 0.05 < 0.05 | < 0.05 < 0.05 < 0.05 0.06 | 0.24 0.13 0.14 0.12 | Study: 1998/10394 Trial: X 96 62 52 Schulz, 1998b, Kresoxim_114 Max. frozen storage: 16 month |
| Northern France La Roche Clermault 1998 (Dalton) | Foliar spray | 4×150 | Mature | 0 4 7 14 | Fruit | 0.20 0.16 0.08 <0.05 | < 0.05 < 0.05 < 0.05 < 0.05 | < 0.05 < 0.05 < 0.05 < 0.05 | 0.25 0.21 0.13 <0.10 | Study: 1998/10390 Trial: A97011 Schulz, 1998a, Kresoxim_113 Max. frozen storage: 4 month |
| Northern France Leméré 1998 (Exel) | Foliar spray | 4×150 | BBCH 83 | 0 5 9 15 | Fruit | <0.05 0.05 <0.05 <0.05 | < 0.05 < 0.05 < 0.05 < 0.05 | < 0.05 < 0.05 < 0.05 < 0.05 | <0.10 0.10 <0.10 <0.10 | Study: 1998/10390 Trial: A97020 Schulz, 1998a, Kresoxim_113 Max. frozen storage: 4 month |
| Spain Cadiz Sanlucar de Bda 1997 (Makthimon) | Foliar spray | 4×150 | BBCH 89 | 4 7 | Fruit | 0.13 0.06 | < 0.05 < 0.05 | < 0.05 < 0.05 | 0.18 0.11 | Study: 1998/10218 Trial: ME/97/01 Rabe, 1998, Kresoxim_116 Max. frozen storage: 3 month |

| | Treatment | Application | Growth stage | DALT | Crop | Residue Found | d (mg/kg) | | | Dement/Trial No. |
|------------------------------|--------------|-------------------|-------------------------|------|-------|---------------------|-----------|--------|--------------------|--|
| Location, Year (variety)ª | method | rate (g ai/ha) | at final application | | | Kresoxim- methyl | 490M2 | 490M9 | Sum of narent + | Report/Trial No., Reference Storage period |
| Spain | Foliar spray | 4×150 | BBCH 89 | 4 | Fruit | 0.07 | < 0.05 | < 0.05 | 0.12 | Study: 1998/10218 |
| Cadiz | | | | 7 | | 0.07 | < 0.05 | 0.10 | 0.17 | Trial: ME/97/02 |
| Sanlucar de | | | | | | | | | | Rabe, 1998, |
| Bda | | | | | | | | | | Kresoxim_116 |
| 1997 | | | | | | | | | | Max. frozen |
| (Melina) | | | | | | | | | | storage: 3 month |

^a Trials performed at the same location and year could not be considered as independent. Hence, only the highest residue was considered in these trials.

Additionally, a total of six field trials were conducted with cantaloupe in the United States of America in the 1997/98 growing season (Wofford & Riley, 1998, Kresoxim_110). Plants received 6 spray applications of kresoxim-methyl at nominal rates of 196 g ai/ha with a 7±1 day interval between applications. Exceptions occurred in trial 97035 and 97036, where the first spray was applied at 168 g ai/ha, the second spray was applied at 224 g ai/ha and the remaining four sprays were applied at 196 kg ai/ha. Samples of cantaloupe were collected at 0, 3 and 7 days after the last application. Residues of kresoxim-methyl (as 490M1) and metabolites 490M2 and 490M9 were determined using method BASF method 350/3 with a limit of quantification of 0.05 mg/kg. Overall mean procedural recoveries for kresoxim-methyl (as 490M1) and metabolites 490M2 and 490M9 in cantaloupe spiked at 0.05 and 1 mg/kg were 88% (n=4), 99% (n=4) and 99% (n=4), respectively.

| Location. | Treatment | Application | Growth | DALT | Crop | | | | | |
|-------------------|-----------|-------------|----------------------|------|-------|-----------|--------|--------|--------|---------------------------|
| LOCATION, | method | rate | stage at | | part | Kresoxim- | 490M2 | 490M9 | Sum | Report/Trial No., |
| Year | | (g ai/ha) | final | | | methyl | | | as | Reference |
| (variety) | | | application | | | | | | parent | Storage period |
| . ,, | | | | | | | | | equiv. | |
| United | Foliar | 6×196 | 8 inch tall | 0 | Fruit | 0.12 | < 0.05 | < 0.05 | 0.22 | Study: |
| States | spray | | plants at | 3 | | 0.07 | < 0.05 | < 0.05 | 0.17 | 1998/5121 |
| Alabama | | | flowering/ | 7 | | < 0.05 | < 0.05 | < 0.05 | <0.15 | Trial: 97024 |
| Henry | | | fruit | | | | | | | Wofford & Riley, 1998, |
| County 1997/98 | | | maturity | | | | | | | Kresoxim 110 |
| (Athena) | | | | | | | | | | Max. frozen |
| (Athena) | | | | | | | | | | storage: 10 |
| | | | | | | | | | | month |
| United | Foliar | 6×196 | 10-14 inch | 0 | Fruit | 0.08 | < 0.05 | < 0.05 | 0.18 | Study: |
| States | spray | | tall plants at | 3 | | 0.06 | < 0.05 | < 0.05 | 0.16 | 1998/5121 |
| Michigan | | | maturity | 7 | | 0.06 | 0.07 | < 0.05 | 0.18 | Trial: 97030 |
| Ottawa | | | - | | | | | | | Wofford & Riley, |
| County | | | | | | | | | | 1998, |
| 1997/98 | | | | | | | | | | Kresoxim_110 |
| (Superstar) | | | | | | | | | | Max. frozen |
| | | | | | | | | | | storage: 10 |
| | | | | | | | | | | month |
| United | Foliar | 6×196 | 12 foot tall | 0 | Fruit | 0.09 | < 0.05 | < 0.05 | 0.19 | Study: |
| States | spray | | plants at | 3 | | 0.07 | < 0.05 | < 0.05 | 0.17 | 1998/5121 |
| Oklahoma | | | fruit set/ | 7 | | 0.06 | < 0.05 | < 0.05 | 0.16 | Trial: 97033 |
| Caddo County | | | with mature fruit | | | | | | | Wofford & Riley, 1998, |
| 1997/98 | | | nuit | | | | | | | Kresoxim 110 |
| (Hybrid | | | | | | | | | | Max. frozen |
| Tesero) | | | | | | | | | | storage: 10 |
| 1000.0) | | | | | | | | | | month |

Table 105 Residues of kresoxim-methyl in cantaloupe following foliar treatment (cGAP: 4×168 g ai/ha; 0 days PHI)

| 1 | Treatment | Application | Growth | DALT | Crop | Residue Fou | ind (mg/kg |) | | |
|--|-----------------|-------------------------|--|------|-------|---------------------|------------|--------|-------------------------------|---|
| Location, Year (variety) | method | rate (g ai/ha) | stage at final application | | part | Kresoxim- methyl | 490M2 | 490M9 | Sum as parent equiv. | Report/Trial No., Reference Storage period |
| United States California Tulare County 1997/98 (PMR-45) | Foliar spray | 1×168 1×224 4×196 | 3-4 inch tall at maturity | 0 | Fruit | 0.16 | < 0.05 | < 0.05 | 0.26 | Study: 1998/5121 Trial: 97035 Wofford & Riley, 1998, Kresoxim_110 Max. frozen storage: 10 month |
| United States California Glann County 1997/98 (Charentais Melons) | Foliar spray | 1×168 1×224 4×196 | 9 inch tall plants at fruit development | 0 | Fruit | 0.05 | < 0.05 | < 0.05 | 0.15 | Study: 1998/5121 Trial: 97036 Wofford & Riley, 1998, Kresoxim_110 Max. frozen storage: 10 month |
| United States California Fresno County 1997/98 (Hybrid Primo) | Foliar spray | 6×196 | 12 inch tall plants with 6 inch fruit | 0 | Fruit | 0.07 | < 0.05 | < 0.05 | 0.17 | Study: 1998/5121 Trial: 97037 Wofford & Riley, 1998, Kresoxim_110 Max. frozen storage: 10 month |

During the 1996 growing season, a total of eight trials were conducted with melon in Spain under greenhouse conditions (Fuchs & Rabe, 1997, Kresoxim_115). Plants received 4 spray applications of kresoxim-methyl at nominal rates of 150 g ai/ha with interval between applications of about 10 days. Samples of melon were collected at 0, 3–5, 7 and 13–14 days after the last application. Residues of kresoxim-methyl (as 490M1) and metabolites 490M2 and 490M9 were determined using method BASF method 350/3 with a limit of quantification of 0.05 mg/kg. Overall mean procedural recoveries for kresoxim-methyl (as 490M1) and metabolites 490M2 and 90M9 in melon spiked at 0.05 and 5 mg/kg were 90% (n=10), 94% (n=10) and 90% (n=10), respectively.

Table 106 Residues of kresoxim-methyl in melon following foliar treatment under greenhouse conditions

| Location, | Treatment | Application | Growth | DALT | Crop | Residue Foun | d (mg/kg) ^a | | Doport/Trial No |
|--|-----------------|-------------------|----------------------------------|-------------------|-------|--|--------------------------------------|--------------------------------------|---|
| Year (variety)ª | method | rate (g ai/ha) | stage at final application | | part | Kresoxim- methyl | 490M2 | 490M9 | Report/Trial No., Reference Storage period |
| Spain Sevilla Alcala de Guadiara 1996 (Elisap) | Foliar spray | 4×150 | BBCH 79 | 0 4 7 14 | Fruit | < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 | < 0.05 < 0.05 < 0.05 < 0.05 | < 0.05 < 0.05 < 0.05 < 0.05 | Study: 1997/10155 Trial: ALO/21/95 Fuchs & Rabe, 1997, Kresoxim_115 Max. frozen storage: 9 month |
| Spain Sevilla Alcala de Guadiara 1996 (Mak-Dimon) | Foliar spray | 4×150 | BBCH 78-79 | 0 4 7 14 | Fruit | < 0.05 < 0.05 0.09 < 0.05 | < 0.05 < 0.05 < 0.05 < 0.05 | < 0.05 < 0.05 < 0.05 < 0.05 | Study: 1997/10155 Trial: ALO/22/95 Fuchs & Rabe, 1997, Kresoxim_115 Max. frozen storage: 9 month |

| Location, | Treatment | Application | Growth | DALT | Crop | Residue Four | id (mg/kg) ^a | | Dement/Trial Na |
|--|-----------------|-------------------|----------------------------------|-------------------|-------|--|--------------------------------------|--------------------------------------|---|
| Year (variety)ª | method | rate (g ai/ha) | stage at final application | | part | Kresoxim- methyl | 490M2 | 490M9 | Report/Trial No., Reference Storage period |
| Spain Sevilla Palacios 1996 (Mak-Dimon) | Foliar spray | 4×150 | BBCH 77-78 | 0 4 7 14 | Fruit | 0.07 0.11 0.07 < 0.05 | < 0.05 < 0.05 < 0.05 < 0.05 | < 0.05 0.06 < 0.05 < 0.05 | Study: 1997/10155 Trial: ALO/23/95 Fuchs & Rabe, 1997, Kresoxim_115 Max. frozen storage: 9 month |
| Spain Sevilla Palacios 1996 (Galia) | Foliar spray | 4×150 | BBCH 79-81 | 0 5 7 14 | Fruit | < 0.05 0.06 < 0.05 < 0.05 | < 0.05 < 0.05 < 0.05 < 0.05 | < 0.05 < 0.05 < 0.05 < 0.05 | Study: 1997/10155 Trial: ALO/24/95 Fuchs & Rabe, 1997, Kresoxim_115 Max. frozen storage: 9 month |
| Spain Cadiz Algaida- Sanlucar 1996 (Melina) | Foliar spray | 4×150 | BBCH 73-81 | 0 3 7 13 | Fruit | 0.07 0.06 < 0.05 < 0.05 | < 0.05 < 0.05 < 0.05 < 0.05 | < 0.05 < 0.05 < 0.05 < 0.05 | Study: 1997/10155 Trial: ALO/25/95 Fuchs & Rabe, 1997, Kresoxim_115 Max. frozen storage: 9 month |
| Spain Cadiz Algaida- Sanlucar 1996 (Melina) | Foliar spray | 4×150 | BBCH 73-80 | 0 3 7 13 | Fruit | < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 | < 0.05 < 0.05 < 0.05 < 0.05 | < 0.05 < 0.05 < 0.05 < 0.05 | Study: 1997/10155 Trial: ALO/26/95 Fuchs & Rabe, 1997, Kresoxim_115 Max. frozen storage: 9 month |
| Spain Cadiz Algaida- Sanlucar 1996 (Melina) | Foliar spray | 4×150 | BBCH 73-81 | 0 3 7 13 | Fruit | 0.05 0.08 < 0.05 < 0.05 | < 0.05 < 0.05 < 0.05 < 0.05 | < 0.05 < 0.05 < 0.05 < 0.05 | Study: 1997/10155 Trial: ALO/27/95 Fuchs & Rabe, 1997, Kresoxim_115 Max. frozen storage: 9 month |
| Spain Sevilla Palacios 1996 (Galia) | Foliar spray | 4×150 | BBCH 79-80 | 0 5 7 14 | Fruit | 0.10 0.16 < 0.05 < 0.05 | < 0.05 < 0.05 < 0.05 < 0.05 | < 0.05 < 0.05 < 0.05 < 0.05 | Study: 1997/10155 Trial: ALO/28/95 Fuchs & Rabe, 1997, Kresoxim_115 Max. frozen storage: 9 month |

^a Trials performed at the same location and year could not be considered as independent. Hence, only the highest residue was considered in these trials.

Fruiting vegetables, other than Cucurbits

Sweet pepper

A total of four field trials were conducted with sweet pepper in Brazil in the 2011/12 growing season (Silva & Cardoso, 2011, Kresoxim_117; Silva, 2011 Kresoxim_118, Silva, 2011 Kresoxim_118). Plants received 4 spray applications of kresoxim-methyl at nominal rates of 97 g ai/ha with a 7±1 day interval between applications. Samples of sweet pepper were collected at 0, 3 and 7

days after the last application. Residues of kresoxim-methyl (as 490M1) and metabolites 490M2 and 490M9 were determined using method BASF method 350/4 (identical to method L0095/01 and SOP-PA.0295) with a limit of quantification of 0.01 mg/kg. Overall mean procedural recoveries for kresoxim-methyl (as 490M1) and metabolites 490M2 and 490M9 in sweet pepper spiked at 0.01 and 1 mg/kg were 79% (n=13), 87% (n=12) and 93% (n=12), respectively.

| | Treatment | Application | Growth | DALT | Crop | Residue Fou | nd (mg/kg |) | | |
|--|-----------------|-------------------|-------------------|-------------|-------|----------------------|-------------------------|-------------------------|--------------------------------|--|
| Location, Year | method | rate (g ai/ha) | stage at final | | part | Kresoxim- methyl | 490M2 | 490M9 | Sum of | Report/Trial No., Reference |
| (variety) | | (y ai/ila) | application | | | metnyi | | | parent + | Storage period |
| (vunoty) | | | approation | | | | | | 490M9 | otoruge periou |
| Brazil Sto Antônio de Posse / SP 2010/11 (Magali R) | Foliar spray | 4×97 | BBCH 89 | 0 3 7 | Fruit | 0.07 0.04 0.01 | <0.01 <0.01 <0.01 | <0.01 <0.01 <0.01 | 490079 0.08 0.05 0.02 | Study: 2016/3004182, 2016/3004183. 2016/3004442 Trial: G100274 Silva & Cardoso, 2011, Kresoxim_117; Silva, 2011 Kresoxim_118, Silva, 2011 Kresoxim_118 Max. frozen storage: 12 month |
| Brazil Palmeira / PR 2010/11 (Hibrido Kormim) | Foliar spray | 4×97 | BBCH 76 | 0 3 7 | Fruit | 0.03 0.02 0.03 | <0.01 <0.01 <0.01 | <0.01 <0.01 <0.01 | 0.04 0.03 0.04 | Study: 2016/3004182, 2016/3004183, 2016/3004442 Trial: G100275 Silva & Cardoso, 2011, Kresoxim_117; Silva, 2011 Kresoxim_118, Silva, 2011 Kresoxim_118 Max. frozen storage: 4 month |
| Brazil Senador Canedo / PR 2010/11 (Magali R) | Foliar spray | 4×97 | BBCH 83 | 3 | Fruit | 0.02 | <0.01 | <0.01 | 0.03 | Study: 2016/3004182, 2016/3004183. 2016/3004442 Trial: G100276 Silva & Cardoso, 2011, Kresoxim_117; Silva, 2011 Kresoxim_118, Silva, 2011 Kresoxim_118 Max. frozen storage: 10 month |

Table 107 Residues of kresoxim-methyl in sweet pepper following foliar treatment (cGAP: 4×100 g ai/ha; 3 days PHI) in Brazil

| | Treatment | Application | Growth | DALT | Crop | Residue Fou | nd (mg/kg |) | | |
|-------------|-----------|-------------|-------------|------|-------|-------------|-----------|-------|--------|-------------------|
| Location, | method | rate | stage at | | part | Kresoxim- | 490M2 | 490M9 | Sum of | Report/Trial No., |
| Year | | (g ai/ha) | final | | | methyl | | | parent | Reference |
| (variety) | | | application | | | | | | + | Storage period |
| | | | | | | | | | 490M9 | |
| Brazil | Foliar | 4×97 | BBCH 83 | 3 | Fruit | <0.01 | <0.01 | <0.01 | <0.02 | Study: |
| Jaboticabal | spray | | | | | | | | | 2016/3004182, |
| / SP | | | | | | | | | | 2016/3004183. |
| 2010/11 | | | | | | | | | | 2016/3004442 |
| (Magali) | | | | | | | | | | Trial: G100277 |
| | | | | | | | | | | Silva & Cardoso, |
| | | | | | | | | | | 2011, |
| | | | | | | | | | | Kresoxim_117; |
| | | | | | | | | | | Silva, 2011 |
| | | | | | | | | | | Kresoxim_118, |
| | | | | | | | | | | Silva, 2011 |
| | | | | | | | | | | Kresoxim_118 |
| | | | | | | | | | | Max. frozen |
| | | | | | | | | | | storage: 9 |
| | | | | | | | | | | month |

During the 1995/96/97 growing seasons, a total of 10 trials were conducted with sweet pepper in Spain under greenhouse conditions (Fuchs & Rabe, 1997, Kresoxim_122) and under field conditions (Rabe, 1998, Kresoxim_123). Plants received 4 spray applications of kresoxim-methyl at nominal rates of 100 g ai/ha or 250 g ai/ha with interval between applications of about 10 days. Samples of sweet pepper were collected at 0, 3–5, 7–8 and 13–14 days after the last application. Residues of kresoxim-methyl (as 490M1) and metabolites 490M2 and 490M9 were determined using method BASF method 350/3 with a limit of quantification of 0.05 mg/kg. Overall mean procedural recoveries for kresoxim-methyl (as 490M1) and metabolites 490M2 and 490M9 in sweet pepper spiked at 0.05 and 5 mg/kg were 94% (n=8), 84% (n=8) and 83% (n=8), respectively.

Table 108 Residues of kresoxim-methyl in sweet pepper following foliar treatment in Spain under greenhouse conditions

| Location. | Treatment | Application | Growth | DALT | Crop | Residue Fou | und (mg/kg) | | Report/Trial No., |
|----------------|-----------|-------------------|-------------------|------|-------|-------------|-------------|--------|----------------------|
| LOCATION, | method | rate (g ai/ha) | stage at final | | part | Kresoxim- | 490M2 | 490M9 | Reference |
| Year (variety) | | (y ai/na) | application | | | methyl | | | Storage period |
| Spain | Foliar | 4×100 | BBCH 68- | 0 | Fruit | < 0.05 | < 0.05 | < 0.05 | Study: 1997/10144 |
| Cardiz | spray | | 77 | 5 | | < 0.05 | < 0.05 | < 0.05 | Trial: ALO/09/95 |
| La Algaida | | | | 7 | | < 0.05 | < 0.05 | < 0.05 | Fuchs & Rabe, 1997, |
| 1995 | | | | 13 | | < 0.05 | < 0.05 | < 0.05 | Kresoxim_122 |
| (Italico) | | | | | | | | | Max. frozen storage: |
| Green house | | | | | | | | | 7 month |
| Spain | Foliar | 4×100 | BBCH 69- | 0 | Fruit | < 0.05 | < 0.05 | < 0.05 | Study: 1997/10144 |
| Cardiz | spray | | 75 | 4 | | < 0.05 | < 0.05 | < 0.05 | Trial: ALO/10/95 |
| La Algaida | | | | 7 | | < 0.05 | < 0.05 | < 0.05 | Fuchs & Rabe, 1997, |
| 1995 | | | | 14 | | < 0.05 | < 0.05 | < 0.05 | Kresoxim_122 |
| (Italico) | | | | | | | | | Max. frozen storage: |
| Green house | | | | | | | | | 7 month |
| Spain | Foliar | 4×250 | BBCH 77 | 0 | Fruit | 0.15 | < 0.05 | < 0.05 | Study: 1997/10144 |
| Sevilla | spray | | | 4 | | 0.15 | < 0.05 | < 0.05 | Trial: ALO/11/95 |
| Alcala de | | | | 7 | | 0.27 | 0.05 | < 0.05 | Fuchs & Rabe, 1997, |
| Guadaira | | | | 14 | | 0.17 | 0.05 | < 0.05 | Kresoxim_122 |
| 1996 | | | | | | | | | Max. frozen storage: |
| (Italico) | | | | | | | | | 6 month |
| Green house | | | | | | | | | |
| Spain | Foliar | 4×250 | BBCH 77- | 0 | Fruit | 0.41 | 0.05 | < 0.05 | Study: 1997/10144 |
| Sevilla | spray | | 79 | 3 | | 0.21 | < 0.05 | < 0.05 | Trial: ALO/12/95 |
| Alcala de | | | | 7 | | 0.15 | < 0.05 | < 0.05 | Fuchs & Rabe, 1997, |
| Guadaira | | | | 14 | | 0.14 | < 0.05 | < 0.05 | Kresoxim_122 |
| 1996 | | | | | | | | | Max. frozen storage: |
| (Italico) | | | | | | | | | 6 month |
| Green house | | | | | | | | | |

| | Treatment | Application | Growth stage | DALT | Crop | Residue Four | id (mg/kg) | | | _ |
|--|--------------|-------------------|----------------------|--------|-------|---------------------|------------------|------------------|-----------------------------|---|
| Location, Year (variety)ª | method | rate (g ai/ha) | at final application | | part | Kresoxim- methyl | 490M2 | 490M9 | Sum of parent + 490M9 | Report/Trial No., Reference Storage period |
| Spain Sevilla Los Palacios 1997 (Italico) Field | Foliar spray | 4×250 | BBCH 75-76 | 4 7 | Fruit | 0.44* 0.38 | < 0.05 < 0.05 | < 0.05 < 0.05 | 0.49* 0.43 | Study: 1998/10217 Trial: Pi/97/07 Rabe, 1998, Kresoxim_123 Max. frozen storage: 2 month |
| Spain ^a Sevilla Los Palacios 1997 (Italico) Field | Foliar spray | 4×250 | BBCH 75-76 | 4 8 | Fruit | 0.39 0.32 | < 0.05 < 0.05 | < 0.05 < 0.05 | 0.44 0.37 | Study: 1998/10217 Trial: Pi/97/08 Rabe, 1998, Kresoxim_123 Max. frozen storage: 2 month |
| Spain Sevilla Los Palacios 1997 (Italico) Field | Foliar spray | 4×250 | BBCH 78-80 | 4 7 | Fruit | 0.37 0.36 | < 0.05 < 0.05 | < 0.05 < 0.05 | 0.42 0.41 | Study: 1998/10217 Trial: Pi/97/09 Rabe, 1998, Kresoxim_123 Max. frozen storage: 2 month |
| Spain Cardiz La Algaida 1997 (Italico) Field | Foliar spray | 4×250 | BBCH 75-76 | 4 8 | Fruit | 0.10 0.16* | < 0.05 < 0.05 | < 0.05 < 0.05 | 0.15 0.21* | Study: 1998/10217 Trial: Pi/97/10 Rabe, 1998, Kresoxim_123 Max. frozen storage: 2 month |
| Spain Cardiz La Algaida 1997 (Italico) Field | Foliar spray | 4×250 | BBCH 75-77 | 4 8 | Fruit | 0.19 0.11 | < 0.05 < 0.05 | < 0.05 < 0.05 | 0.24 0.16 | Study: 1998/10217 Trial: Pi/97/11 Rabe, 1998, Kresoxim_123 Max. frozen storage: 2 month |
| Spain Cardiz La Algaida 1997 (Italico) Field | Foliar spray | 4×250 | BBCH 75-76 | 4 7 | Fruit | 0.10 0.12 | < 0.05 < 0.05 | < 0.05 < 0.05 | 0.15 0.17 | Study: 1998/10217 Trial: Pi/97/12 Rabe, 1998, Kresoxim_123 Max. frozen storage: 2 month |

Table 109 Residues of kresoxim-methyl in sweet pepper following foliar treatment (cGAP: 4×100 g ai/ha; 3 days PHI) in Spain under field conditions

^a Trials performed at the same location and year could not be considered as independent. Hence, only the highest residue was considered in these trials.

(*): Residues were scaled based on a calculated scaling factor of 0.4. Scaling factor = intended GAP rate (100 g ai/ha) ÷ trials/GAP rate (250 g ai/ha). N.B. Scaling only applies to residue levels ≥0.01 mg/kg (≥LOQ).

Tomato

A total of five field trials were conducted with tomato in Brazil in the 2004 growing season (Dantas, 2005, Kresoxim_120). Plants received 4 spray applications of kresoxim-methyl at nominal rates of 200 g ai/ha or at an exaggerated rate of 400 g ai/ha with a 7 day interval between applications. Samples of tomato were collected at 0, 1, 3, 7 and 10 days after the last application. Residues of kresoxim-methyl were determined using method SOP-PA.0243 (identical to method 445/0) with a limit of quantification of 0.01 mg/kg. Overall mean procedural recoveries for kresoxim-methyl in tomato spiked at 0.01 and 1 mg/kg were 100% (n=6).

One additional field trial was conducted with tomato in Brazil in the 1996 growing season (Tornisielo, 1997, Kresoxim_121). Plants received 6 spray applications of kresoxim-methyl at nominal rates of 200 g ai/ha or at an exaggerated rate of 400 g ai/ha with a 7 day interval between applications. Samples of tomato were collected at 1 and 3 days after the last application. Residues of kresoxim-methyl were determined according to BASF method 351/2 with a limit of quantification of 0.05 mg/kg. Overall mean procedural recoveries for kresoxim-methyl in tomato spiked at 0.05 were 109% (n=2).

Table 110 Residues of kresoxim-methyl in tomato following foliar treatment (cGAP: 2×200 g ai/ha; 3 days PHI) in Brazil

| nethod | Application rate (g ai/ha) | at final | | | Residue Fou | | 1 | - |
|-------------|----------------------------------|---|---|---|---|--|---|---|
| | (g ai/ha) | | | part | Kresoxim- | 490M2 | 490M9 | Report/Trial No., Reference |
| | (g all may | application | | | methyl | | | Storage period |
| | | | | | | | | |
| oliar spray | 4×200 | BBCH 71 | 0 | Fruit | < 0.01 | - | - | Study: 2016/3004461 |
| | | | 1 | | < 0.01 | | | Trial: |
| | | | 3 | | < 0.01 | | | EC/CD/F/2004/003/BRU/A1 |
| | | | 7 | | < 0.01 | | | Dantas, 2005, Kresoxim_120 |
| | | | 10 | | < 0.01 | | | Max. frozen storage: 5 month |
| oliar spray | 4×200 | BBCH 61 | 3 | Fruit | < 0.01 | - | - | Study: 2016/3004461 |
| | | | | | | | | Trial: |
| | | | | | | | | EC/R/F/2004/003/BRU/A1 |
| | 4×400 | BBCH 61 | 3 | Fruit | < 0.01 | - | - | Dantas, 2005, Kresoxim_120 |
| | | | - | | | | | Max. frozen storage: 5 month |
| | | | | | | | | |
| | | | | | | | - | |
| oliar spray | 4×200 | BBCH 61 | 3 | Fruit | < 0.01 | - | - | Study: 2016/3004461 |
| | | | | | | | | Trial: |
| | | | | | | | | EC/R/F/2004/003/BRU/B1 |
| | 4×400 | BBCH 61 | 3 | Fruit | < 0.01 | - | - | Dantas, 2005, Kresoxim_120 |
| | | | - | | | | | Max. frozen storage: 5 month |
| | | | | | | | | |
| oliar enrav | 4~200 | | 2 | Erwit | 0.06 | | | Study: 2016/3004461 |
| ullal splay | 4×200 | DDCI109 | 3 | Tun | 0.00 | - | - | Trial: |
| | | | | | | | | EC/R/F/2004/003/BRT/A1 |
| | | | | | | | | Dantas, 2005, Kresoxim 120 |
| | 4×400 | BBCH 89 | 3 | Fruit | 0.04 | - | - | Max. frozen storage: 5 month |
| | | | | | | | | Max. Hozen storage. e month |
| | | | | | | | | |
| oliar spray | 4×200 | BBCH 82 | 3 | Fruit | 0.06 | - | - | Study: 2016/3004461 |
| | | | | | | | | Trial: |
| | | | | | | | | EC/R/F/2004/003/BRV/A1 |
| | 4 | | 2 | Erwit | 0.15 | | | Dantas, 2005, Kresoxim_120 |
| | 4×400 | | 3 | FIUIL | 0.15 | - | - | Max. frozen storage: 6 month |
| | | | | | | | | |
| | | | | | | | | |
| oliar spray | 6×200 | BBCH 75-82 | 1 | Fruit | | - | - | Study: 2016/3004181 |
| | | | 3 | | 0.52 | | | Trial: R/033/96/BR8/006 |
| | | | | | | | | Tornisielo, 1997, |
| | 6×400 | BBCH 75-82 | 1 | Fruit | 0.44 | - | - | Kresoxim_121 |
| | | | 3 | | 0.30 | | | Max. frozen storage: not |
| | | | | | | | | stated |
| 0 | liar spray | 4×400 liar spray 4×200 4×400 | Iiar spray 4×200 BBCH 61 Iiar spray 4×200 BBCH 61 Iiar spray 4×200 BBCH 61 Iiar spray 4×200 BBCH 89 Iiar spray 4×200 BBCH 89 Iiar spray 4×200 BBCH 89 Iiar spray 4×200 BBCH 82 Iiar spray 4×200 BBCH 82 Iiar spray 4×200 BBCH 82 Iiar spray 4×200 BBCH 82 | Io Io liar spray 4×200 BBCH 61 3 4×400 BBCH 61 3 liar spray 4×200 BBCH 89 3 liar spray 4×200 BBCH 82 3 liar spray 6×200 BBCH 82 3 liar spray 6×200 BBCH 75-82 1 | Io Io liar spray 4×200 BBCH 61 3 Fruit 4×400 BBCH 61 3 Fruit liar spray 4×200 BBCH 89 3 Fruit liar spray 4×200 BBCH 82 3 Fruit liar spray 4×200 BBCH 82 3 Fruit liar spray 6×200 BBCH 75-82 1 Fruit | IO < 0.01 liar spray 4×200 BBCH 61 3 Fruit < 0.01 | IO < 0.01 liar spray 4×200 BBCH 61 3 Fruit < 0.01 $ 4 \times 400$ BBCH 61 3 Fruit < 0.01 $-$ liar spray 4×200 BBCH 61 3 Fruit < 0.01 $-$ liar spray 4×200 BBCH 61 3 Fruit < 0.01 $-$ liar spray 4×200 BBCH 89 3 Fruit < 0.01 $-$ liar spray 4×200 BBCH 89 3 Fruit 0.06 $-$ liar spray 4×200 BBCH 82 3 Fruit 0.06 $-$ liar spray 4×200 BBCH 82 3 Fruit 0.06 $-$ liar spray 4×400 BBCH 82 3 Fruit 0.06 $-$ liar spray 6×200 BBCH 75-82 1 Fruit 0.45 $-$ liar spray 6×400 BBCH 75-82 1 Fruit | Ind Ind |

During the 1995/96/97 growing seasons, a total of 12 trials were conducted with tomato in Spain under greenhouse conditions (Fuchs & Rabe, 1997, Kresoxim_122; Rabe, 1998, Kresoxim_123). Plants received 4 spray applications of kresoxim-methyl at nominal rates of 100 g ai/ha or 250 g ai/ha with interval between applications of about 10 days. Samples of tomato were collected at 0, 3–5, 7–8 and 13–14 days after the last application. Residues of kresoxim-methyl (as 490M1) and metabolites 490M2 and 490M9 were determined using method BASF method 350/3 with a limit of quantification of 0.05 mg/kg. Overall mean procedural recoveries for kresoxim-methyl (as 490M1) and metabolites 490M2 and 490M9 in tomato spiked at 0.05 and 5 mg/kg were 88% (n=12), 94% (n=12) and 89% (n=12), respectively.

| | Treatment | Application | Growth | DALT | Crop | Residue Fou | ind (ma/ka) | 3 | I |
|---|-----------------|-------------|----------------|-------------------|-------|---------------------------------|--------------------------------------|--------------------------------------|--|
| Location, | method | rate | stage at final | DALT | part | Kresoxim- | 490M2 | 490M9 | Report/Trial No., Reference |
| Year (variety) | | (g ai/ha) | application | | | methyl | | | Storage period |
| Spain Sevilla Alcala de Guadaira 1995 | Foliar spray | 4×100 | BBCH 69- 83 | 0 3 7 14 | Fruit | <0.05 <0.05 <0.05 0.07 | < 0.05 < 0.05 < 0.05 < 0.05 | < 0.05 < 0.05 < 0.05 < 0.05 | Study: 1997/10144 Trial: ALO/01/95 Fuchs & Rabe, 1997, Kresoxim_122 Max. frozen storage: |
| (Presto) | | | | | | | | | 14 month |
| Spain Sevilla Alcala de Guadaira 1995 (Gerano) | Foliar spray | 4×100 | BBCH 69- 83 | 0 3 7 14 | Fruit | 0.06 0.05 0.06 <0.05 | < 0.05 < 0.05 < 0.05 < 0.05 | < 0.05 < 0.05 < 0.05 < 0.05 | Study: 1997/10144 Trial: ALO/02/95 Fuchs & Rabe, 1997, Kresoxim_122 Max. frozen storage: 14 month |
| Spain Cardiz La Algaida 1995 (Fimande) | Foliar spray | 4×100 | BBCH 68- 81 | 0 5 7 13 | Fruit | 0.07 0.08 <0.05 <0.05 | < 0.05 < 0.05 < 0.05 < 0.05 | < 0.05 < 0.05 < 0.05 < 0.05 | Study: 1997/10144 Trial: ALO/03/95 Fuchs & Rabe, 1997, Kresoxim_122 Max. frozen storage: 14 month |
| Spain Cardiz La Algaida 1995 (Fimande) | Foliar spray | 4×100 | BBCH 68- 82 | 0 4 7 14 | Fruit | <0.05 0.06 0.05 0.11 | < 0.05 < 0.05 < 0.05 < 0.05 | < 0.05 < 0.05 < 0.05 < 0.05 | Study: 1997/10144 Trial: ALO/04/95 Fuchs & Rabe, 1997, Kresoxim_122 Max. frozen storage: 14 month |
| Spain Sevilla Alcala de Guadaira 1996 (Prieto) | Foliar spray | 4×250 | BBCH 68- 82 | 0 3 7 14 | Fruit | 0.35 0.31 0.25 0.10 | < 0.05 < 0.05 < 0.05 < 0.05 | < 0.05 < 0.05 < 0.05 < 0.05 | Study: 1997/10144 Trial: ALO/05/95 Fuchs & Rabe, 1997, Kresoxim_122 Max. frozen storage: 7 month |
| Spain Sevilla Alcala de Guadaira 1996 (Prieto) | Foliar spray | 4×250 | BBCH 69- 83 | 0 4 7 14 | Fruit | 0.20 0.14 0.13 0.08 | < 0.05 < 0.05 < 0.05 < 0.05 | < 0.05 < 0.05 < 0.05 < 0.05 | Study: 1997/10144 Trial: ALO/06/95 Fuchs & Rabe, 1997, Kresoxim_122 Max. frozen storage: 7 month |
| Spain Sevilla Palacios 1996 (Gerano) | Foliar spray | 4×250 | BBCH 69- 82 | 0 4 7 14 | Fruit | 0.26 0.25 0.12 0.10 | < 0.05 < 0.05 < 0.05 < 0.05 | < 0.05 < 0.05 < 0.05 < 0.05 | Study: 1997/10144 Trial: ALO/07/95 Fuchs & Rabe, 1997, Kresoxim_122 Max. frozen storage: 7 month |
| Spain Sevilla Palacios 1996 (Empire) | Foliar spray | 4×250 | BBCH 69- 82 | 0 4 7 14 | Fruit | 0.11 0.09 0.08 0.06 | < 0.05 < 0.05 < 0.05 < 0.05 | < 0.05 < 0.05 < 0.05 < 0.05 | Study: 1997/10144 Trial: ALO/08/95 Fuchs & Rabe, 1997, Kresoxim_122 Max. frozen storage: 7 month |

Table 111 Residues of kresoxim-methyl in tomato following foliar treatment in Spain under greenhouse conditions

| Location. | Treatment | Application | Growth | DALT | Crop | Residue Fou | Ind (mg/kg) ^a | | Deport/Trial No |
|----------------------|-----------|-------------------|-------------------|------|-------|---------------------|--------------------------|--------|--------------------------------------|
| LUCATION, | method | rate (g ai/ha) | stage at final | | part | Kresoxim- methyl | 490M2 | 490M9 | Report/Trial No., Reference |
| Year (variety) | | (g ai/na) | application | | | metnyi | | | Storage period |
| Spain | Foliar | 4×250 | BBCH 76- | 4 | Fruit | 0.17 | < 0.05 | < 0.05 | Study: 1998/10217 |
| Sevilla | spray | | 78 | 7 | | 0.20 | < 0.05 | < 0.05 | Trial: To/97/03 |
| Los Palacios 1997 | | | | | | | | | Rabe, 1998, Kresoxim 123 |
| (Gerano) | | | | | | | | | Max. frozen storage: |
| . , | | | | | | | | | 2 month |
| Spain | Foliar | 4×250 | BBCH 76- | 4 | Fruit | 0.27 | < 0.05 | < 0.05 | Study: 1998/10217 |
| Sevilla | spray | | 79 | 7 | | 0.18 | < 0.05 | < 0.05 | Trial: To/97/04 |
| Los Palacios 1997 | | | | | | | | | Rabe, 1998, Kresoxim 123 |
| (Optima) | | | | | | | | | Max. frozen storage: |
| (0) | | | | | | | | | 2 month |
| Spain | Foliar | 4×250 | BBCH 77- | 4 | Fruit | 0.23 | < 0.05 | < 0.05 | Study: 1998/10217 |
| Cardiz | spray | | 79 | 7 | | 0.16 | < 0.05 | < 0.05 | Trial: To/97/05 |
| La Algaida | | | | | | | | | Rabe, 1998, |
| 1997 (Bonne) | | | | | | | | | Kresoxim_123 Max. frozen storage: |
| (BOIIIIe) | | | | | | | | | 2 month |
| Spain | Foliar | 4×250 | BBCH 75- | 4 | Fruit | 0.13 | < 0.05 | < 0.05 | Study: 1998/10217 |
| Cardiz | spray | | 76 | 7 | | 0.08 | < 0.05 | < 0.05 | Trial: To/97/06 |
| La Algaida | | | | | | | | | Rabe, 1998, |
| 1997 | | | | | | | | | Kresoxim_123 |
| (Bonne) | | | | | | | | | Max. frozen storage: |
| | | | | | | | | | 2 month |

Table 112 Residues of kresoxim-methyl in tomato following foliar treatment in Spain under field conditions

Leafy vegetables (including Brassica leafy vegetables)

Grape leaves

During the 2007 growing seasons, a total of 8 trials were conducted with grapes and leaves in Northern and Southern France, Germany, Italy and Spain (Schulz, 2008, Kresoxim_124). Plants received 3 spray applications of kresoxim-methyl at nominal rates of 150 g ai/ha with an interval between applications of 8–10 days. Samples of leaves and fruit were collected directly before the 2nd and 3rd application (leaves only) and at 0, 20–21, 26–29, 33–36 and 41–43 days after the last application. Residues of kresoxim-methyl (as 490M1) and metabolites 490M2 and 490M9 were determined using method BASF method 445/0 for leaves and method L0095/1 (identical to BASF method 350/4) in fruit. The LOQ for both methods was equal to 0.01 mg/kg. For method L0095/1, the overall mean procedural recoveries for kresoxim-methyl (as 490M1) and metabolites 490M2 and 79% (n=12), respectively. For method 445/0, the overall mean procedural recoveries for kresoxim-methyl (as 490M1) and metabolites 490M2 and 490M9 in leaves spiked at 0.01 and 1 mg/kg (for parent additionally at 20 mg/kg) were 92% (n=17), 85% (n=16) and 91% (n=13), respectively.

Another 8 trials were conducted with grapes and leaves in Northern and Southern France, Germany, Greece, Italy and Spain in the 2008 growing season (Schulz, 2009, Kresoxim_125). Plants received 3 spray applications of kresoxim-methyl at nominal rates of 150 g ai/ha with an interval between applications of 8±1 days. Samples of leaves and fruit were collected directly before the 2nd and 3rd application (leaves only) and at 0 and 21–22 days after the last application. Residues of kresoxim-methyl (as 490M1) and metabolites 490M2 and 490M9 were determined in grape leaves using method L0095/1 (identical to BASF method 350/4) with an LOQ of 0.01 mg/kg. The overall mean procedural recoveries for kresoxim-methyl (as 490M1) and metabolites 490M2 and 490M9 in leaves spiked at 0.01–50 mg/kg were 100% (n=14), 85% (n=12) and 100% (n=12), respectively.

| | Treatment | Application | Growth | DALT | Crop | Residue Fou | nd (ma/ka |) | | |
|--|-----------|-------------|-------------|----------------|--------|------------------|-----------|------------|--------|----------------|
| Location, | method | rate | stage at | | part | Kresoxim- | 490M2 | , 490M9 | Sum | Report/Trial |
| Year | | (g ai/ha) | final | | | methyl | | | as | No., Reference |
| (variety) | | | application | | | | | | parent | Storage period |
| . ,, | | | | | | | | | equiv. | 5 1 |
| Germany | Foliar | 3×150 | BBCH 85 | 8 ^a | Leaves | 6.6 | 0.04 | 0.08 | 6.7 | Study: |
| Eschbach | spray | | | 8 ^b | | 7.8 | 0.06 | 0.10 | 8.0 | 2008/1014860 |
| 2007 | . , | | | 0 | | 15 | 0.06 | 0.09 | 15 | Trial: L070856 |
| (Portugieser) | | | | 21 | | 10 | 0.08 | 0.13 | 10 | Schulz, 2008, |
| (^a ^g | | | | 28 | | 5.6 | 0.08 | 0.11 | 5.8 | Kresoxim_124 |
| | | | | 35 | | 7.8 | 0.09 | 0.15 | 8.0 | Max. frozen |
| | | | | 42 | | 6.5 | 0.12 | 0.16 | 6.8 | storage: 10 |
| | | | | | | | | | | month |
| Germany | Foliar | 3×150 | BBCH 85 | 8 ^a | Leaves | 3.0 | 0.06 | 0.04 | 3.1 | Study: |
| Ockenheim | spray | | | 8 ^b | | 4.7 | 0.06 | 0.05 | 4.8 | 2008/1014860 |
| 2007 | | | | 0 | | 10 | 0.05 | 0.05 | 10 | Trial: L070857 |
| (Regent) | | | | 21 | | 3.4 | 0.07 | 0.07 | 3.5 | Schulz, 2008, |
| | | | | 28 | | 4.3 | 0.13 | 0.10 | 4.5 | Kresoxim_124 |
| | | | | 35 | | 3.4 | 0.10 | 0.10 | 3.6 | Max. frozen |
| | | | | 42 | | 3.6 | 0.16 | 0.10 | 3.9 | storage: 10 |
| | | | | | | | | | | month |
| France | Foliar | 3×150 | BBCH 81 | 8 ^a | Leaves | 8.1 ^c | 0.05 | 0.08 | 8.3 | Study: |
| Amboise | spray | | | 8 ^b | | 12 ^c | 0.07 | 0.11 | 12 | 2008/1014860 |
| 2007 | | | | 0 | | 22 ^c | 0.09 | 0.12 | 22 | Trial: L070858 |
| (Chenin) | | | | 21 | | 7.1 ^c | 0.11 | 0.20 | 7.5 | Schulz, 2008, |
| | | | | 28 | | 6.7 ^c | 0.11 | 0.18 | 7.0 | Kresoxim_124 |
| | | | | 35 | | 6.4 ^c | 0.15 | 0.27 | 6.8 | Max. frozen |
| | | | | 42 | | 5.8 ^c | 0.11 | 0.26 | 6.2 | storage: 10 |
| | | | | | | | | | | month |
| France | Foliar | 3×150 | BBCH 81 | 8 ^a | Leaves | 4.6 | 0.02 | 0.04 | 4.7 | Study: |
| Noizay | spray | | | 8 ^b | | 8.4 | 0.04 | 0.14 | 8.6 | 2008/1014860 |
| 2007 | | | | 0 | | 11 | 0.04 | 0.12 | 11 | Trial: L070859 |
| (Chenin) | | | | 21 | | 4.5 | 0.08 | 0.28 | 4.8 | Schulz, 2008, |
| | | | | 28 | | 4.3 | 0.08 | 0.24 | 4.6 | Kresoxim_124 |
| | | | | 35 | | 3.5 | 0.06 | 0.22 | 3.7 | Max. frozen |
| | | | | 42 | | 2.5 | 0.09 | 0.28 | 2.8 | storage: 10 |
| | | | | | | | | | | month |
| France | Foliar | 3×150 | BBCH 85 | 8 ^a | Leaves | 8.1 | 0.07 | 0.05 | 8.2 | Study: |
| Saint Martial | spray | | | 8 ^b | | 6.7 | 0.06 | 0.08 | 6.9 | 2008/1014860 |
| 2007 | | | | 0 | | 11 | 0.06 | 0.08 | 11 | Trial: L070860 |
| (Cabernet | | | | 21 | | 5.4 | 0.11 | 0.18 | 5.7 | Schulz, 2008, |
| Sauvignon) | | | | 28 | | 4.5 | 0.11 | 0.17 | 4.8 | Kresoxim_124 |
| - | | | | 35 | | 2.6 | 0.12 | 0.20 | 2.9 | Max. frozen |
| | | | | 42 | | 2.5 | 0.12 | 0.18 | 2.8 | storage: 10 |
| | | | | | | | | | | month |
| Italy | Foliar | 3×150 | BBCH 81- | 8 ^a | Leaves | 1.4 | 0.04 | 0.06 | 1.5 | Study: |
| Russi | spray | | 83 | 8 ^b | | 1.5 | 0.04 | 0.07 | 1.6 | 2008/1014860 |
| 2007 | | | 1 | 0 | | 7.7 | 0.07 | 0.09 | 7.9 | Trial: L070861 |
| (Trebbiano | | | 1 | 21 | | 0.17 | 0.09 | 0.14 | 0.40 | Schulz, 2008, |
| di | | | | 28 | | 0.13 | 0.08 | 0.16 | 0.37 | Kresoxim_124 |
| Romagner) | | | | 35 | | 0.03 | 0.07 | 0.13 | 0.23 | Max. frozen |
| | | | | 42 | | 0.05 | 0.08 | 0.17 | 0.30 | storage: 10 |
| | | | | | | | | | | month |
| France | Foliar | 3×150 | BBCH 85 | 8 ^a | Leaves | 5.8 | 0.07 | 0.10 | 6.0 | Study: |
| Saint | spray | | | 8 ^b | | 6.7 | 0.12 | 0.23 | 7.0 | 2008/1014860 |
| Sauveur de | | | | 0 | | 18 | 0.10 | 0.19 | 18 | Trial: L070862 |
| Meilhan | | | | 21 | | 4.4 ^c | 0.21 | 0.37 | 4.9 | Schulz, 2008, |
| 2007 | | | 1 | 28 | | 3.7 ^c | 0.20 | 0.23 | 4.2 | Kresoxim_124 |
| (Abourriot) | | | | 35 | | 3.6 | 0.21 | 0.25 | 4.1 | Max. frozen |
| . , | | | | 42 | | 1.6 | 0.09 | 0.20 | 1.9 | storage: 10 |
| | | | | | | | | | | otoragoi ro |

Table 113 Residues of kresoxim-methyl in grape leaves following foliar treatment (cGAP: 3×30 g ai/ha; 14 days PHI)

| | Treatment | Application | Growth | DALT | Crop | Residue Fou | Ind (ma/ka |) | | |
|---|-----------------|-------------------|----------------------------------|---|--------|--|--|--|--|--|
| Location, Year (variety) | method | rate (g ai/ha) | stage at final application | | part | Kresoxim- methyl | 490M2 | 490M9 | Sum as parent equiv. | Report/Trial No., Reference Storage period |
| Spain Malaga 2007 (Moscatel de Alejandria) | Foliar spray | 3×150 | BBCH 81 | 8 ^a 8 ^b 0 21 28 35 42 | Leaves | 0.06 0.11 0.16 0.11 0.08 0.15 0.10 | 0.01 0.02 0.01 0.01 < 0.01 < 0.01 < 0.01 | 0.02 0.03 0.02 0.02 0.01 0.01 0.02 | 0.09 0.16 0.19 0.14 0.10 0.17 0.13 | Study: 2008/1014860 Trial: L070863 Schulz, 2008, Kresoxim_124 Max. frozen storage: 10 month |
| Germany Wiesloch 2008 (Müller- Thurgau) | Foliar spray | 3×150 | BBCH 81 | 8 ^a 8 ^b 0 21 | Leaves | 7.4 6.2 20 2.7 | 0.02 0.09 0.11 0.13 | 0.02 0.13 0.16 0.19 | 7.4 6.4 20 3.0 | Study: 2009/1018523 Trial: L080368 Schulz, 2009, Kresoxim_125 Max. frozen storage: 4 month |
| Germany Ockenheim 2008 (Johanniter) | Foliar spray | 3×150 | BBCH 81 | 8 ^a 8 ^b 0 21 | Leaves | 2.5 4.2 10 4.2 | 0.03 0.04 0.03 0.08 | 0.06 0.12 0.08 0.19 | 2.6 4.4 10 4.5 | Study: 2009/1018523 Trial: L080369 Schulz, 2009, Kresoxim_125 Max. frozen storage: 4 month |
| France Noizay 2008 (Chenin) | Foliar spray | 3×150 | BBCH 83 | 8 ^a 8 ^b 0 21 | Leaves | 4.6 8.0 22 6.4 | 0.01 0.05 0.04 0.06 | 0.03 0.13 0.12 0.18 | 4.6 8.2 22 6.6 | Study: 2009/1018523 Trial: L080370 Schulz, 2009, Kresoxim_125 Max. frozen storage: 3 month |
| France Brimont 2008 (Pinot noir) | Foliar spray | 3×150 | BBCH 79 | 8ª 8 ^b 0 21 | Leaves | 8.8 8.3 16 4.5 | 0.05 0.06 0.05 0.06 | 0.09 0.17 0.16 0.30 | 8.9 8.5 16 4.9 | Study: 2009/1018523 Trial: L080371 Schulz, 2009, Kresoxim_125 Max. frozen storage: 4 month |
| France Lot-et- Garonne 2008 (Cabernet Sauvignon) | Foliar spray | 3×150 | BBCH 85 | 8 ^a 8 ^b 0 21 | Leaves | 6.8 11 30 9.2 | 0.04 0.09 0.08 0.08 | 0.19 0.39 0.34 0.29 | 7.0 11 30 9.6 | Study: 2009/1018523 Trial: L080372 Schulz, 2009, Kresoxim_125 Max. frozen storage: 3 month |
| Italy Bologna 2008 (Trebbiano di Romagna) | Foliar spray | 3×150 | BBCH 81 | 8 ^a 8 ^b 0 21 | Leaves | 2.8 3.1 15 0.88 | 0.02 0.02 0.04 0.04 | 0.09 0.12 0.21 0.25 | 2.9 3.2 15 1.2 | Study: 2009/1018523 Trial: L080373 Schulz, 2009, Kresoxim_125 Max. frozen storage: 4 month |

| | Treatment | Application | Growth | DALT | Crop | Residue Fou | nd (mg/kg |) | | |
|--------------|-----------|-------------|-------------|----------------|--------|------------------|--------------------|-------------------|--------|----------------|
| Location, | method | rate | stage at | | part | Kresoxim- | 490M2 | 490M9 | Sum | Report/Trial |
| Year | | (g ai/ha) | final | | | methyl | | | as | No., Reference |
| (variety) | | | application | | | | | | parent | Storage period |
| | | | | | | | | | equiv. | |
| Greece | Foliar | 3×150 | BBCH 83 | 8 ^a | Leaves | 4.3 | 0.03 | 0.06 | 4.4 | Study: |
| Piera | spray | | | 8 ^b | | 2.6 | 0.04 | 0.08 | 2.7 | 2009/1018523 |
| 2008 | | | | 0 | | 17 | 0.04 | 0.07 | 17 | Trial: L080374 |
| (Muscat) | | | | 21 | | 1.7 | 0.07 | 0.11 | 1.9 | Schulz, 2009, |
| | | | | | | | | | | Kresoxim_125 |
| | | | | | | | | | | Max. frozen |
| | | | | | | | | | | storage: 4 |
| | | | | | | | | | | month |
| Spain | Foliar | 3×150 | BBCH 75 | 8 ^a | Leaves | 0.06 | <0.01 | 0.03 | 0.10 | Study: |
| Malaga | spray | | | 8 ^b | | 4.3 ^c | <0.01 ^c | 0.03 ^c | 4.3 | 2009/1018523 |
| 2008 | | | | 0 | | 7.4 ^c | 0.02 ^c | 0.07 ^c | 7.5 | Trial: L080375 |
| (Moscatel de | | | | 21 | | 3.1 | <0.01 | 0.03 | 3.1 | Schulz, 2009, |
| Alejandria) | | | | | | | | | | Kresoxim_125 |
| | | | | | | | | | | Max. frozen |
| | | | | | | | | | | storage: 6 |
| | | | | | | | | | | month |

^a days after first application

 $^{\rm b}$ days after second application

 $^{\rm c}$ Mean of duplicate analysis.

Root and tuber vegetables

Sugar beet

During the 1996 and 1997 growing seasons, a total of ten field trials were conducted with sugar beet in France and Germany (Raunft, 1998, Kresoxim_126; Raunft, 1998, Kresoxim_127). Plants received 2 spray applications of kresoxim-methyl at nominal rates of 125 g ai/ha with a 21 day interval between applications. Samples of sugar beet were collected at 0, 27–29 and 41–43 days after the last application. Residues of kresoxim-methyl (as 490M1) and metabolites 490M2 and 490M9 were determined using method BASF method 350/3 with a limit of quantification of 0.05 mg/kg. Overall mean procedural recoveries for kresoxim-methyl (as 490M1) and metabolites 490M2 and 5 mg/kg were 86% (n=9), 85% (n=10) and 87% (n=9), respectively.

Table 114 Residues of kresoxim-methyl in sugar beet roots following foliar treatment (cGAP: 2 × 125 g ai/ha; 28 days PHI)

| | Treatment | Application | Growth | DALT | Crop | Residue Fou | und (mg/kg | | | |
|--|-----------------|-------------------|----------------------------------|---------------|------|-------------------------|-------------------------|-------------------------|--------------------------------|---|
| Location, Year (variety) | method | rate (g ai/ha) | stage at final application | | part | Kresoxim- methyl | 490M2 | 490M9 | Sum of parent + 490M9 | Report/Trial No., Reference Storage period |
| Germany Brandenburg Mehrow 1996 (Victoria) | Foliar spray | 2×125 | BBCH 49 | 0 29 43 | Root | <0.05 <0.05 <0.05 | <0.05 <0.05 <0.05 | <0.05 <0.05 <0.05 | <0.10 <0.10 <0.10 | Study: 1998/10321 Trial: ACK/17/96 Raunft, 1998, Kresoxim_126 Max. frozen storage: 13 month |
| Germany Schleswig- Holstein Bothkamp- Siek 1996 (Reka) | Foliar spray | 2×125 | BBCH 46 | 0 28 41 | Root | <0.05 <0.05 <0.05 | <0.05 <0.05 <0.05 | <0.05 <0.05 <0.05 | <0.10 <0.10 <0.10 | Study: 1998/10321 Trial: D05/05/96 Raunft, 1998, Kresoxim_126 Max. frozen storage: 13 month |

| | Treatment | Application | Growth | DALT | Crop | Residue Fou | und (ma/ka | 1) | | |
|---|-----------------|-------------------|----------------------------------|---------------|------|-------------------------|-------------------------|-------------------------|--------------------------------|---|
| Location, Year (variety) | method | rate (g ai/ha) | stage at final application | | part | Kresoxim- methyl | 490M2 | 490M9 | Sum of parent + 490M9 | Report/Trial No., Reference Storage period |
| Germany Bayern Holzen 1996 (Elan) | Foliar spray | 2×125 | BBCH 45 | 0 29 42 | Root | <0.05 <0.05 <0.05 | <0.05 <0.05 <0.05 | <0.05 <0.05 <0.05 | <0.10 <0.10 <0.10 | Study: 1998/10321 Trial: D07/04/96 Raunft, 1998, Kresoxim_126 Max. frozen storage: 13 month |
| Germany Nordrhein- Westfalen Bad Sassendorf 1996 (Elan) | Foliar spray | 2×125 | BBCH 47 | 0 27 41 | Root | <0.05 <0.05 <0.05 | <0.05 <0.05 <0.05 | <0.05 <0.05 <0.05 | <0.10 <0.10 <0.10 | Study: 1998/10321 Trial: D08/05/96 Raunft, 1998, Kresoxim_126 Max. frozen storage: 13 month |
| Germany Rheinland- Pfalz Meckenheim 1996 (Ribella) | Foliar spray | 2×125 | BBCH 45 | 0 27 41 | Root | <0.05 <0.05 <0.05 | <0.05 <0.05 <0.05 | <0.05 <0.05 <0.05 | <0.10 <0.10 <0.10 | Study: 1998/10321 Trial: DU2/14/96 Raunft, 1998, Kresoxim_126 Max. frozen storage: 13 month |
| Germany Brandenburg Brunne 1997 (Sonja) | Foliar spray | 2×125 | BBCH 47 | 0 29 42 | Root | <0.05 <0.05 <0.05 | <0.05 <0.05 <0.05 | <0.05 <0.05 <0.05 | <0.10 <0.10 <0.10 | Study: 1998/10320 Trial: ACK/04/97 Raunft, 1998, Kresoxim_127 Max. frozen storage: 5 month |
| Germany Bayern Holzen 1997 (Patricia) | Foliar spray | 2×125 | BBCH 45 | 0 28 41 | Root | <0.05 <0.05 <0.05 | <0.05 <0.05 <0.05 | <0.05 <0.05 <0.05 | <0.10 <0.10 <0.10 | Study: 1998/10320 Trial: D07/02/97 Raunft, 1998, Kresoxim_127 Max. frozen storage: 5 month |
| Germany Nordrhein- Westfalen Lippetal- Brockhausen 1997 (Sonja) | Foliar spray | 2×125 | BBCH 49 | 0 26 41 | Root | <0.05 <0.05 <0.05 | <0.05 <0.05 <0.05 | <0.05 <0.05 <0.05 | <0.10 <0.10 <0.10 | Study: 1998/10320 Trial: D08/02/97 Raunft, 1998, Kresoxim_127 Max. frozen storage: 5 month |
| France Pas de Calais Neuville 1997 (Roberta) | Foliar spray | 2×125 | BBCH 45 | 0 28 42 | Root | <0.05 <0.05 <0.05 | <0.05 <0.05 <0.05 | <0.05 <0.05 <0.05 | <0.10 <0.10 <0.10 | Study: 1998/10320 Trial: FR2/03/97 Raunft, 1998, Kresoxim_127 Max. frozen storage: 5 month |

| | Treatment | Application | Growth | DALT | Crop | Residue Fou | | | | |
|-------------|-----------|-------------|-------------|------|------|-------------|-------|-------|--------|------------------|
| Location, | method | rate | stage at | | part | Kresoxim- | 490M2 | 490M9 | Sum of | Report/Trial |
| Year | | (g ai/ha) | final | | | methyl | | | parent | No., Reference |
| (variety) | | | application | | | | | | + | Storage period |
| | | | | | | | | | 490M9 | |
| France | Foliar | 2×125 | BBCH 44 | 0 | Root | <0.05 | <0.05 | <0.05 | <0.10 | Study: |
| Cote D'Or | spray | | | 28 | | <0.05 | <0.05 | <0.05 | <0.10 | 1998/10320 |
| Rouvres-en- | | | | 41 | | <0.05 | <0.05 | <0.05 | <0.10 | Trial: FR3/03/97 |
| Plaine | | | | | | | | | | Raunft, 1998, |
| 1997 | | | | | | | | | | Kresoxim_127 |
| (Rebecca) | | | | | | | | | | Max. frozen |
| | | | | | | | | | | storage: 5 |
| | | | | | | | | | | month |

Cereal grains

Wheat

A total of four field trials were conducted with wheat in Southern France, Italy and Spain during the 2007 (Schroth & Martin, 2008, Kresoxim_128; Schroth, 2009, Kresoxim_129) growing seasons. Wheat plants received 2 spray applications of kresoxim-methyl at nominal rates of 125 g ai/ha with a 21±1 day interval between applications. Samples of wheat grain were collected at 35, 42 and 49 days after the last application. In case no mature grain could be harvested at the proposed PHI of 35 days, additional sampling was done when BBCH 89 (ripened grain) was reached (e.g. trial L070375 at 58 DALT). Residues of kresoxim-methyl were determined using method BASF method 445/0 with a limit of quantification of 0.01 mg/kg. Additionally, metabolites 490M2 and 490M9 were determined using method BASF method L0095/01 (identical to BASF method 350/4) with a limit of quantification of 0.01 mg/kg. Overall mean procedural recoveries for kresoxim-methyl and metabolites 490M2 and 490M9 in wheat grain spiked at 0.01–10 mg/kg were 88% (n=21), 79% (n=16) and 85% (n=18), respectively.

During the 2008 growing season, a total of four field trials were conducted with wheat in Southern France, Italy and Spain (Schroth & Martin, 2008, Kresoxim_130; Schroth, 2009, Kresoxim_131). Wheat plants received 2 spray applications of kresoximmethyl at nominal rates of 125 g ai/ha with an 11–21 day interval between applications. Samples of wheat grain were collected at 35, 42 and 49 days after the last application. Residues of kresoxim-methyl were determined using method BASF method L0010/01 with a limit of quantification of 0.01 mg/kg. Overall mean procedural recoveries for kresoxim-methyl in wheat grain spiked at 0.01– 1 mg/kg were 78% (n=3) Additionally, kresoxim-methyl and metabolites 490M2 and 490M9 were determined using method BASF method L0095/01 (identical to BASF method 350/4) with a limit of quantification of 0.01 mg/kg. Overall mean procedural recoveries for kresoxim-methyl and metabolites 490M2 and 490M9 in wheat grain spiked at 0.01–1 mg/kg were 82% (n=5), 78% (n=5) and 80% (n=5), respectively.

During the 2009 growing season, a total of four field trials were conducted with wheat in Belgium, Germany, the Netherlands and the United Kingdom (Erdmann, 2010, Kresoxim_132). Wheat plants received 2 spray applications of kresoxim-methyl at nominal rates of 125 g ai/ha with a 13–24 day interval between applications (second application at BBCH 69). Samples of wheat grain were collected at 28, 35 and 42 days after the last application. Kresoxim-methyl and metabolites 490M2 and 490M9 were determined using method BASF method L0095/01 (identical to BASF method 350/4) with a limit of quantification of 0.01 mg/kg. Overall mean procedural recoveries for kresoxim-methyl and metabolites 490M2 and 490M9 in wheat grain spiked at 0.01–0.05 mg/kg were 81% (n=6), 79% (n=6) and 81% (n=6), respectively.

| Location, Year (variety) | Treatment method | Application rate (g ai/ha) | Growth stage at final application | DALT | Crop part | Residue Fo Kresoxim -methyl | und (mg/kg 490M2 |) 490M9 | Sum of parent + 490M9 | Report/Trial No., Reference Storage period |
|---------------------------------|---------------------|----------------------------------|--|----------|--------------|-----------------------------------|---------------------|------------------|-----------------------------|--|
| Spain Sevilla Las Cabezas | Foliar spray | 2×125 (BAS 493 05 F) | BBCH 69 | 42 49 | Grain | 0.02 0.04 | < 0.01 < 0.01 | < 0.01 < 0.01 | 0.03 0.05 | Study: 2008/1043814/ 2009/1102142 |

Table 115 Residues of kresoxim-methyl in wheat grain following foliar treatment (cGAP: 2 × 125 g ai/ha; 35 day PHI)

| | Treatment | Application | Growth | DALT | Crop | Residue Fo | Deport/Trial | | | |
|-------------------------------------|-----------------|-------------------------|----------------------------------|----------------|-------|--------------------------|----------------------------|----------------------------|-----------------------------|--|
| Location, Year (variety) | method | rate (g ai/ha) | stage at final application | | part | Kresoxim -methyl | 490M2 | 490M9 | Sum of parent + 490M9 | Report/Trial No., Reference Storage period |
| 2007 (Diablon) | | 2×125 (BAS 494 04 F) | BBCH 69 | 42 49 | Grain | 0.02 | < 0.01 < 0.01 | < 0.01 < 0.01 | 0.03 | Trial: L070269 Schroth & Martin, 2008, Kresoxim_128; Schroth, 2009, Kresoxim_129 Max. frozen storage: 10 month |
| Italy Milano Cusago 2007 | Foliar spray | 2×125 (BAS 493 05 F) | BBCH 69 | 35 41 49 | Grain | < 0.01 < 0.01 0.01 | < 0.01 < 0.01 < 0.01 | < 0.01 < 0.01 < 0.01 | <0.02 <0.02 0.02 | Study: 2008/1043814/ 2009/1102142 Trial: L070270 |
| (Africa) | | 2×125 (BAS 494 04 F) | BBCH 69 | 35 41 49 | Grain | 0.01 0.01 0.01 | < 0.01 < 0.01 < 0.01 | < 0.01 < 0.01 < 0.01 | 0.02 0.02 0.02 | Schroth & Martin, 2008, Kresoxim_128; Schroth, 2009, Kresoxim_129 Max. frozen storage: 9 month |
| France Lot Sérignac 2007 | Foliar spray | 2×125 (BAS 493 05 F) | BBCH 69 | 35 42 49 | Grain | < 0.01 < 0.01 0.01 | < 0.01 < 0.01 < 0.01 | < 0.01 < 0.01 < 0.01 | <0.02 <0.02 0.02 | Study: 2008/1043814/ 2009/1102142 Trial: L070374 |
| (Apache) | | 2×125 (BAS 494 04 F) | BBCH 69 | 35 42 49 | Grain | 0.01 0.01 0.01 | < 0.01 < 0.01 < 0.01 | < 0.01 < 0.01 < 0.01 | 0.02 0.02 0.02 | Schroth & Martin, 2008, Kresoxim_128; Schroth, 2009, Kresoxim_129 Max. frozen storage: 9 month |
| France Vienne Messais 2007 | Foliar spray | 2×125 (BAS 493 05 F) | BBCH 69 | 58 | Grain | < 0.01 | < 0.01 | < 0.01 | <0.02 | Study: 2008/1043814/ 2009/1102142 Trial: L070375 |
| (Andalou) | | 2×125 (BAS 494 04 F) | BBCH 69 | 58 | Grain | < 0.01 | < 0.01 | < 0.01 | <0.02 | Schroth & Martin, 2008, Kresoxim_128; Schroth, 2009, Kresoxim_129 Max. frozen storage: 9 month |
| Spain Sevilla Utrera 2008 | Foliar spray | 2×125 | BBCH 69 | 42 | Grain | < 0.01 | - | - | - | Study: 2008/1090699/ 2009/1102117 Trial: L080161 |
| (Califa Sur) | | | | 42 49 | Grain | < 0.01 < 0.01 | < 0.01 | < 0.01 < 0.01 | <0.02 | Schroth & Martin, 2008, Kresoxim_130; Schroth, 2009, Kresoxim_131 Max. frozen storage: 5 month |
| Italy Milano Cusago | Foliar spray | 2×125 | BBCH 69 | 35 42 49 | Grain | <0.01 <0.01 <0.01 | - | - | - | Study: 2008/1090699/ 2009/1102117 |

| | Treatment | Application | Growth | DALT | Crop | Residue Fo | ound (mg/k | (q) | | |
|---|-----------------|-------------------------|----------------------------------|----------------|-------|-------------------------|-------------------------|--|---|---|
| Location, Year (variety) | method | rate (g ai/ha) | stage at final application | | part | Kresoxim -methyl | 490M2 | 490M9 | Sum of parent + 490M9 | Report/Trial No., Reference Storage period |
| 2008 (Isengrain) | | | аррисации | 35 42 49 | Grain | <0.01 <0.01 <0.01 | <0.01 <0.01 <0.01 | <0.01 <0.01 <0.01 | <0.02 <0.02 <0.02 <0.02 | Trial: L080162 Schroth & Martin, 2008, |
| | | | | | | 0.01 | 0.01 | 0.01 | <0.02 | Kresoxim_130; Schroth, 2009, Kresoxim_131 Max. frozen storage: 4 month |
| France Lot-et- Garonne Trentels | Foliar spray | 2×125 | BBCH 69 | 49 | Grain | < 0.01 | - | - | - | Study: 2008/1090699/ 2009/1102117 Trial: L080163 |
| 2008 (PR 2258) | | | | 49 | Grain | < 0.01 | < 0.01 | < 0.01 | < 0.03 | Schroth & Martin, 2008, Kresoxim_130; Schroth, 2009, Kresoxim_131 Max. frozen storage: 3 month |
| Spain Sevilla El Coronil 2008 | Foliar spray | 2×125 | BBCH 69 | 43 49 | Grain | < 0.01 < 0.01 | - | - | - | Study: 2008/1090699/ 2009/1102117 Trial: L080164 |
| (Yecora) | | | | 43 49 | Grain | < 0.01 < 0.01 | < 0.01 < 0.01 | < 0.01 <0.02 Mari Kres Schi Kres Max stor | Schroth & Martin, 2008, Kresoxim_130; Schroth, 2009, Kresoxim_131 Max. frozen storage: 5 month | |
| Germany Brandenbur g Lentzke | Foliar spray | 2×125 (BAS 493 06 F) | BBCH 69 | 41 | Grain | < 0.01 | < 0.01 | < 0.01 | <0.02 | Study: 2010/1006345 Trial: L090050 Erdmann, 2010, |
| 2008/09 (Herrmann) | | 2×125 (BAS 494 04 F) | BBCH 69 | 41 | Grain | < 0.01 | < 0.01 | < 0.01 | <0.02 | Kresoxim_132 Max. frozen storage: 10 month |
| The Netherlands Limburg Ottesum | Foliar spray | 2×125 (BAS 493 06 F) | BBCH 69 | 41 58 | Grain | < 0.01 < 0.01 | < 0.01 < 0.01 | < 0.01 < 0.01 | <0.02 <0.02 | Study: 2010/1006345 Trial: L090051 Erdmann, 2010, |
| 2008/09 (Limes) | | 2×125 (BAS 494 04 F) | BBCH 69 | 41 58 | Grain | < 0.01 < 0.01 | < 0.01 < 0.01 | < 0.01 < 0.01 | <0.02 <0.02 | Kresoxim_132 Max. frozen storage: 10 month |
| Belgium Brabant Kortenaken 2008/09 | Foliar spray | 2×125 (BAS 493 06 F) | BBCH 69 | 41 55 | Grain | < 0.01 < 0.01 | < 0.01 < 0.01 | < 0.01 < 0.01 | <0.02 <0.02 | Study: 2010/1006345 Trial: L090052 Erdmann, 2010, |
| (Molan) | | 2×125 (BAS 494 04 F) | BBCH 69 | 41 55 | Grain | < 0.01 < 0.01 | < 0.01 < 0.01 | < 0.01 < 0.01 | <0.02 <0.02 | Kresoxim_132 Max. frozen storage: 10 month |
| UK Essex Lawford | Foliar spray | 2×125 (BAS 493 06 F) | BBCH 69 | 42 | Grain | < 0.01 | < 0.01 | < 0.01 | <0.02 | Study: 2010/1006345 Trial: L090053 |

| Location, Year (variety) | Treatment | Application | Growth | DALT | Crop | Crop Residue Found (mg/kg) | | | | Report/Trial |
|--------------------------------|-----------|-------------------------|----------------------------------|------|-------|----------------------------|--------|--------|-----------------------------|---|
| | method | rate (g ai/ha) | stage at final application | | part | Kresoxim -methyl | 490M2 | 490M9 | Sum of parent + 490M9 | No., Reference Storage period |
| 2008/09 (Solistice) | | 2×125 (BAS 494 04 F) | BBCH 69 | 42 | Grain | < 0.01 | < 0.01 | < 0.01 | <0.02 | Erdmann, 2010, Kresoxim_132 Max. frozen storage: 10 month |

Barley

A total of four field trials were conducted with barley in Southern France, Italy and Spain during the 2007 (Schroth & Martin, 2008, Kresoxim_128; Schroth, 2009, Kresoxim_129) growing season. Barley plant received 2 spray applications of kresoxim-methyl at nominal rates of 125 g ai/ha with a 21±1 day interval between applications. Samples of barley grain were collected at 35, 42 and 49 days after the last application. Residues of kresoxim-methyl were determined using method BASF method 445/0 with a limit of quantification of 0.01 mg/kg. Additionally, metabolites 490M2 and 490M9 were determined using method BASF method L0095/01 (identical to BASF method 350/4) with a limit of quantification of 0.01 mg/kg. Overall mean procedural recoveries for kresoxim-methyl and metabolites 490M2 and 490M9 in wheat grain spiked at 0.01–10 mg/kg were 88% (n=21), 79% (n=16) and 85% (n=18), respectively.

During the 2008 growing seasons a total of six field trials were conducted with barley in Southern France, Greece, Italy and Spain (Schroth & Martin, 2009, Kresoxim_133; Schroth, 2009, Kresoxim_134). Barley plant received 2 spray applications of kresoxim-methyl at nominal rates of 125 g ai/ha with a 14–25 day interval between applications (second application at BBCH 69). Samples of barley grain were collected at about 35, 42 and 49 days after the last application. Residues of kresoxim-methyl were determined using method BASF method L0010/01 with a limit of quantification of 0.01 mg/kg. Overall mean procedural recoveries for kresoxim-methyl in wheat grain spiked at 0.01–1 mg/kg were 110% (n=3). Additionally, kresoxim-methyl and metabolites 490M2 and 490M9 were determined using method BASF method L0095/01 (identical to BASF method 350/4) with a limit of quantification of 0.01 mg/kg. Overall mean procedural recoveries for kresoxim-methyl and procedural recoveries for kresoxim-methyl and metabolites 490M2 and 490M9 were determined using method BASF method L0095/01 (identical to BASF method 350/4) with a limit of quantification of 0.01 mg/kg. Overall mean procedural recoveries for kresoxim-methyl and metabolites 490M2 and 490M9 in wheat grain spiked at 0.01 mg/kg. Overall mean procedural recoveries for kresoxim-methyl and metabolites 490M2 and 490M9 in wheat grain spiked at 0.01 mg/kg were 78% (n=3), 67% (n=3) and 71% (n=3), respectively.

| 1 | Treatment | Application | Growth stage at final application | DALT | Crop part | Residue Found (mg/kg) | | | | Denert/Trial |
|--|-----------------|-------------------------|--|----------|--------------|-----------------------|------------------|------------------|-----------------------------|--|
| Location, Year (variety) | method | rate (g ai/ha) | | | | Kresoxim -methyl | 490M2 | 490M9 | Sum of parent + 490M9 | Report/Trial No., Reference Storage period |
| Spain Sevilla Las Cabezas 2007 (Belen) | Foliar spray | 2×125 (BAS 494 04 F) | BBCH 69 | 42 49 | Grain | 0.03 0.02 | < 0.01 < 0.01 | < 0.01 < 0.01 | 0.04 0.03 | Study: 2008/1043814 / 2009/1102142 |
| | | 2×125 (BAS 493 05 F) | BBCH 69 | 42 49 | Grain | 0.03 0.03 | < 0.01 < 0.01 | < 0.01 < 0.01 | 0.04 0.04 | 2009/1102142 Trial: L070265 Schroth & Martin, 2008, Kresoxim_128; Schroth, 2009, Kresoxim_129 Max. frozen storage: 10 month |
| Spain Cadiz Puerto de Sta, Maria | Foliar spray | 2×125 (BAS 494 04 F) | BBCH 69 | 42 49 | Grain | 0.01 0.01 | < 0.01 < 0.01 | < 0.01 < 0.01 | 0.02 0.02 | Study: 2008/1043814 / 2009/1102142 |
| Sta. Maria 2007 (Cecilia) | | 2×125 (BAS 493 05 F) | BBCH 69 | 42 49 | Grain | 0.02 0.01 | < 0.01 < 0.01 | < 0.01 < 0.01 | 0.03 0.02 | Trial: L070266 Schroth & Martin, 2008, Kresoxim_128; Schroth, 2009, Kresoxim_129 Max. frozen storage: 8 month |

Table 116 Residues of kresoxim-methyl in barley grain following foliar treatment (cGAP: 2 × 125 g ai/ha; 35 day PHI)

| | Treatment | Application | Growth | DALT | Crop | Residue Fo | ound (mg/k | g) | | |
|------------------------------------|-----------------|-------------------------|----------------------------------|----------------|-------|--------------------------|----------------------------|----------------------------|-----------------------------|--|
| Location, Year (variety) | method | rate (g ai/ha) | stage at final application | | part | Kresoxim -methyl | 490M2 | 490M9 | Sum of parent + 490M9 | Report/Trial No., Reference Storage period |
| France Lot Goujounac 2007 | Foliar spray | 2×125 (BAS 494 04 F) | BBCH 69 | 35 42 49 | Grain | 0.06 0.05 0.05 | < 0.01 < 0.01 < 0.01 | 0.01 0.02 0.01 | 0.07 0.07 0.06 | Study: 2008/1043814 / 2009/1102142 |
| (Diadem) | | 2×125 (BAS 493 05 F) | BBCH 69 | 35 42 49 | Grain | 0.08 0.06 0.07 | 0.01 0.01 0.01 | < 0.01 < 0.01 < 0.01 | 0.09 0.07 0.08 | Trial: L070267 Schroth & Martin, 2008, Kresoxim_128; Schroth, 2009, Kresoxim_129 Max. frozen storage: 9 month |
| Italy Varese Cardano al | Foliar spray | 2×125 (BAS 494 04 F) | BBCH 69-71 | 35 42 49 | Grain | 0.01 0.02 0.01 | < 0.01 < 0.01 < 0.01 | < 0.01 < 0.01 < 0.01 | 0.02 0.03 0.02 | Study: 2008/1043814 / 2000/1103142 |
| Campo 2007 (Aliseo) | | 2×125 (BAS 493 05 F) | BBCH 69-71 | 35 42 49 | Grain | 0.02 0.02 0.02 | < 0.01 < 0.01 < 0.01 | < 0.01 < 0.01 < 0.01 | 0.03 0.03 0.03 | 2009/1102142 Trial: L070268 Schroth & Martin, 2008, Kresoxim_128; Schroth, 2009, Kresoxim_129 Max. frozen storage: 10 month |
| Spain Sevilla Utrera | Foliar spray | 2×125 | BBCH 69 | 42 49 | Grain | 0.01 0.01 | - | - | - | Study: 2008/1090698 / |
| 2008 (Belen) | | 2×125 | BBCH 69 | 42 49 | Grain | 0.04 0.03 | < 0.01 < 0.01 | < 0.01 < 0.01 | 0.05 0.04 | 2009/1102149 Trial: L080138 Schroth & Martin, 2009, Kresoxim_133; Schroth, 2009, Kresoxim_134 Max. frozen storage: 6 month |
| France Vienne Morton 2008 | Foliar spray | 2×125 | BBCH 69 | 42 49 | Grain | 0.01 < 0.01 | - | - | - | Study: 2008/1090698 / 2009/1102149 |
| (Prestige) | | 2×125 | BBCH 69 | 42 49 | Grain | 0.01 0.01 | < 0.01 < 0.01 | < 0.01 < 0.01 | 0.02 | Trial: L080139 Schroth & Martin, 2009, Kresoxim_133; Schroth, 2009, Kresoxim_134 Max. frozen storage: 4 month |
| Italy Milano Lazzate | Foliar spray | 2×125 | BBCH 71 | 34 42 49 | Grain | 0.01 < 0.01 < 0.01 | - | - | - | Study: 2008/1090698 / |

| | Treatment | Application | Growth | DALT | Crop | Residue Fo | und (ma/k | n) | | |
|---|-----------------|-------------------|----------------------------------|----------------|-------|----------------------------|----------------------------|----------------------------|-----------------------------|---|
| Location, Year (variety) | method | rate (g ai/ha) | stage at final application | DALI | part | Kresoxim -methyl | 490M2 | 490M9 | Sum of parent + 490M9 | Report/Trial No., Reference Storage period |
| 2008 (Amillis) | | 2×125 | BBCH 71 | 34 42 49 | Grain | 0.03 0.01 0.01 | < 0.01 < 0.01 < 0.01 | < 0.01 < 0.01 < 0.01 | 0.04 0.02 0.02 | 2009/1102149 Trial: L080140 Schroth & Martin, 2009, Kresoxim_133; Schroth, 2009, Kresoxim_134 Max. frozen storage: 5 month |
| Spain Cadiz Puerto de Sta. Maria | Foliar spray | 2×125 | BBCH 69 | 42 48 | Grain | 0.01 0.01 | - | - | - | Study: 2008/1090698 / 2009/1102149 |
| 2008 (Cecilia) | | 2×125 | BBCH 69 | 42 48 | Grain | 0.02 0.02 | < 0.01 < 0.01 | < 0.01 < 0.01 | 0.03 | Trial: L080141 Schroth & Martin, 2009, Kresoxim_133; Schroth, 2009, Kresoxim_134 Max. frozen storage: 6 month |
| Italy Varese Cardano al Campo | Foliar spray | 2×125 | BBCH 69 | 35 42 49 | Grain | < 0.01 < 0.01 < 0.01 | - | - | - | Study: 2008/1090698 / 2009/1102149 |
| 2008 (Scirocco) | | 2×125 | BBCH 69 | 35 42 49 | Grain | 0.01 0.02 0.01 | < 0.01 < 0.01 < 0.01 | < 0.01 < 0.01 < 0.01 | 0.02 0.03 0.02 | Trial: L080142 Schroth & Martin, 2009, Kresoxim_133; Schroth, 2009, Kresoxim_134 Max. frozen storage: 5 month |
| Greece Thessaloniki Lakkia 2008 | Foliar spray | 2×125 | BBCH 69 | 35 41 48 | Grain | 0.02 0.01 0.01 | - | - | - | Study: 2008/1090698 / 2009/1102149 |
| (Aliseo) | | 2×125 | BBCH 69 | 35 41 48 | Grain | 0.05 0.06 0.05 | < 0.01 < 0.01 < 0.01 | < 0.01 < 0.01 < 0.01 | 0.06 0.07 0.06 | Trial: L080143 Schroth & Martin, 2009, Kresoxim_133; Schroth, 2009, Kresoxim_134 Max. frozen storage: 5 month |

Tree nuts

Pecan

A total of six field trials were conducted with pecan nuts in the United States of America (Wofford, 1997, Kresoxim_135). Plants received 8 spray applications of kresoxim-methyl at nominal rates of 224 g ai/ha (per site two plots were treated with different spay volumes) with a 14 day interval for the first three applications, followed by a 21 day interval for the remaining five applications. An exception was one trial (RCN 96106) which had an 18 day interval between the second and third application and a 17 day interval between the third and fourth application. Samples of pecan nuts were collected 45 days after the last application, with the exception

of trial RCN 96109, where sample were collected at 44 DALT. In addition, at one site samples were collected at 35, 55, and 65 DALT. Residues of kresoxim-methyl (as 490M1) and metabolites 490M2 and 490M9 were determined using method D9611 (identical to BASF method 350/3) with a limit of quantification of 0.05 mg/kg. Overall mean procedural recoveries for kresoxim-methyl and metabolites 490M2 and 490M9 in pecan nut spiked at 0.05 and 1 mg/kg were 83% (n=6), 84% (n=6) and 77% (n=6), respectively.

| Lesetten | Treatment | Application | Growth | DALT | Crop | Residue Fou | und (mg/kg |) | | Dement/Trial |
|---|------------------------------------|---|----------------------------------|----------------------|----------------------------------|---|---|---|---|---|
| Location, Year (variety) | method | rate (g ai/ha) | stage at final application | | part | Kresoxim- methyl | 490M2 | 490M9 | Sum of parent + 490M9 | Report/Trial No., Reference Storage period |
| USA Mitchell County, GA 1997 | Foliar spray | 8×224 (468 L/ha) | Mature fruit | 35 45 55 65 | Whole pecan | <0.05 <0.05 <0.05 <0.05 | <0.05 <0.05 <0.05 <0.05 | <0.05 <0.05 <0.05 <0.05 | <0.10 <0.10 <0.10 <0.10 | Study: 1997/5064 Trial: RCN 96104 |
| (Stuart) | Foliar spray | 8×224 (2338 L/ha L/ha) | | 35 45 55 65 | Whole pecan | <0.05 <0.05 <0.05 <0.05 <0.05 | <0.05 <0.05 <0.05 <0.05 <0.05 | <0.05 <0.05 <0.05 <0.05 <0.05 | <0.10 <0.10 <0.10 <0.10 <0.10 | Wofford, 1997, Kresoxim_135 Max. frozen storage: 2 month |
| USA Houston County, AL 1997 (Cape Fear) | Foliar spray Foliar spray | 8×224 (468 L/ha) 8×224 (2338 L/ha L/ha) | Mature fruit | 45 45 | Whole pecan Whole pecan | <0.05 | <0.05 | <0.05 | <0.10 | Study: 1997/5064 Trial: RCN 96105 Wofford, 1997, Kresoxim_135 Max. frozen storage: 3 |
| USA Tift County, GA 1997 (Sumner) | Foliar spray Foliar spray | 8×224 (468 L/ha) 8×224 (2338 L/ha L/ha) | Mature fruit | 45 | Whole pecan Whole pecan | <0.05 | <0.05 | <0.05 | <0.10 | month Study: 1997/5064 Trial: RCN 96106 Wofford, 1997, Kresoxim_135 Max. frozen storage: 3 month |
| USA Washington | Foliar spray | 8×224 (468 L/ha) | Fruiting | 45 | Whole pecan | <0.05 | <0.05 | <0.05 | <0.10 | month Study: 1997/5064 |
| County, MS 1997 (Cheyanne) | Foliar spray | 8×224 (2338 L/ha L/ha) | 1 | 45 | Whole pecan | <0.05 | <0.05 | <0.05 | <0.10 | Wofford, 1997, Trial: RCN 96107 Kresoxim_135 Max. frozen storage: 2 month |
| USA Uvalde | Foliar spray | 8×224 (468 L/ha) | Nut past gel | 45 | Whole pecan | <0.05 | <0.05 | <0.05 | <0.10 | Study: 1997/5064 |
| County, TX 1997 (Wichita) | Foliar spray | 8×224 (2338 L/ha L/ha) | | 45 | Whole pecan | <0.05 | <0.05 | <0.05 | <0.10 | Trial: RCN 96108 Wofford, 1997, Kresoxim_135 Max. frozen storage: 3 month |
| USA Lubbock | Foliar spray | 8×224 (468 L/ha) | Mature fruit | 44 | Whole pecan | <0.05 | <0.05 | <0.05 | <0.10 | Study: 1997/5064 |
| County, TX 1997 (Western Schley) | Foliar spray | 8×224 (2338 L/ha L/ha) | | 44 | Whole pecan | <0.05 | <0.05 | <0.05 | <0.10 | Trial: RCN 96109 Wofford, 1997, Kresoxim_135 Max. frozen storage: 2 month |

Table 117 Residues of kresoxim-methyl in pecan nuts following foliar treatment (cGAP: 3 × 168 g ai/ha; 45 day PHI)

Animal feed stuffs

Sugar beet tops

During the 1996 and 1997 growing seasons, a total of ten field trials were conducted with sugar beet in Germany and France (Raunft, 1998, Kresoxim_126; Raunft, 1998, Kresoxim_127). Plants received 2 spray applications of kresoxim-methyl at nominal rates of 125 g ai/ha with a 21 day interval between applications. Samples of sugar beet were collected at 0, 27–29 and 41–43 days after the last application. Residues of kresoxim-methyl (as 490M1) and metabolites 490M2 and 490M9 were determined using method BASF method 350/3 with a limit of quantification of 0.05 mg/kg. Overall mean procedural recoveries for kresoxim-methyl (as 490M1) and metabolites 490M2 and 490M9 and 91% (n=10), respectively.

| | Treatment | Application | Growth | DALT | Crop | Residue Fou | ind (ma/ka |) | | |
|---|-----------------|-------------------|----------------------------------|---------------|------|----------------------|-------------------------|-------------------------|-------------------------------|--|
| Location, Year (variety) | method | rate (g ai/ha) | stage at final application | DALT | part | Kresoxim- methyl | 490M2 | 490M9 | Sum as parent equiv. | Report/Trial No., Reference Storage period |
| Germany Brandenburg Mehrow 1996 (Victoria) | Foliar spray | 2×125 | BBCH 49 | 0 29 43 | Тор | 1.15 0.17 0.07 | <0.05 <0.05 0.06 | 0.08 0.13 0.14 | 1.3 0.35 0.27 | Study: 1998/10321 Trial: ACK/17/96 Raunft, 1998, Kresoxim_126 Max. frozen storage: 13 month |
| Germany Schleswig- Holstein Bothkamp- Siek 1996 (Reka) | Foliar spray | 2×125 | BBCH 46 | 0 28 41 | Тор | 1.81 0.35 0.08 | <0.05 <0.05 <0.05 | 0.14 0.07 <0.05 | 2.0 0.47 0.18 | Study: 1998/10321 Trial: D05/05/96 Raunft, 1998, Kresoxim_126 Max. frozen storage: 13 month |
| Germany Bayern Holzen 1996 (Elan) | Foliar spray | 2×125 | BBCH 45 | 0 29 42 | Тор | 2.20 0.17 0.10 | <0.05 <0.05 <0.05 | <0.05 <0.05 <0.05 | 2.3 0.27 0.20 | Study: 1998/10321 Trial: D07/04/96 Raunft, 1998, Kresoxim_126 Max. frozen storage: 13 month |
| Germany Nordrhein- Westfalen Bad Sassendorf 1996 (Elan) | Foliar spray | 2×125 | BBCH 47 | 0 27 41 | Тор | 2.17 0.14 0.06 | <0.05 <0.05 <0.05 | 0.11 0.09 0.07 | 2.3 0.28 0.18 | Study: 1998/10321 Trial: D08/05/96 Raunft, 1998, Kresoxim_126 Max. frozen storage: 13 month |
| Germany Rheinland- Pfalz Meckenheim 1996 (Ribella) | Foliar spray | 2×125 | BBCH 45 | 0 27 41 | Тор | 2.35 0.28 0.07 | <0.05 <0.05 <0.05 | 0.11 0.14 0.07 | 2.5 0.47 0.19 | Study: 1998/10321 Trial: DU2/14/96 Raunft, 1998, Kresoxim_126 Max. frozen storage: 13 month |

Table 118 Residues of kresoxim-methyl in sugar beet tops following foliar treatment (cGAP: 2 × 125 g ai/ha; 28 days PHI)

| | Treatment | Application | Growth | DALT | Crop | Residue Fou | und (ma/ka |) | | |
|---|-----------------|-------------------|----------------------------------|---------------|------|-----------------------|-----------------------|-----------------------|-------------------------------|---|
| Location, Year (variety) | method | rate (g ai/ha) | stage at final application | | part | Kresoxim- methyl | 490M2 | 490M9 | Sum as parent equiv. | Report/Trial No., Reference Storage period |
| Germany Brandenburg Brunne 1997 (Sonja) | Foliar spray | 2×125 | BBCH 47 | 0 29 42 | Тор | 2.2 0.10 0.14 | 0.11 0.06 0.10 | 0.28 0.16 <0.05 | 2.6 0.32 0.29 | Study: 1998/10320 Trial: ACK/04/97 Raunft, 1998, Kresoxim_127 Max. frozen storage: 5 month |
| Germany Bayern Holzen 1997 (Patricia) | Foliar spray | 2×125 | BBCH 45 | 0 28 41 | Тор | 1.57 0.20 0.14 | <0.05 0.08 0.07 | 0.07 0.19 0.19 | 1.7 0.47 0.40 | Study: 1998/10320 Trial: D07/02/97 Raunft, 1998, Kresoxim_127 Max. frozen storage: 5 month |
| Germany Nordrhein- Westfalen Lippetal- Brockhausen 1997 (Sonja) | Foliar spray | 2×125 | BBCH 49 | 0 26 41 | Тор | 1.5 0.10 <0.05 | <0.05 0.07 0.09 | 0.11 0.19 0.24 | 1.6 0.36 0.38 | Study: 1998/10320 Trial: D08/02/97 Raunft, 1998, Kresoxim_127 Max. frozen storage: 5 month |
| France Pas de Calais Neuville 1997 (Roberta) | Foliar spray | 2×125 | BBCH 45 | 0 28 42 | Тор | 2.13 0.17 <0.05 | 0.09 0.09 0.07 | 0.29 0.23 0.18 | 2.5 0.49 0.30 | Study: 1998/10320 Trial: FR2/03/97 Raunft, 1998, Kresoxim_127 Max. frozen storage: 5 month |
| France Cote D'Or Rouvres-en- Plaine 1997 (Rebecca) | Foliar spray | 2×125 | BBCH 44 | 0 28 41 | Тор | 2.1 0.13 <0.05 | 0.10 0.15 0.14 | 0.31 0.38 0.34 | 2.5 0.66 0.53 | Study: 1998/10320 Trial: FR3/03/97 Raunft, 1998, Kresoxim_127 Max. frozen storage: 5 month |

Wheat forage (whole plant)

Residues of kresoxim-methyl in wheat forage from field trials performed during the 2007 and 2008 growing seasons in Southern France, Italy and Spain (Schroth & Martin, 2008, Kresoxim_128; Schroth, 2009, Kresoxim_129; Schroth & Martin, 2008, Kresoxim_130; Schroth, 2009, Kresoxim_131), well as in 2009 growing season in the Netherlands, Belgium, UK and Germany (Erdmann, 2010, Kresoxim_132) were determined using BASF method L0010/01 (method 445/0) or BASF method L0095/01 (method 350/4), each with a limit of quantification of 0.01 mg/kg. For method L0010/01, the overall mean procedural recoveries for kresoxim-methyl in whole plant (forage) spiked at 0.01–10 mg/kg were 91% (n=3). For method 350/4, overall mean procedural recoveries for kresoxim-methyl (as 490M1) and metabolites 490M2 and 490M9 in wheat forage spiked at 0.01–2.5 mg/kg were 86% (n=14), 85% (n=14) and 72% (n=14), respectively.

| | Treatment | Application | Growth | DALT | Crop | Residue For | und (mg/ka | a) | | |
|---|-----------------|----------------------------|----------------------------------|------|----------------|---------------------|------------|-------|----------------------------|--|
| Location, Year (variety) | method | rate (g ai/ha) | stage at final application | | part | Kresoxim- methyl | 490M2 | 490M9 | Sum as parent equiv. | Report/Trial No., Reference Storage period |
| Spain Sevilla Las Cabezas 2007 | Foliar spray | 2×125 (BAS 493 05 F) | BBCH 69 | 0 | Whole plant | 2.4 | 0.02 | 0.13 | 2.6 | Study: 2008/1043814/ 2009/1102142 Trial: L070269 |
| (Diablon) | | 2×125 (BAS 494 04 F) | BBCH 69 | 0 | Whole plant | 2.9 | 0.02 | 0.05 | 3.0 | Schroth & Martin, 2008, Kresoxim_128; Schroth, 2009, Kresoxim_129 Max. frozen storage: 10 month |
| Italy Milano Cusago 2007 | Foliar spray | 2×125 (BAS 493 05 F) | BBCH 69 | 0 | Whole plant | 1.1 | 0.02 | 0.07 | 1.2 | Study: 2008/1043814/ 2009/1102142 Trial: L070270 |
| (Africa) | | 2×125 (BAS 494 04 F) | BBCH 69 | 0 | Whole plant | 0.94 | 0.01 | 0.06 | 1.0 | Schroth & Martin, 2008, Kresoxim_128; Schroth, 2009, Kresoxim_129 Max. frozen storage: 9 month |
| France Lot Sérignac 2007 | Foliar spray | 2×125 (BAS 493 05 F) | BBCH 69 | 0 | Whole plant | 1.6 | 0.01 | 0.09 | 1.7 | Study: 2008/1043814/ 2009/1102142 Trial: L070374 |
| (Apache) | | 2×125 (BAS 494 04 F) | BBCH 69 | 0 | Whole plant | 1.1 | 0.01 | 0.05 | 1.2 | Schroth & Martin, 2008, Kresoxim_128; Schroth, 2009, Kresoxim_129 Max. frozen storage: 9 month |
| France Vienne Messais 2007 | Foliar spray | 2×125 (BAS 493 05 F) | BBCH 69 | 0 | Whole plant | 1.8 | 0.02 | 0.03 | 1.9 | Study: 2008/1043814/ 2009/1102142 Trial: L070375 |
| (Andalou) | | 2×125 (BAS 494 04 F) | BBCH 69 | 0 | Whole plant | 2.5 | < 0.01 | 0.03 | 2.5 | Schroth & Martin, 2008, Kresoxim_128; Schroth, 2009, Kresoxim_129 Max. frozen storage: 9 month |
| Spain Sevilla Utrera | Foliar spray | 2×125 | BBCH 69 | 0 | Whole plant | 3.2 | - | - | - | Study: 2008/1090699/ 2009/1102117 |

Table 119 Residues of kresoxim-methyl in wheat forage (whole plant) following foliar treatment (cGAP: 2 × 125 g ai/ha; 35 day PHI)

| | Treatment | Application | Growth | DALT | Crop | Residue Fo | und (mg/kg | 1) | | D I/T I |
|--|-----------------|----------------------------|----------------------------------|------|----------------|---------------------|------------|-------|----------------------------|---|
| Location, Year (variety) | method | rate (g ai/ha) | stage at final application | | part | Kresoxim- methyl | 490M2 | 490M9 | Sum as parent equiv. | Report/Trial No., Reference Storage period |
| 2008 (Califa Sur) | | | | 0 | Whole plant | 3.0 | 0.01 | 0.09 | 3.1 | Trial: L080161 Schroth & Martin, 2008, Kresoxim_130; Schroth, 2009, Kresoxim_131 Max. frozen storage: 5 month |
| Italy Milano Cusago 2008 | Foliar spray | 2×125 | BBCH 69 | 0 | Whole plant | 1.4 | - | - | - | Study: 2008/1090699/ 2009/1102117 Trial: L080162 |
| (Isengrain) | | | | 0 | Whole plant | 1.4 | 0.02 | 0.07 | 1.5 | Schroth & Martin, 2008, Kresoxim_130; Schroth, 2009, Kresoxim_131 Max. frozen storage: 4 month |
| France Lot-et- Garonne Trentels | Foliar spray | 2×125 | BBCH 69 | 0 | Whole plant | 1.9 | - | - | - | Study: 2008/1090699/ 2009/1102117 Trial: L080163 |
| 2008 (PR 2258) | | | | 0 | Whole plant | 1.8 | 0.02 | 0.09 | 1.9 | Schroth & Martin, 2008, Kresoxim_130; Schroth, 2009, Kresoxim_131 Max. frozen storage: 3 month |
| Spain Sevilla El Coronil 2008 | Foliar spray | 2×125 | BBCH 69 | 0 | Whole plant | 2.8 | - | - | - | Study: 2008/1090699/ 2009/1102117 Trial: L080164 |
| (Yecora) | | | | 0 | Whole plant | 3.0 | 0.03 | 0.18 | 3.2 | Schroth & Martin, 2008, Kresoxim_130; Schroth, 2009, Kresoxim_131 Max. frozen storage: 5 month |
| Germany Brandenburg Lentzke 2008/09 | Foliar spray | 2×125 (BAS 493 06 F) | BBCH 69 | 0 | Whole plant | 1.6 | 0.02 | 0.07 | 1.7 | Study: 2010/1006345 Trial: L090050 Erdmann, 2010, |
| (Herrmann) | | 2×125 (BAS 494 04 F) | BBCH 69 | 0 | Whole plant | 1.4 | 0.02 | 0.07 | 1.5 | Kresoxim_132 Max. frozen storage: 10 month |
| The Netherlands Limburg Ottesum | Foliar spray | 2×125 (BAS 493 06 F) | BBCH 69 | 0 | Whole plant | 2.0 | 0.03 | 0.13 | 2.2 | Study: 2010/1006345 Trial: L090051 Erdmann, 2010, |
| Ottesum 2008/09 (Limes) | | (BAS 494 | | 0 | Whole plant | 2.8 | 0.02 | 0.11 | 2.9 | Kresoxim_132 Max. frozen storage: 10 month |

| Leastion | Treatment | Application | Growth | DALT | Crop | Residue Fou | und (mg/kg | J) | | Demont/Trial |
|---|-----------------|----------------------------|----------------------------------|------|----------------|---------------------|------------|-------|----------------------------|--|
| Location, Year (variety) | method | rate (g ai/ha) | stage at final application | | part | Kresoxim- methyl | 490M2 | 490M9 | Sum as parent equiv. | Report/Trial No., Reference Storage period |
| Belgium Brabant Kortenaken 2008/09 | Foliar spray | 2×125 (BAS 493 06 F) | BBCH 69 | 0 | Whole plant | 2.4 | 0.04 | 0.17 | 2.6 | Study: 2010/1006345 Trial: L090052 Erdmann, 2010, |
| (Molan) | | 2×125 (BAS 494 04 F) | BBCH 69 | 0 | Whole plant | 2.4 | 0.03 | 0.13 | 2.6 | Kresoxim_132 Max. frozen storage: 10 month |
| UK Essex Lawford 2008/09 | Foliar spray | 2×125 (BAS 493 06 F) | BBCH 69 | 0 | Whole plant | 1.9 | 0.03 | 0.14 | 2.1 | Study: 2010/1006345 Trial: L090053 Erdmann, 2010, |
| (Solistice) | | 2×125 (BAS 494 04 F) | BBCH 69 | 0 | Whole plant | 2.2 | 0.02 | 0.1 | 2.3 | Kresoxim_132 Max. frozen storage: 10 month |

Barley forage (whole plant)

Residues of kresoxim-methyl in barley forage from field trials performed during the 2007 and 2008 growing seasons in Southern France, Greece, Italy and Spain (Schroth & Martin, 2008, Kresoxim_128; Schroth, 2009, Kresoxim_129; Schroth & Martin, 2009, Kresoxim_133; Schroth, 2009, Kresoxim_134) were determined using BASF method L0010/01 (method 445/0) or BASF method L0095/01 (method 350/4), each with a limit of quantification of 0.01 mg/kg. For method L0010/01, the overall mean procedural recoveries for kresoxim-methyl in whole plant (forage) spiked at 0.01–10 mg/kg were 91% (n=3). For method 350/4, overall mean procedural recoveries for kresoxim-methyl (as 490M1) and metabolites 490M2 and 490M9 in barley forage spiked at 0.01–2.5 mg/kg were 81% (n=7), 82% (n=7) and 80% (n=7), respectively.

| Table 120 Residues of kresoxim-methyl in barley forage following foliar treatment (cGAP: 2 × 125 g ai/ha; 35 | day PHI) |
|--|----------|
| | |

| | Treatment | Application | Growth | DALT | Crop | Residue Fou | ind (mg/kg |) | | |
|---|-----------------|-------------------------|----------------------------------|------|----------------|---------------------|------------|-------|-------------------------------|--|
| Location, Year (variety) | method | rate (g ai/ha) | stage at final application | | part | Kresoxim- methyl | 490M2 | 490M9 | Sum as parent equiv. | Report/Trial No., Reference Storage period |
| Spain Sevilla Las Cabezas 2007 | Foliar spray | 2×125 (BAS 494 04 F) | BBCH 69 | 0 | Whole plant | 2.8 | <0.01 | 0.01 | 2.8 | Study: 2008/1043814/ 2009/1102142 Trial: L070265 |
| (Belen) | | 2×125 (BAS 493 05 F) | BBCH 69 | 0 | Whole plant | 4.0 | 0.02 | 0.06 | 4.1 | Schroth & Martin, 2008, Kresoxim_128; Schroth, 2009, Kresoxim_129 Max. frozen storage: 10 month |
| Spain Cadiz Puerto de Sta, Maria | Foliar spray | 2×125 (BAS 494 04 F) | BBCH 69 | 0 | Whole plant | 3.0 | 0.02 | 0.04 | 3.1 | Study: 2008/1043814/ 2009/1102142 Trial: L070266 |
| 2007 (Cecilia) | | 2×125 (BAS 493 05 F) | BBCH 69 | 0 | Whole plant | 2.3 | 0.03 | 0.04 | 2.4 | Schroth & Martin, 2008, Kresoxim_128; Schroth, 2009, Kresoxim_129 Max. frozen storage: 8 month |

834

| | Treatment | Application | Growth | DALT | Crop | Residue Fou | und (ma/ka |) | | |
|-------------------------------------|-------------------------|-------------------------|----------------------------------|----------------|----------------|---------------------|------------|-------|---|--|
| Location, Year (variety) | method | rate (g ai/ha) | stage at final application | | part | Kresoxim- methyl | 490M2 | 490M9 | Sum as parent equiv. | Report/Trial No., Reference Storage period |
| France Lot Goujounac 2007 | Foliar spray | 2×125 (BAS 494 04 F) | BBCH 69 | 0 | Whole plant | 1.5 | 0.03 | 0.01 | 1.5 | Study: 2008/1043814/ 2009/1102142 Trial: L070267 |
| (Diadem) | | 2×125 (BAS 493 05 F) | BBCH 69 | 0 | Whole plant | 2.0 | 0.04 | 0.04 | 2.1 | Schroth & Martin, 2008, Kresoxim_128; Schroth, 2009, Kresoxim_129 Max. frozen storage: 9 month |
| Varese spray Cardano al Campo | 2×125 (BAS 494 04 F) | BBCH 69- 71 | 0 | Whole plant | 1.8 | 0.01 | 0.06 | 1.9 | Study: 2008/1043814/ 2009/1102142 Trial: L070268 | |
| 2007 (Aliseo) | | 2×125 (BAS 493 05 F) | BBCH 69- 71 | 0 | Whole plant | 1.7 | 0.01 | 0.05 | 1.8 | Schroth & Martin, 2008, Kresoxim_128; Schroth, 2009, Kresoxim_129 Max. frozen storage: 10 month |
| Spain Sevilla Utrera 2008 | Foliar spray | 2×125 | BBCH 69 | 0 | Whole plant | 2.8 | - | - | - | Study: 2008/1090698/ 2009/1102149 Trial: L080138 |
| (Belen) | | 2×125 | BBCH 69 | 0 | Whole plant | 3.6 | 0.02 | 0.07 | 3.7 | Schroth & Martin, 2009, Kresoxim_133; Schroth, 2009, Kresoxim_134 Max. frozen storage: 6 month |
| France Vienne Morton 2008 | Foliar spray | 2×125 | BBCH 69 | 0 | Whole plant | 2.3 | - | - | - | Study: 2008/1090698/ 2009/1102149 Trial: L080139 |
| (Prestige) | | 2×125 | BBCH 69 | 0 | Whole plant | 2.5 | 0.02 | 0.07 | 2.6 | Schroth & Martin, 2009, Kresoxim_133; Schroth, 2009, Kresoxim_134 Max. frozen storage: 4 month |
| Italy Milano Lazzate 2008 | Foliar spray | 2×125 | BBCH 71 | 0 | Whole plant | 1.8 | - | - | - | Study: 2008/1090698/ 2009/1102149 Trial: L080140 |
| 2008 (Amillis) | | 2×125 | BBCH 71 | 0 | Whole plant | 2.5 | 0.04 | 0.13 | 2.7 | Schroth & Martin, 2009, Kresoxim_133; Schroth, 2009, Kresoxim_134 Max. frozen storage: 5 month |

| | Treatment | Application | Growth | DALT | Crop | Residue Fou | ind (mg/kg |) | | |
|---|-----------------|-------------------|----------------------------------|------|----------------|---------------------|------------|-------|-------------------------------|---|
| Location, Year (variety) | method | rate (g ai/ha) | stage at final application | | part | Kresoxim- methyl | 490M2 | 490M9 | Sum as parent equiv. | Report/Trial No., Reference Storage period |
| Spain Foliar Cadiz spray Puerto de Sta. Maria 2008 (Cecilia) | Foliar spray | 2×125 | BBCH 69 | 0 | Whole plant | 2.9 | - | - | - | Study: 2008/1090698/ 2009/1102149 Trial: L080141 |
| | | 2×125 | BBCH 69 | 0 | Whole plant | 3.2 | 0.06 | 0.16 | 3.4 | Schroth & Martin, 2009, Kresoxim_133; Schroth, 2009, Kresoxim_134 Max. frozen storage: 6 month |
| Italy Varese Cardano al Campo | Foliar spray | 2×125 | BBCH 69 | 0 | Whole plant | 1.4 | - | - | - | Study: 2008/1090698/ 2009/1102149 Trial: L080142 |
| 2008 (Scirocco) | | 2×125 | BBCH 69 | 0 | Whole plant | 2.3 | 0.03 | 0.16 | 2.5 | Schroth & Martin, 2009, Kresoxim_133; Schroth, 2009, Kresoxim_134 Max. frozen storage: 5 month |
| Greece Thessaloniki Lakkia 2008 | Foliar spray | 2×125 | BBCH 69 | 0 | Whole plant | 2.6 | - | - | - | Study: 2008/1090698/ 2009/1102149 Trial: L080143 |
| (Aliseo) | | 2×125 | BBCH 69 | 0 | Whole plant | 5.0 | 0.03 | 0.09 | 5.1 | Schroth & Martin, 2009, Kresoxim_133; Schroth, 2009, Kresoxim_134 Max. frozen storage: 5 month |

Wheat straw

Residues of kresoxim-methyl in wheat straw from field trials performed during the 2007 and 2008 growing seasons in Southern France, Italy and Spain (Schroth & Martin, 2008, Kresoxim_128; Schroth, 2009, Kresoxim_129; Schroth & Martin, 2008, Kresoxim_130; Schroth, 2009, Kresoxim_131), well as in 2009 growing season in the Netherlands, Belgium, UK and Germany (Erdmann, 2010, Kresoxim_132) were determined using BASF method L0010/01 (method 445/0) or BASF method L0095/01 (method 350/4), each with a limit of quantification of 0.01 mg/kg. For method L0010/01, the overall mean procedural recoveries for kresoxim-methyl in straw spiked at 0.01–1 mg/kg were 85% (n=3). For method 350/4, overall mean procedural recoveries for kresoxim-methyl (as 490M1) and metabolites 490M2 and 490M9 in wheat straw spiked at 0.01–1 mg/kg were 84% (n=12), 73% (n=12) and 72% (n=12), respectively.

| Table 121 Residues of kresoxim-meth | yl in wheat straw following | foliar treatment (cGAP: 2 × 125 | g ai/ha; 35 day PHI) |
|-------------------------------------|-----------------------------|---------------------------------|----------------------|
| | | | |

| Location | Treatment | Application | Growth | DALT | Crop | Residue F | ound (mg/k | :g) | | Deport/Trial |
|--------------------------------|-----------|-------------------|----------------------------------|------|-------|---------------------|------------|-------|-----------------------------|--|
| Location, Year (variety) | method | rate (g ai/ha) | stage at final application | | part | Kresoxim -methyl | 490M2 | 490M9 | Sum of parent + 490M9 | Report/Trial No., Reference Storage period |
| Spain | Foliar | 2×125 (BAS | BBCH 69 | 42 | Straw | 0.52 | 0.01 | 0.05 | 0.57 | Study: |
| Sevilla | spray | 493 05 F) | | 49 | | 0.49 | 0.02 | 0.10 | 0.59 | 2008/1043814 |
| Las Cabezas | | | | | | | | | | / |

836

| | Treatment | Application | Growth | DALT | Crop | Residue F | ound (mg/ | ka) | | |
|-------------------------------------|-----------------|-------------------------|----------------------------------|----------------|-------|----------------------|----------------------|----------------------------|-----------------------------|--|
| Location, Year (variety) | method | rate (g ai/ha) | stage at final application | | part | Kresoxim -methyl | 490M2 | 490M9 | Sum of parent + 490M9 | Report/Trial No., Reference Storage period |
| 2007 (Diablon) | | 2×125 (BAS 494 04 F) | BBCH 69 | 42 49 | Straw | 0.45 0.36 | 0.01 < 0.01 | 0.04 0.02 | 0.49 0.38 | 2009/1102142 Trial: L070269 Schroth & Martin, 2008, Kresoxim_128; Schroth, 2009, Kresoxim_129 Max. frozen storage: 10 month |
| Italy Milano Cusago 2007 | Foliar spray | 2×125 (BAS 493 05 F) | BBCH 69 | 35 41 49 | Straw | 0.10 0.06 0.04 | 0.03 0.04 0.03 | 0.03 0.02 < 0.01 | 0.13 0.08 0.05 | Study: 2008/1043814 / 2009/1102142 |
| (Africa) | | 2×125 (BAS 494 04 F) | BBCH 69 | 35 41 49 | Straw | 0.11 0.08 0.06 | 0.03 0.03 0.03 | 0.04 0.02 0.01 | 0.15 0.10 0.07 | Trial: L070270 Schroth & Martin, 2008, Kresoxim_128; Schroth, 2009, Kresoxim_129 Max. frozen storage: 9 month |
| France Lot Sérignac 2007 | Foliar spray | 2×125 (BAS 493 05 F) | BBCH 69 | 35 42 49 | Straw | 0.25 0.25 0.24 | 0.06 0.03 0.04 | 0.05 0.04 0.04 | 0.30 0.29 0.28 | Study: 2008/1043814 / 2009/1102142 |
| (Apache) | | 2×125 (BAS 494 04 F) | BBCH 69 | 35 42 49 | Straw | 0.06 0.10 0.06 | 0.03 0.03 0.03 | < 0.01 < 0.01 < 0.01 | 0.07 0.11 0.07 | Trial: L070374 Schroth & Martin, 2008, Kresoxim_128; Schroth, 2009, Kresoxim_129 Max. frozen storage: 9 month |
| France Vienne Messais 2007 | Foliar spray | 2×125 (BAS 493 05 F) | BBCH 69 | 58 | Straw | 0.11 | 0.02 | < 0.06 | 0.17 | Study: 2008/1043814 / 2009/1102142 |
| (Andalou) | | 2×125 (BAS 494 04 F) | BBCH 69 | 58 | Straw | 0.13 | 0.02 | < 0.01 | 0.14 | Trial: L070375 Schroth & Martin, 2008, Kresoxim_128; Schroth, 2009, Kresoxim_129 Max. frozen storage: 9 month |
| Spain Sevilla Utrera | Foliar spray | 2×125 | BBCH 69 | 42 49 | Straw | 0.38 0.30 | - | - | - | Study: 2008/1090699 / |

| | Treatment | Application | Growth | DALT | Crop | Residue F | ound (mg/ | ka) | | |
|--|-----------------|-------------------------|----------------------------------|----------------|-------|-------------------------|------------------------|--------------------------|-----------------------------|---|
| Location, Year (variety) | method | rate (g ai/ha) | stage at final application | | part | Kresoxim -methyl | 490M2 | 490M9 | Sum of parent + 490M9 | Report/Trial No., Reference Storage period |
| 2008 (Califa Sur) | | | | 42 49 | Straw | 1.5 1.4 | 0.04 | 0.05 | 1.6 1.4 | 2009/1102117 Trial: L080161 Schroth & Martin, 2008, Kresoxim_130; Schroth, 2009, Kresoxim_131 Max. frozen storage: 5 month |
| Italy Milano Cusago 2008 | Foliar spray | 2×125 | BBCH 69 | 35 42 49 | Straw | <0.01 <0.01 <0.01 | - | - | - | Study: 2008/1090699 / 2009/1102117 |
| (Isengrain) | | | | 35 42 49 | Straw | 0.04 0.03 0.02 | < 0 01 0.01 0.01 | < 0.01 0.01 < 0.01 | 0.05 0.04 0.03 | Trial: L080162 Schroth & Martin, 2008, Kresoxim_130; Schroth, 2009, Kresoxim_131 Max. frozen storage: 4 month |
| France Lot-et- Garonne Trentels | Foliar spray | 2×125 | BBCH 69 | 49 | Straw | 0.03 | - | - | - | Study: 2008/1090699 / 2009/1102117 |
| 2008 (PR 2258) | | | | 49 | Straw | 0.07 | 0.01 | < 0.01 | 0.08 | Trial: L080163 Schroth & Martin, 2008, Kresoxim_130; Schroth, 2009, Kresoxim_131 Max. frozen storage: 3 month |
| Spain Sevilla El Coronil 2008 | Foliar spray | 2×125 | BBCH 69 | 43 49 | Straw | 0.22 0.16 | - | - | - | Study: 2008/1090699 / 2009/1102117 |
| (Yecora) | | | | 43 49 | Straw | 0.51 0.52 | 0.04 0.03 | 0.04 0.02 | 0.55 0.54 | Trial: L080164 Schroth & Martin, 2008, Kresoxim_130; Schroth, 2009, Kresoxim_131 Max. frozen storage: 5 month |
| Germany Brandenburg Lentzke 2008/09 | Foliar spray | 2×125 (BAS 493 06 F) | BBCH 69 | 41 | Straw | 0.32 | 0.06 | 0.05 | 0.37 | Study: 2010/1006345 Trial: L090050 Erdmann, 2010, |
| (Herrmann) | | 2×125 (BAS 494 04 F) | BBCH 69 | 41 | Straw | 0.17 | 0.03 | 0.03 | 0.20 | Kresoxim_132 Max. frozen storage: 10 month |
| The Netherlands Limburg | Foliar spray | 2×125 (BAS 493 06 F) | BBCH 69 | 41 58 | Straw | 0.10 0.07 | 0.03 0.02 | 0.05 < 0.01 | 0.15 0.08 | Study: 2010/1006345 Trial: L090051 |

| Leastion | Treatment | Application | Growth | DALT | Crop | Residue F | ound (mg/l | (g) | | Depert/Trial |
|---|-----------------|-------------------------|----------------------------------|----------|-------|---------------------|--------------|----------------|-----------------------------|---|
| Location, Year (variety) | method | rate (g ai/ha) | stage at final application | | part | Kresoxim -methyl | 490M2 | 490M9 | Sum of parent + 490M9 | Report/Trial No., Reference Storage period |
| Ottesum 2008/09 (Limes) | | 2×125 (BAS 494 04 F) | BBCH 69 | 41 58 | Straw | 0.23 0.23 | 0.03 0.02 | 0.04 < 0.01 | 0.27 0.24 | Erdmann, 2010, Kresoxim_132 Max. frozen storage: 10 month |
| Belgium Brabant Kortenaken 2008/09 | Foliar spray | 2×125 (BAS 493 06 F) | BBCH 69 | 41 55 | Straw | 0.52 0.22 | 0.06 0.03 | 0.09 0.02 | 0.61 0.24 | Study: 2010/1006345 Trial: L090052 Erdmann, 2010, |
| (Molan) | | 2×125 (BAS 494 04 F) | BBCH 69 | 41 55 | Straw | 0.56 0.30 | 0.05 0.02 | 0.07 0.01 | 0.63 0.31 | Kresoxim_132 Max. frozen storage: 10 month |
| UK Essex Lawford 2008/09 | Foliar spray | 2×125 (BAS 493 06 F) | BBCH 69 | 42 | Straw | 0.68 | 0.08 | 0.08 | 0.76 | Study: 2010/1006345 Trial: L090053 Erdmann, 2010, |
| (Solistice) | | 2×125 (BAS 494 04 F) | BBCH 69 | 42 | Straw | 0.47 | 0.05 | 0.05 | 0.52 | Kresoxim_132 Max. frozen storage: 10 month |

Barley straw

Residues of kresoxim-methyl in barley straw from field trials performed during the 2007 and 2008 growing seasons in Southern France, Greece, Italy and Spain (Schroth & Martin, 2008, Kresoxim_128; Schroth, 2009, Kresoxim_129; Schroth & Martin, 2009, Kresoxim_133; Schroth, 2009, Kresoxim_134) were determined using BASF method L0010/01 (method 445/0) or BASF method L0095/01 (method 350/4), each with a limit of quantification of 0.01 mg/kg. For method L0010/01, the overall mean procedural recoveries for kresoxim-methyl in whole plant (forage) spiked at 0.01–10 mg/kg were 91% (n=3). For method 350/4, overall mean procedural recoveries for kresoxim-methyl (as 490M1) and metabolites 490M2 and 490M9 in barley straw spiked at 0.01–1 mg/kg were 82% (n=6), 71% (n=6) and 60% (n=6), respectively.

| Table 122 Residues of kresoxim-me | thyl in barley | straw following foliar | treatment (cGAP: 2 × 1 | 25 g ai/ha: 35 day PHI) |
|-----------------------------------|----------------|------------------------|------------------------|-------------------------|
| | | | | |

| Location, | Treatment | Application | Growth | DALT | Crop | Residue Four | nd (mg/kg) | | Report/Trial No., | |
|---|-----------------|-------------------------|----------------------------------|----------|-------|---------------------|--------------|--------------|-----------------------------|--|
| Year (variety) | method | rate (g ai/ha) | stage at final application | | part | Kresoxim- methyl | 490M2 | 490M9 | Sum of parent + 490M9 | Reference Storage period |
| Spain Sevilla Las Cabezas 2007 | Foliar spray | 2×125 (BAS 494 04 F) | BBCH 69 | 42 49 | Straw | 1.2 0.66 | 0.03 0.03 | 0.06 0.05 | 1.3 0.71 | Study: 2008/1043814/ 2009/1102142 Trial: L070265 |
| (Belen) | | 2×125 (BAS 493 05 F) | BBCH 69 | 42 49 | Straw | 0.89 0.39 | 0.04 0.02 | 0.06 0.03 | 0.95 0.42 | Schroth & Martin, 2008, Kresoxim_128; Schroth, 2009, Kresoxim_129 Max. frozen storage: 10 month |
| Spain Cadiz Puerto de | Foliar spray | 2×125 (BAS 494 04 F) | BBCH 69 | 42 49 | Straw | 0.57 0.33 | 0.03 0.03 | 0.05 0.03 | 0.62 0.36 | Study: 2008/1043814/ 2009/1102142 |

| | Treatment | Application | Growth | DALT | Crop | Residue Fou | und (mg/kg) |) | | |
|--|-----------------|-------------------------|----------------------------------|----------------|-------|----------------------|----------------------|------------------------|-----------------------------|---|
| Location, Year (variety) | method | rate (g ai/ha) | stage at final application | 27121 | part | Kresoxim- methyl | 490M2 | 490M9 | Sum of parent + 490M9 | Report/Trial No., Reference Storage period |
| Sta. Maria 2007 (Cecilia) | | 2×125 (BAS 493 05 F) | BBCH 69 | 42 49 | Straw | 0.36 | 0.03 | 0.07 | 0.43 0.49 | Trial: L070266 Schroth & Martin, 2008, Kresoxim_128; Schroth, 2009, Kresoxim_129 Max. frozen storage: 8 month |
| France Lot Goujounac 2007 | Foliar spray | 2×125 (BAS 494 04 F) | BBCH 69 | 35 42 49 | Straw | 0.21 0.27 0.42 | 0.05 0.05 0.06 | 0.02 0.02 0.03 | 0.23 0.29 0.45 | Study: 2008/1043814/ 2009/1102142 Trial: L070267 |
| (Diadem) | | 2×125 (BAS 493 05 F) | BBCH 69 | 35 42 49 | Straw | 0.43 0.62 0.67 | 0.07 0.06 0.03 | 0.03 0.02 0.01 | 0.46 0.64 0.68 | Schroth & Martin, 2008, Kresoxim_128; Schroth, 2009, Kresoxim_129 Max. frozen storage: 9 month |
| Italy Varese Cardano al Campo | Foliar spray | 2×125 (BAS 494 04 F) | BBCH 69- 71 | 35 42 49 | Straw | 0.09 0.16 0.12 | 0.02 0.02 0.01 | 0.01 0.01 < 0.01 | 0.10 0.17 0.13 | Study: 2008/1043814/ 2009/1102142 Trial: L070268 |
| 2007 (Aliseo) | | 2×125 (BAS 493 05 F) | BBCH 69- 71 | 35 42 49 | Straw | 0.09 0.09 0.04 | 0.01 0.02 0.02 | 0.01 0.01 < 0.01 | 0.10 0.10 0.05 | Schroth & Martin, 2008, Kresoxim_128; Schroth, 2009, Kresoxim_129 Max. frozen storage: 10 month |
| Spain Sevilla Utrera 2008 | Foliar spray | 2×125 | BBCH 69 | 42 49 | Straw | 0.15 0.10 | - | - | - | Study: 2008/1090698/ 2009/1102149 Trial: L080138 |
| (Belen) | | 2×125 | BBCH 69 | 42 49 | Straw | 0.41 0.33 | 0.15 0.10 | 0.11 0.06 | 0.52 0.39 | Schroth & Martin, 2009, Kresoxim_133; Schroth, 2009, Kresoxim_134 Max. frozen storage: 6 month |
| France Vienne Morton 2008 | Foliar spray | 2×125 | BBCH 69 | 42 49 | Straw | 0.44 0.43 | - | - | - | Study: 2008/1090698/ 2009/1102149 Trial: L080139 |
| (Prestige) | | 2×125 | BBCH 69 | 42 49 | Straw | 0.87 0.87 | 0.03 | 0.02 | 0.89 | Schroth & Martin, 2009, Kresoxim_133; Schroth, 2009, Kresoxim_134 Max. frozen storage: 4 month |
| Italy Milano Lazzate | Foliar spray | 2×125 | BBCH 71 | 34 42 49 | Straw | 0.04 0.01 0.01 | - | - | - | Study: 2008/1090698/ 2009/1102149 |

| | Treatment | Application | Growth | DALT | Crop | Residue Fou | nd (ma/ka) |) | | |
|---|-----------------|-------------------|----------------------------------|----------------|-------|----------------------|----------------------|-------------------------|-----------------------------|---|
| Location, Year (variety) | method | rate (g ai/ha) | stage at final application | | part | Kresoxim- methyl | 490M2 | 490M9 | Sum of parent + 490M9 | Report/Trial No., Reference Storage period |
| 2008 (Amillis) | | 2×125 | BBCH 71 | 34 42 49 | Straw | 0.17 0.06 0.05 | 0.04 0.03 0.04 | 0.05 0.02 0.01 | 0.22 0.08 0.06 | Trial: L080140 Schroth & Martin, 2009, Kresoxim_133; Schroth, 2009, Kresoxim_134 Max. frozen storage: 5 month |
| Spain Cadiz Puerto de Sta. Maria | Foliar spray | 2×125 | BBCH 69 | 42 48 | Straw | 0.22 0.17 | - | - | - | Study: 2008/1090698/ 2009/1102149 Trial: L080141 |
| 2008 (Cecilia) | | 2×125 | BBCH 69 | 42 48 | Straw | 0.43 0.29 | 0.05 | 0.05 0.04 | 0.48 0.33 | Schroth & Martin, 2009, Kresoxim_133; Schroth, 2009, Kresoxim_134 Max. frozen storage: 6 month |
| Italy Varese Cardano al Campo | Foliar spray | 2×125 | BBCH 69 | 35 42 49 | Straw | 0.13 0.04 0.01 | - | - | - | Study: 2008/1090698/ 2009/1102149 Trial: L080142 |
| 2008 (Scirocco) | | 2×125 | BBCH 69 | 35 42 49 | Straw | 0.32 0.26 0.12 | 0.03 0.02 0.01 | <0.01 <0.01 <0.01 | 0.33 0.27 0.13 | Schroth & Martin, 2009, Kresoxim_133; Schroth, 2009, Kresoxim_134 Max. frozen storage: 5 month |
| Greece Thessaloniki Lakkia 2008 | Foliar spray | 2×125 | BBCH 69 | 35 41 48 | Straw | 1.1 0.56 0.43 | - | - | - | Study: 2008/1090698/ 2009/1102149 Trial: L080143 |
| (Aliseo) | | 2×125 | BBCH 69 | 35 41 48 | Straw | 2.1 1.4 1.0 | 0.14 0.06 0.05 | 0.20 0.07 0.06 | 2.3 1.5 1.1 | Schroth & Martin, 2009, Kresoxim_133; Schroth, 2009, Kresoxim_134 Max. frozen storage: 5 month |

FATE OF RESIDUES IN STORAGE AND PROCESSING

Nature of residue during processing

Radiolabelled [phenyl-¹⁴C]-kresoxim-methyl was incubated in aqueous buffer solutions at concentrations of about 0.1 mg/L under three sets of conditions, each designed to simulate an appropriate process: 90 °C (pH 4, 20 minutes) to simulate pasteurisation, 100 °C (pH 5, 60 minutes), to simulate boiling, baking and brewing, and 120 °C (pH 6, 20 minutes) to simulate sterilisation (Hassink, 2008, Kresoxim_138).

Total recovered radioactivity was measured for each test solution by LSC. Radioactive components were characterised by fractionation and co-chromatography with authenticated reference compounds using HPLC-UV.

| Compound | % applied radioactivity recov | vered as | |
|-----------------|-------------------------------|----------|-------|
| | Kresoxim-methyl | 490M1 | 490M2 |
| pH 4 90°C 20 mi | ns | | |
| Before test | 100 | n.d. | n.d. |
| After test | 99.2 | n.d. | n.d. |
| pH 5 100°C 60 m | nins | | |
| Before test | 96.8 | n.d. | 3.2 |
| After test | 89.6 | 8.0 | 2.4 |
| pH 6 120°C 20 m | nins | | |
| Before test | 100.0 | n.d. | n.d. |
| After test | 23.8 | 70.8 | 4.6 |

Table 123 Hydrolysis of kresoxim-methyl under simulated processing conditions

Residues after processing

The fate of kresoxim-methyl during processing of raw agricultural commodity (RAC) was investigated in apples, grapes, gherkin and olive for oil production. As a measure of the transfer of residues into processed products, a processing factor was used, which is defined as:

Processing factor = Residue in processed product (mg/kg) ÷ Residue in raw agricultural commodity (mg/kg)

If residues in the RAC were below the LOQ, no processing factor could be derived. In case of residues below the LOQ in the processed product, the numeric value of the LOQ was used for the calculation and the PF was expressed as "less than" (e.g. <0.5).

Apple

The transfer of residues of kresoxim-methyl into processed commodities was investigated in apples from four supervised field trial conducted in Germany (Plier, 2011, Kresoxim_139). The trials were performed with exaggerated rates at 2×500 g ai/ha, followed by 2×625 g ai/ha with an interval of 7±1 days and harvest at 35 DALT. Apples were processed into apple sauce, canned apples, wet and dry pomace, juice, dried apples and fruit syrup using common commercial practices. All samples were analysed according to BASF method L0095/01 (identical to BASF method 350/4). Overall mean procedural recoveries for kresoxim-methyl and metabolites 490M2 and 490M9 in apple matrices spiked at 0.01–10 mg/kg were 89% (n=30), 87% (n=30) and 92% (n=30), respectively.

| Table 124 Summary of kresoxim-methyl residues in apples and processed commodities | |
|---|--|
|---|--|

| Trial Identification (City, State/Region, Country, Year) | Form. | Crop/ Variety | Commodity or Matrix | Total Rate (g ai/ha) | PHI (days) | Total Residues (mg/kg) | Processing Factor |
|--|-------|------------------|------------------------|----------------------------|---------------|---------------------------|----------------------|
| | | | Apple (RAC) | | | 0.30 | - |
| | | | Washed apple | | | 0.20 | 0.67 |
| | | | Wash water | | | 0.08 | 0.27 |
| Trial 100070 | | | Apple sauce | | | 0.07 | 0.23 |
| Trial L100273 | WG | Apple/ | Canned apples | 2250 | 34 | 0.03 | 0.10 |
| (Pirna, Germany, 2010) | WG | Jonica | Wet pomace | 2250 | 34 | 0.41 | 1.4 |
| 2010) | | | Juice | | | 0.03 | 0.10 |
| | | | Dried apples | | | 0.07 | 0.23 |
| | | | Dried pomace | | | 1.3 | 4.5 |
| | | | Fruit syrup | | | 0.03 | 0.10 |
| | | | Apple (RAC) | | | 0.10 | 1.0 |
| | | | Washed apple | | | 0.19 | 1.9 |
| | | | Wash water | | | 0.07 | 0.70 |
| T-1-11 100074 | | | Apple sauce | | | 0.05 | 0.50 |
| Trial L100274 (Sornzig, Germany, | WG | Apple/ | Canned apples | 2250 | 35 | 0.03 | 0.30 |
| (3011/21g, Germany, 2010) | WG | Gala | Wet pomace | 2230 | 30 | 0.40 | 4.0 |
| 2010) | | | Juice | | | 0.03 | 0.30 |
| | | | Dried apples | | | 0.03 | 0.30 |
| | | | Dried pomace | | | 1.6 | 16 |
| | | | Fruit syrup | | | 0.03 | 0.30 |
| Trial L100275 | WG | Apple/ | Apple (RAC) | 2250 | 35 | 0.26 | 1.0 |
| (Leisnig, Germany, | WG | Jonagored | Washed apple | 2200 | 30 | 0.29 | 1.1 |

842

| Trial Identification (City, State/Region, Country, Year) | Form. | Crop/ Variety | Commodity or Matrix | Total Rate (g ai/ha) | PHI (days) | Total Residues (mg/kg) | Processing Factor |
|--|-------|------------------|------------------------|----------------------------|---------------|---------------------------|----------------------|
| 2010) | | | Wash water | | | 0.07 | 0.27 |
| | | | Apple sauce | | | 0.07 | 0.27 |
| | | | Canned apples | | | 0.03 | 0.12 |
| | | | Wet pomace | | | 0.57 | 2.2 |
| | | | Juice | | | 0.03 | 0.12 |
| | | | Dried apples | | | 0.11 | 0.42 |
| | | | Dried pomace | | | 2.3 | 8.7 |
| | | | Fruit syrup | | | 0.03 | 0.12 |
| | | | Apple (RAC) | | | 0.31 | 1.0 |
| | | | Washed apple | | | 0.33 | 1.1 |
| | | | Wash water | | | 0.10 | 0.32 |
| Trial L100276 | | | Apple sauce | | | 0.09 | 0.29 |
| (Werneuchten, | WG | Apple/ | Canned apples | 2250 | 35 | 0.04 | 0.13 |
| Germany, 2010) | WG | Jonagold | Wet pomace | 2230 | 33 | 0.85 | 2.7 |
| Germany, 2010) | | | Juice | | | 0.04 | 0.13 |
| | | | Dried apples | | | 0.19 | 0.61 |
| | | | Dried pomace | | | 2.8 | 9.1 |
| | | | Fruit syrup | | | 0.10 | 0.10 |

RAC: raw agricultural commodity

In a second study with apples from one field trial in the United States of America, the transfer of residues of kresoximmethyl into apple juice and wet pomace was investigated (Wofford, 1998, Kresoxim_140). The trial was performed at the intended use rate of 4×225 g ai/ha (1×) and exaggerated rates of 4×675 g ai/ha (3×) and 4×1120 g ai/ha (5×) with an interval of 7±1 days between applications. However, only apples from the untreated control and the 5× plots were harvested (30 DALT) and processed according to typical commercial practices. All samples were analysed according to BASF method 350/3. Overall mean procedural recoveries for kresoxim-methyl and metabolites 490M2 and 490M9 in apple matrices spiked at 0.05 and 1.0 mg/kg were 97% (n=6), 96% (n=6) and 94% (n=6), respectively.

| Trial Identification (City, State/Region, Country, Year) | Form | Crop/ Variety | Commodity or Matrix | Total Rate (g ai/ha) | PHI (days) | Kresoxim- methyl (mg/kg) | Processing Factor (MRL) | Total Residues (mg/kg)ª | Processing Factor (STMR) |
|--|------|------------------|------------------------|-------------------------------|---------------|--------------------------------|-------------------------------|-------------------------------|--------------------------------|
| Trial: RCN 97122 | | Apple/ Red | Apple (RAC) Juice | | | 0.82 | - 0.10 | 0.92 | 0.10 |
| (Ephrata, WA, USA, 1997) | WG | Delicio us | Wet pomace | 4480 | 30 | 2.3 | 2.8 | 2.4 | 2.6 |

Table 125 Summary of residues of kresoxim-methyl in apples and processed commodities

In a third study with apples from 3 field trials performed in Germany, the transfer of residues of kresoxim-methyl into apple juice, wet pomace and apple sauce was investigated (Fuchs, 1995, Kresoxim_141). The trials were performed at use rates of $8 \times 200 \text{ g}$ ai/ha with an interval of 10 ± 1 days between applications and harvest at 14 DALT. Apples were processed using common commercial practices. All samples were analysed according to BASF method 351/2. Overall mean procedural recoveries for kresoxim-methyl in apple matrices spiked at 0.05–0.5 mg/kg were 101% (n=7).

Table 126 Summary of kresoxim-methyl residues in apples and processed commodities

| Trial Identification (City, State/Region, Country, Year) | Form. | Crop/ Variety | Commodity or Matrix | Total Rate (g ai/ha) | PHI (days) | Total Residues (mg/kg) | Processing Factor |
|--|-------|--------------------|------------------------|----------------------------|---------------|---------------------------|----------------------|
| | | | Apple (RAC) | | | 0.16 | - |
| Trial DU1/12/93 | | Apple/ | Washed apple | | | 0.19 | 1.2 |
| (Gundheim, Germany, | WG | Roter | Juice | 1600 | 14 | <0.05 | <0.31 |
| 1993) | | Boskop | Wet pomace | | | <0.05 | <0.31 |
| | | | Apple sauce | | | <0.05 | <0.31 |
| Trial DU2/52/93 | | Apple/ | Apple (RAC) | | | 0.19 | - |
| (Stetten, Germany, | WG | Apple/ Jonagold | Washed apple | 1600 | 14 | 0.07 | 0.37 |
| 1993) | | Juliayulu | Juice | | | <0.05 | <0.26 |

| Trial Identification (City, State/Region, Country, Year) | Form. | Crop/ Variety | Commodity or Matrix | Total Rate (g ai/ha) | PHI (days) | Total Residues (mg/kg) | Processing Factor |
|--|-------|--------------------|------------------------|----------------------------|---------------|---------------------------|----------------------|
| | | | Wet pomace | | | 0.09 | 0.47 |
| | | | Apple sauce | | | <0.05 | <0.26 |
| | | | Apple (RAC) | | | 0.08 | - |
| Trial DU3/36/93 | | Apple/ | Washed apple | | | 0.16 | 2.0 |
| (Rödersheim-Gronau, | WG | Apple/ Jonagold | Juice | 1600 | 14 | <0.05 | <0.63 |
| Germany, 1993) | | Juliayulu | Wet pomace | | | 0.17 | 2.1 |
| | | | Apple sauce | | | <0.05 | <0.63 |

In a fourth study with apples from two field trials performed in the United States of America (Washington and Ohio), the transfer of residues of kresoxim-methyl into apple juice and wet pomace was investigated (Wofford, 1996, Kresoxim_142. The trials were performed at the intended use rate of 4×225 g ai/ha (1×) or with either of the exaggerated rates of 670 g ai/ha (3×) or 1120 g ai/ha (5×), respectively. However, only apples from the untreated control and the 5× plots were harvested (90 DALT) and processed according to typical commercial practices. All samples were analysed according to BASF method 350/3. Overall mean procedural recoveries for kresoxim-methyl and metabolites 490M2 and 490M9 in apple matrices spiked at 0.05 and 1.0 mg/kg were 97% (n=6), 91% (n=6) and 86% (n=6), respectively. It should be noted that the report did not contain data from the Washington site.

Table 127 Summary of residues of kresoxim-methyl and metabolites 490M2 and 490M9 in apples and processed commodities

| Trial Identification (City, State/Region, Country, Year) | From. | Crop/ Variety | Commodity or Matrix | Total Rate (g ai/ha) | PHI (days) | Average Residues (mg/kg) | Processing Factor |
|--|-------|------------------|------------------------|----------------------------|---------------|-----------------------------|----------------------|
| Trial: RCN 94206 | | Apple/ | Apple (RAC) | | | < 0.05 | - |
| (Ohio, USA, 1996) | WG | not | Juice | 4480 | 90 | < 0.05 | - |
| (0110, 03A, 1990) | | stated | Wet pomace | | | < 0.05 | - |

Grape

The transfer of residues of kresoxim-methyl into processed commodities was investigated in grapes from two supervised field trial conducted in France (Benz & Mackenroth, 2000, Kresoxim_143; Perret et al., 2000, Kresoxim_144). The trials were performed with treatment rates at 3×50 g ai/ha with an interval of 14–15 days between applications and harvest at 42 DALT. Apples were processed into must, wet pomace and wine using common commercial practices. All samples were analysed according to BASF method 350/3, but no metabolites were determined. Overall mean procedural recoveries for kresoxim-methyl in grape matrices spiked at 0.05 and 5 mg/kg were 96% (n=12).

| Table 128 Summary | v of kresoxim-meth | vl residues in grapes a | and processed commodities |
|-------------------|--------------------|-------------------------|---------------------------|
| | | | |

| Trial Identification (City, State/Region, Country, Year) | Form. | Crop/ Variety | Commodity or Matrix | Total Rate (g ai/ha) | PHI (days) | Total Residues ^a (mg/kg) (Individual Values) | Processing Factor |
|--|-------|------------------|------------------------|----------------------------|---------------|--|----------------------|
| Trial DAC (002 | | | Grape (RAC) | | | <0.05 | - |
| Trial BAS/003 (Beaumes de Venise, | SE | Grape/ | Must | 150 | 42 | <0.05 | - |
| (Beauriles de Verlise, France, 1999) | JE | Grenache | Wet pomace | 150 | 42 | 0.052 | - |
| Trance, 1999) | | | Wine | | | <0.05 (<0.05, <0.05) | - |
| | | | Grape (RAC) | | | <0.05 | - |
| Trial BAS/004 (Pagumos de Venise | SE. | Grape/ | Must | 150 | 42 | <0.05 | - |
| (Beaumes de Venise, SE France, 1999) | | Muscat | Wet pomace | 150 | 42 | <0.05 | - |
| 11d1100, 1799) | | | Wine | | | <0.05 (<0.05, <0.05) | - |

^a Sum of kresoxim-methyl (as 490M1) and metabolites 490M2 and 490M9 expressed as parent kresoxim-methyl

In a second study, the transfer of residues of kresoxim-methyl into processed commodities was investigated in grapes from four supervised field trial conducted in Germany (Fuchs et al, 1996, Kresoxim_145). The trials were performed with treatment rates at 6×150 g ai/ha at intervals of 9–15 days and harvest at 34/35 DALT. Grape samples were processed to juice/must, wine and wet pomace using common commercial practices. All samples were analysed according to BASF method 350/3. Overall mean procedural recoveries for kresoxim-methyl and metabolites 490M2 and 490M9 in grape matrices spiked at 0.05 and 5 mg/kg ranged between 77–103% (n=48).

| Trial Identification (City, State/Region, Country, Year) | Form. | Crop/ Variety | Commodity or Matrix | Total Rate (g ai/ha) | PHI (days) | Sum of parent + 490M9 | Processing Factor | |
|--|-------|------------------|--------------------------------|----------------------------|---------------|-----------------------|----------------------|--|
| | | | Grape (RAC) | | | 0.32 | - | |
| | | | Must, cold | | | 0.10 | 0.31 | |
| T.: 1 DU0 /02 /05 | | One of the | Must , heated | | | <0.10 | <0.31 | |
| Trial DU2/03/95 (Wiesloch, Germany, | SE | Grape/ not | Wet pomace | 900 | 34/35 | 0.76 | Factor - 0.31 | |
| (wiesioch, Germany, 1996) | SE | stated | Wine (rosé) from | 900 | 34/35 | <0.10 | 0.01 | |
| 1990) | | Stateu | must cold | | | <0.10 | <0.31 | |
| | | | Wine (red) from | | | 0.10 | 0.01 | |
| | | | must heated | | | <0.10 | <0.31 | |
| | | | Grape (RAC) | | | 0.81 | - | |
| | | | Must, cold | | | 0.24 | 0.30 | |
| | | Cronol | Must , heated | | 34/35 | 0.12 | 0.15 | |
| Trial DU2/04/95 | SE | Grape/ not | Wet pomace | 900 | | 1.6 | 2.0 | |
| (Wiesloch, Germany, 1999) | y, JE | stated | Wine (rosé) from | | | <0.10 | 0.10 | |
| 1999) | | Stateu | must cold | | | <0.10 | <0.12 | |
| | | | Wine (red) from | from 0.10 | | 0.10 | 0.12 | |
| | | | must heated | | | 0.10 | 0.12 | |
| | | | Grape (RAC) | | | 0.49 | - | |
| | | | Must, cold | | | 0.13 | 0.27 | |
| Trial DU3/01/95 | | Grape/ | Must , heated | | | 0.12 | 0.25 | |
| (Grünstadt- | SE | not | Wet pomace | 900 | 34/35 | 0.46 | 0.94 | |
| Sausenheim, Germany, 1999) | JL | stated | Wine (rosé) from must cold | 700 | 34/33 | <0.10 | <0.20 | |
| | | | Wine (red) from | | | 0.40 | 0.00 | |
| | | | must heated | | | <0.10 | <0.20 | |
| | | | Grape (RAC) | | | 0.22 | - | |
| | | | Must, cold | | | 0.14 | 0.64 | |
| Trial DU3/02/95 | | Grand | Must , heated | 1 | | <0.10 | 0.46 | |
| | SE | Grape/ | Wet pomace | 900 | 34/35 | 0.57 | 2.6 | |
| (Grünstadt- Sausenheim, 1999) | SE | not stated | Wine (rosé) from | 900 | 34/35 | <0.10 | <0.46 | |
| | | Juicu | must cold | | | 10.10 | \0.40 | |
| | | | Wine (red) from must heated | | | <0.10 | <0.46 | |

Table 129 Summary of kresoxim-methyl residues in grapes and processed commodities

In a third study, the transfer of residues of kresoxim-methyl into processed commodities was investigated in grapes from one supervised field trial conducted in the United States (Movassaghi *et al*, 1996, Kresoxim_146). The trials were performed at the intended use rate of (1×) and with exaggerated rates (3× or 5×). Whole grape samples were collected from the treated plots at 75, 90, and 105 DALT and processed to grape juice and raisins according to typical commercial practices. However, only grapes from the 1× and 5× plots at 75 day PHI were analysed, but since in the RAC of the 1× trial residues were <LOQ no processing factor could be derived. All samples were analysed according to BASF method 350/3. The overall average recovery was 100% (n=24).

Table 130 Summary of kresoxim-methyl residues in grapes and processed commodities

| Trial Identification (City, State/Region, Country, Year) | Form. | Crop/ Variety | Commodity or Matrix | Total Rate (g ai/ha) | PHI (days) | Kresoxim- methyl (mg/kg) | Processing Factor (MRL) | Sum of parent + 490M9 | Processing Factor (STMR) |
|--|-------|------------------|------------------------|-------------------------------|---------------|--------------------------------|----------------------------|-----------------------------|--------------------------------|
| Trial 95008 | | Grape/ | Grape (RAC) | Net | | 0.18 | - | 0.40 | - |
| (California, | SE | not | Juice | Not stated | 75 | 0.07 | 0.39 | 0.27 | 0.68 |
| USA, 1996) | | stated | Raisins | Stateu | | 0.40 | 2.2 | 0.72 | 1.8 |

In a forth study, the transfer of residues of kresoxim-methyl into processed commodities was investigated in grapes from two supervised field trial conducted in the United States of America (Thornton et al., 1997, Kresoxim_147). The trials were performed at the intended use rate of (1×) or at an exaggerated rates (3×). Whole grape samples were collected from the treated plots at 90 DALT from the 1× rate and at 1 and 14 DALT for the 3× rate. Whole grapes were processed into grape juice and raisins

for analysis. However, only grapes from the 3× plot at 14 day PHI were analysed. All samples were analysed according to BASF method 350/3. The overall average recovery was 89% (n=30).

| Trial Identification (City, State/Region, Country, Year) | Form. | Crop/ Variety | Commodity or Matrix | Total Rate (g ai/ha) | PHI (days) | Kresoxim- methyl (mg/kg) | Processing Factor (MRL) | Sum of parent + 490M9 | Processing Factor (STMR) |
|--|-------|------------------|------------------------|-------------------------------|-------------------|--------------------------------|----------------------------|-----------------------------|--------------------------------|
| Trial 94013 | | Grape/ | Grape (RAC) | Not | | 0.16 | - | 0.21 | - |
| (California, USA, | SE | not | Juice | stated | 14 | <0.05 | 0.31 | <0.10 | <0.48 |
| 1997) | | stated | Raisins | Sidieu | | 0.26 | 1.6 | 0.32 | 1.5 |
| Trial 94014 | | Grape/ | Grape (RAC) | Not | | 1.8 | - | 1.9 | - |
| (New York, USA, | SE | not | Juice | Not stated | 14 | 0.13 | 0.72 | 0.18 | 0.10 |
| 1996) | | stated | Raisins | Stateu | | 2.8 | 1.6 | 3.0 | 1.6 |

Table 131 Summary of kresoxim-methyl residues in grapes and processed commodities

Gherkin

The transfer of residues of kresoxim-methyl into processed commodities was investigated in gherkin from two supervised field trial conducted in France (Scharm, 2001, Kresoxim_112). The trials were performed with treatment rates at 4×150 g ai/ha with an interval of 6-7 days between applications and harvest at 0 and 3 days after the last application. Gherkins were processed using common commercial practices. Residues of kresoxim-methyl (as 490M1) were determined using method BASF method 445/0 with a limit of quantification of 0.05 mg/kg. Overall mean procedural recoveries for kresoxim-methyl (as 490M1) in gherkin spiked at 0.05 and 0.5 mg/kg was 88% (n=10).

| Trial Identification (City, State/Region, Country, Year) | Form. | Crop/ Variety | Commodity or Matrix | Total Rate (g ai/ha) | PHI (days) | Total Residues (mg/kg) | Processing Factor |
|--|-------|---------------------|------------------------|----------------------------|---------------|------------------------|----------------------|
| | | | Gherkins (RAC) | | | 0.16 | - |
| Trial AT-00/024-1 | | Gherkins/ | Washed gherkins | | | <0.05 | <0.31 |
| (Mühlhausen, | SC | Melody | Wash water | 600 | 0 | <0.05 | <0.31 |
| Germany, 2000) | | weibuy | Canned gherkins | | | <0.05 | <0.31 |
| | | | Brine | | | <0.05 | <0.31 |
| Trial AT-00/024-2 | | | Gherkins (RAC) | | | <0.05 | - |
| (Riedstadt- | | Gherkins/ | Washed gherkins | 600 | 0 | <0.05 | - |
| Crumstadt, Germany, | SC | Pontomac | Wash water | | | <0.05 | - |
| 2000) | | F1 | Canned gherkins | | | <0.05 | - |
| 2000) | | | Brine | | | <0.05 | - |
| | | | Gherkins (RAC) | | | 0.059 | - |
| Trial AT-00/024-3 | | Gherkins/ | Washed gherkins | | | <0.05 | <0.85 |
| (Gernsheim, | SC | Musica | Wash water | 600 | 0 | <0.05 | <0.85 |
| Germany, 2000) | | IVIUSICA | Canned gherkins | | | <0.05 | <0.85 |
| | | | Brine | | | <0.05 | <0.85 |
| | | | Gherkins (RAC) | | | <0.05 | - |
| Trial AT-00/024-4 (Eich, Germany, SC | | Gherkins/ | Washed gherkins |] | | <0.05 | - |
| | SC | Gnerkins/ Mauvin | Wash water | 600 | 0 | <0.05 | - |
| 2000) | | IVIAUVIII | Canned gherkins |] | | <0.05 | - |
| | | | Brine | | | <0.05 | - |

Table 132 Summary of kresoxim-methyl residues in gherkins and processed commodities

Olive for oil production

The transfer of residues of kresoxim-methyl into processed commodities was investigated in olives from four supervised field trial conducted in Spain (Meumann & Rabe, 1999, Kresoxim_136). Olives received 1 spray applications of kresoxim-methyl at a nominal rate of 100 g ai/ha. Samples of olives were collected at 30±1 days after the application and processed to crude oil, waste water and pomace using common commercial practices. Residues of kresoxim-methyl (as 490M1) and metabolites 490M2 and 490M9 were determined using method BASF method 350/3 with a limit of quantification of 0.05 mg/kg. Overall mean procedural recoveries for kresoxim-methyl (as 490M1) and metabolites 490M2 and 490M9 in olive matrices spiked at 0.05–5 mg/kg ranged between 87–124% (n=48).

846

| Trial Identification (City, State/Region, Country, Year) | Form. | Crop/ Variety | Commodity or Matrix | Total Rate (g ai/ha) | PHI (days) | Kresoxim- methyl (mg/kg) | Processing Factor (MRL) | Sum of parent + 490M9 | Processing Factor (STMR) |
|--|------------------------|-------------------|------------------------|----------------------------|---------------|--------------------------------|----------------------------|-----------------------------|--------------------------------|
| Trial AC/22/98 | | | Olive (RAC) | | | <0.05 | - | <0.10 | - |
| (Andalusia, | WG | Olives/ | Waste water | 100 | 29 | <0.05 | - | <0.10 | - |
| Spain, 1998) | 110 | Picual | Pomace | 100 | 27 | <0.05 | - | <0.10 | - |
| Spain, 1770) | | | Crude oil | | | 0.17 | - | 0.22 | - |
| Trial AC (22/00 | | | Olive (RAC) | | | <0.05 | - | <0.10 | - |
| | AC/23/98 alusia. WG | Olives/ Picual | Waste water | 100 | 29 | <0.05 | - | <0.10 | - |
| (Andalusia, | WG | | Pomace | | | <0.05 | - | <0.10 | - |
| Spain, 1998) | | | Crude oil | | | 0.12 | - | 0.17 | - |
| T | | | Olive (RAC) | | | <0.05 | - | <0.10 | - |
| Trial AC/24/98 | 14/0 | Olives/ | Waste water | 100 | 20 | <0.05 | - | <0.10 | - |
| (Andalusia, | WG | Martena | Pomace | 100 | 30 | 0.05 | - | 0.10 | - |
| Spain, 1998) | | | Crude oil | 1 | | 0.13 | - | 0.18 | - |
| | | | Olive (RAC) | | | 0.11 | - | 0.16 | - |
| Trial AC/25/98 | | Olives/ | Waste water | 100 | | <0.05 | 0.5 | <0.10 | <0.63 |
| | Hoj. | Pomace | 100 | 30 | 0.12 | 1.1 | 0.17 | 1.1 | |
| Spain, 1998) | | blanca | Crude oil | | | 0.49 | 4.5 | 0.54 | 3.4 |

Table 133 Summary of kresoxim-methyl residues in olives and processed commodities

Table 134 Overview of processing factors for MRL and STMR-P calcualtion

| Raw commodity | Processed commodity | Individual processing factors | Median or best estimate processing factor |
|--------------------|-----------------------------|--|---|
| Processing factors | for MRL calcuation | | |
| Grapes | Raisins | 1.6, 1.6, 2.2 | 1.6 |
| Olives | Virgin oil | 4.5 | 4.5 |
| Processing factors | for STMR-P calculation | | |
| | Washed apple | 0.37, 0.67, 1.1, 1.1, 1.2, 1.9, 2.0 | 1.1 |
| | Wash water | 0.27, 0.27, 0.32, 0.70, | 0.30 |
| | Apple sauce | 0.23, 0.26, 0.27, 0.29, 0.31, 0.50, 0.63 | 0.29 |
| | Canned apples | 0.10, 0.12, 0.13, 0.30 | 0.13 |
| Apple | Wet pomace | 0.31, 0.47, 1.4, 2.1, 2.2, 2.6, 2.7, 4.0 | 2.2 |
| | Juice | 0.10, 0.10, 0.12, 0.13, 0.26, 0.30, 0.31, 0.63 | 0.20 |
| | Dried apples | 0.23, 0.30, 0.42, 0.61 | 0.39 |
| | Dried pomace | 4.5, 8.7, 9.1, 16 | 8.9 |
| | Fruit syrup | 0.10, 0.10, 0.12, 0.30 | 0.11 |
| Grapes | Must, cold | 0.27, 0.30, 0.31, 0.64 | 0.31 |
| | Must, heated | 0,15, 0.25, 0.31, 0.46 | 0.28 |
| | Wet pomace | 0.94, 2.0, 2.4, 2.6 | 2.2 |
| | Wine (rosé) from must cold | 0.12, 0.20, 0.31, 0.46 | 0.26 |
| | Wine (red) from must heated | 0.12, 0.20, 0.31, 0.46 | 0.26 |
| | Juice | 0.10, 0.48, 0.68 | 0.48 |
| | Raisins | 1.5, 1.6, 1.8 | 1.5 |
| Gherkins | Washed gherkins | 0.31, 0.85 | 0.58 |
| | Wash water | 0.31, 0.85 | 0.58 |
| | Canned gherkins | 0.31, 0.85 | 0.58 |
| | Brine | 0.31, 0.85 | 0.58 |
| | Waste water | 0.63 | 0.63 |
| Olives | Pomace | 1.1 | 1.1 |
| | Crude oil | 3.4 | 3.4 |

RESIDUES IN ANIMAL COMMODITIES

Farm animal feeding studies

Lactating cows

The transfer of residues of kresoxim-methyl into animal matrices was investigated in a study with dairy cows (Redgrave, 1994, Kresoxim_148; Redgrave, 1994, Kresoxim_149). The study was conducted at treatment rates of 7 (1×), 21 (3×), and 70 (10×) ppm (0.23, 0.65 and 2.2 mg/kg bw) for 28–29 days.

The cows in the treatment groups (three animals per group, plus two animals in the depuration group) were treated with kresoxim-methyl, mixed with feed concentrate, twice daily. Milk samples were collected twice daily throughout the dosing period. On days 1, 14 and 28 milk samples were retained to prepare skim milk and cream samples. All cows were sacrificed on the day after the last dose, except for the depuration group and the control cow included in the depuration phase. The depuration group cows were sacrificed on day 31 and day 36. Samples of liver, kidney, muscle, and fat were collected and taken for analysis.

Milk, skim milk and cream were analysed for metabolites 490-M2 and 490-M9 using method no. 354/1, while tissues were analysed for metabolites 490-M1, 490-M2 and 490-M9 using method no. 354/2 with a limit of quantification of 0.002 mg/kg and 0.01 mg/kg, respectively. Maximum storage time of milk samples was 86 days, while for tissues the maximum frozen storage period was 175 days.

In milk, skim milk and cream residues of metabolites 490-M2 and 490-M9 were <LOQ (0.002 mg/kg) in all dosing groups throughout the study. The findings in tissues are summarised in Table 135.

| | Mean residues found in mg/kg (Individual values) | | | |
|---------------------------------|--|--------------|------------------------------|--|
| Dosing group | 490M1 | 490M2 | 490M9 | |
| Liver | | | | |
| 1× (7 ppm) | <0.01 | not measured | <0.01 | |
| 3× (21 ppm) | 0.027 (0.037, 0.016, 0.028) | not measured | 0.016 (0.020, <0.010, 0.020) | |
| 10× (70 ppm) | 0.032 (0.038, 0.019, 0.040) | not measured | 0.015 (0.014, <0.010, 0.021) | |
| 10× (70 ppm), 2 days depuration | 0.014 | not measured | <0.01 | |
| 10× (70 ppm), 7 days depuration | <0.01 | not measured | <0.01 | |
| Kidney | | | | |
| 1× (7 ppm) | 0.030 (0.034, 0.026, 0.029) | <0.01 | <0.01 | |
| 3× (21 ppm) | 0.10 (0.084, 0.065, 0.16) | <0.01 | 0.014 (0.012, 0.012, 0.019) | |
| 10× (70 ppm) | 0.20 (0.29, 0.14, 0.12, 0.050, | <0.01 | 0.022 (0.028, 0.013, 0.013, | |
| | 0.39, 0.23) | | <0.010, 0.047, 0.020) | |
| 10× (70 ppm), 2 days depuration | 0.041 (0.037, 0.044) | <0.01 | <0.01 | |
| 10× (70 ppm), 7 days depuration | 0.028 (<0.01, 0.045) | <0.01 | <0.01 | |
| Muscle | | | | |
| 1× (7 ppm) | <0.01 | <0.01 | not measured | |
| 3× (21 ppm) | <0.01 | <0.01 | not measured | |
| 10× (70 ppm) | <0.01 | <0.01 | not measured | |
| 10× (70 ppm), 2 days depuration | <0.01 | <0.01 | not measured | |
| 10× (70 ppm), 7 days depuration | <0.01 | <0.01 | not measured | |
| Subcutaneous fat | | | | |
| 1× (7 ppm) | <0.01 | <0.01 | <0.01 | |
| 3× (21 ppm) | <0.01 | <0.01 | <0.01 | |
| 10× (70 ppm) | 0.024 (0.030, 0.015, 0.028) | <0.01 | <0.01 | |
| 10× (70 ppm), 2 days depuration | <0.01 | <0.01 | <0.01 | |
| 10× (70 ppm), 7 days depuration | <0.01 | <0.01 | <0.01 | |
| Peritoneal fat | | | | |
| 1× (7 ppm) | <0.01 | <0.01 | <0.01 | |
| 3× (21 ppm) | 0.030 (0.041, 0.010, 0.039) | <0.01 | <0.01 | |
| 10× (70 ppm) | 0.089 (0.13, 0.032, 0.10) | <0.01 | <0.01 | |
| 10× (70 ppm), 2 days depuration | <0.01 | <0.01 | <0.01 | |
| 10× (70 ppm), 7 days depuration | <0.01 | <0.01 | <0.01 | |

Table 135 Residues of kresoxim-methyl metabolites in cow tissues

Since in the first study some unexplainable low procedural recoveries occurred, a re-analysis of the respective sample sets for milk and subcutaneous and peritoneal fat was performed (Maxwell, 1996, Kresoxim_150). For milk and tissue matrices, methods no. 354/1 and no. 354/2 were employed, respectively. Procedural recoveries ranged between 88–111%. The

concentrations of the metabolites BF 490-2 and BF 490-9 were below the limit of quantification of 0.002 mg/kg in all milk samples re-analysed. The findings in tissues are summarised in Table 136.

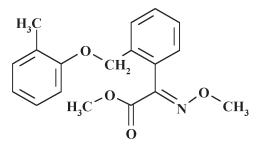
Table 136 Re-analysis of residues of kresoxim-methyl metabolites in cow fat

| Desing group | Mean residues found in mg/kg (Individual values) | | | |
|---------------------------------|--|-----------------|-----------------|--|
| Dosing group | 490-M1 | 490M2 | 490M9 | |
| Subcutaneous fat | | | | |
| 1× (7 ppm) | <0.01 | <0.01 | <0.01 | |
| 3× (21 ppm) | <0.01 | <0.01 | <0.01 | |
| 10× (70 ppm) | not re-analysed | not re-analysed | not re-analysed | |
| 10× (70 ppm), 2 days depuration | not re-analysed | not re-analysed | not re-analysed | |
| 10× (70 ppm), 7 days depuration | not re-analysed | not re-analysed | not re-analysed | |
| Peritoneal fat | | | | |
| 1× (7 ppm) | <0.01 | <0.01 | <0.01 | |
| 3× (21 ppm) | 0.025 (0.030, <0.010, 0.034) | <0.01 | <0.01 | |
| 10× (70 ppm) | 0.055 (0.095, <0.010, 0.060) | <0.01 | <0.01 | |
| 10× (70 ppm), 2 days depuration | <0.01 | <0.01 | <0.01 | |
| 10× (70 ppm), 7 days depuration | <0.01 | <0.01 | <0.01 | |

APPRAISAL

Kresoxim-methyl is a strobilurin fungicide for the control of scab and other fungal diseases on a wide range of crops. It acts by inhibiting the mitochondrial respiration. It was first evaluated by JMPR in 1998 (T, R), when an acceptable daily intake (ADI) of 0– 0.4 mg/kg bw was established. An acute reference dose (ARfD) was not considered necessary. In 2001 the JMPR evaluated the compound for residues and recommended a number of maximum residue levels. Kresoxim-methyl was scheduled by the Forty-ninth Session of the CCPR for the periodic evaluation of residue and toxicology by the 2018 JMPR.

The Meeting received information on identity, physicochemical properties, metabolism (plant, confined rotational crops and animals), environmental fate, methods of residue analysis, freezer storage stability, registered use patterns, supervised residue trials in citrus, apple, peach, black currant, strawberry, grape, mango, leek, onion, garlic, cucumber, squash, gherkin, melon, sweet pepper, tomato, vine leaves, sugar beet, wheat, barley, pecan nuts and olives, fate of residues in processing, and livestock feeding studies.



The IUPAC and CA name of kresoxim-methyl is methyl (2E)-[2-(hydroxymethyl)phenyl](methoxyimino)acetate.

Metabolism and environmental fate studies were conducted using either [phenoxy-¹⁴C]- or [phenyl-¹⁴C]-kresoxim-methyl. Moreover, [¹³C]-kresoxim-methyl was used.

| | The following | abbreviations | are used for | the metabolites | discussed below: |
|--|---------------|---------------|--------------|-----------------|------------------|
|--|---------------|---------------|--------------|-----------------|------------------|

| Code Names | Chemical Names (IUPAC) | Structure | |
|-------------------|---|--------------|-------------|
| 490M1 BF 490-1 | (2E)-(methoxyimino){2-[(2- methylphenoxy)methyl]phenyl}acetic acid | | |
| | | 299.33 g/mol | Molar mass: |

| Code Names | Chemical Names (IUPAC) | Structure |
|-------------------------------------|---|--|
| 490M2 BF 490-2 | (2E)-(2-{[2- (hydroxymethyl)phenoxy]methyl}phenyl)(methoxyimino) acetic acid | |
| 490M04 BF 490-5 | 2-({2-[(E)- carboxy(methoxyimino)methyl]phenyl}methoxy)benzoic acid | Molar mass: 315.33 g/mol |
| 490M6 | (2E)-[2-(hydroxymethyl)phenyl](methoxyimino)acetic acid | |
| 490M9 BF 490-9 | (2E)-{2-[(4-hydroxy-2- methylphenoxy)methyl]phenyl)(methoxyimino)acetic acid | O O O H |
| 490M15 BF 490-4 | methyl (2 <i>E</i>)-{2-[(4-hydroxy-2- methylphenoxy)methyl]phenyl}(methoxyimino)acetate | Molar mass: 315.33 g/mol |
| 490M28 (glucoronid of 490M15) | [2-{{2-[(E)- carboxy(methoxyimino)methyl]phenyl}methoxy)phenyl] methyl glucuronide | $H_{3}C^{-O}$ CH_{3} O OH H H OH H H H H H H H H H |
| 490M46 | methyl (2E)-(2-{[4-hydroxy-2- (hydroxymethyl)phenoxy]methyl)phenyl)(methoxyimino) acetate | HO H ₃ C O CH ₃ |
| 490M48 BF 490-03 | methyl (2 <i>E</i>)-(hydroxyimino){2-[(2- methylphenoxy)methyl]phenyl}acetate | CH ₃ O H ₃ C ^O |

850

| Code Names | Chemical Names (IUPAC) | Structure |
|--|--|---|
| 490M51 (sulfate conjugate of 490M46) | methyl (2 <i>E</i>)-(2-{[2-(hydroxymethyl)-4- (sulfooxy)phenoxy]methyl}phenyl)(methoxyimino) acetate | OH OH H_3C O OH OH OH OH OH OH OH |
| 490M54 | (2 <i>E</i>)-{2-[(5-hydroxy-2- methylphenoxy)methyl]phenyl)(methoxyimino)acetic acid | OH OH |
| 490M58 | [(1E)-1-(2-{[2-(hydroxymethyl)phenoxy]methyl)phenyl)-2- methoxy-2-oxoethylidene]azinic acid | OH OH H ₃ C |
| 490M66 (sulfate conjugate of 490M15) | methyl (2 <i>E</i>)-(methoxyimino)(2-{[2-methyl-4- (sulfooxy)phenoxy]methyl}phenyl)acetate | CH_3 O H_3C O CH_3 O CH_3 O O O O O O O O |
| 490M68/490M69 | 2-[carboxy(methoxyimino)methyl]benzoic acid | |
| 490M76/490M77 | Not applicable | |

Plant metabolism

The Meeting received plant metabolism studies in sugar beet, apples, grapes, and wheat following application of [phenoxy-¹⁴C]- and/or [phenyl-¹⁴C]-kresoxim-methyl.

On sugar beet, [phenoxy-¹⁴C]-kresoxim-methyl was applied in two foliar applications at a rate equivalent to 0.15 kg ai/ha each. The first application took place at BBCH 39 (leaves cover 90% of ground) and the second 3 weeks later or 28 days before harvest (BBCH stage not given). Samples of leaves and root were taken before and directly after the second treatment and at harvest.

Maximum TRR levels found were highest in leaves immediately after the second treatment (1.8 mg eq/kg) and remained fairly steady with 1.3–1.7 mg eq/kg until 28 days after last treatment (DALT). In roots, maximum TRR levels were up to 0.053 mg eq/kg at 0 DALT, but dropped to 0.008–0.009 mg eq/kg after 28 DALT.

Samples were sequentially extracted with methanol followed by water. Extracted radioactivity ranged between 91-99% TRR in leaves and 63–93% TRR in roots.

Due the low TRR, no attempts were undertaken to characterise the radioactivity in sugar beet roots. In sugar beet leaves, parent kresoxim-methyl accounted for 67–98% TRR (1.1–1.4 mg eq/kg). Major metabolites 490M1 and the sugar conjugate of 490M2 were identified in leaves at 28 DALT up to 9.7% TRR (0.12 mg eq/kg) and 9.2% TRR (0.12 mg eq/kg), respectively.

On apples, three different application schemes of [phenyl-14C]-kresoxim-methyl were applied:

- Foliar application (whole tree): 6×0.4 kg ai/ ha at the beginning of flowering (1), petal fall (2), application every 28–42 days (3–5) and 2 weeks before harvest (6). Samples were harvested 14 DALT.
- Early application (whole tree): 2×0.4 kg ai/ ha at the beginning of flowering and at petal fall. Samples were harvested 149 DALT.
- 3. Fruit spray treatment (leaves and branches were covered with foil): 2×0.8 kg ai/ ha at 6 weeks before harvest and 2 weeks before harvest. Samples were harvested 14 DALT.

Based on whole apple, the calculated TRR was highest after fruit treatment at up to 0.84 mg eq/kg. TRR levels were highest for all treatment regimes in apple peel at up to 5.7 mg eq/kg (fruit treatment), followed by apple core at up to 0.044 mg eq/kg (foliar application) and apple flesh at up to 0.036 mg eq/kg (foliar treatment). TRR in leaves ranged from 0.23– 1.0 mg eq/kg.

Samples of apple peel and pulp were extracted with methanol. Extracted radioactivity ranged between 86–98% TRR for all matrices.

In all three treatment regimes, parent kresoxim-methyl was the predominant residue accounting for 74–98% TRR (0.038–0.82 mg eq/kg). Additionally, metabolites 490M1, 490M2 and 490M9 were identified, but none occurred in amounts >3% TRR.

On grapes, [phenoxy-¹⁴C]- and [phenyl-¹⁴C]-kresoxim-methyl was applied in five foliar applications at a rate equivalent to 0.5 kg ai/ha per application with intervals of 14–21 days and harvested at 14 DALT.

Grapes were rinsed with methanol, followed by determination of the radioactivity in the rinsed grapes. TRR levels for the sum of radioactivity in the rinse and the grapes ranged between 3.6–4.7 mg eq/kg, with 37–38% of the TRR recovered in the rinse.

Rinsed, homogenised fruits were further extracted methanol or acetone/water in parallel. Extracted radioactivity for both solvents was similar, ranging between 46–60% TRR. In total, the initial methanol rinse and the subsequent extraction accounted for 86–92% TRR.

Parent kresoxim-methyl was the predominant residue accounting for 55–57% TRR (2.2–2.7 mg eq/kg). As a major metabolite, 490M2 (sum of free and conjugated) was identified at up to 14% TRR (0.55 mg eq/kg). Additionally, metabolites 490M1, 490M9 and 490M54 were identified, but none occurred in amounts >5.8% TRR.

On <u>wheat</u>, [phenyl-¹⁴C]-kresoxim-methyl was applied in two foliar application at 0.25 kg ai/ha (1×), or two foliar application at an exaggerated rate of 1.25 kg ai/ha (5×). The first treatment occurred for both application rates at growth stage BBCH 29 (leaf sheaths lengthen); while the second treatment occurred 56 days later at growth stage BBCH 52 (first ears just visible). Forage samples were taken at 4 hours and 55 days after the first treatment and at 4 hours after the second treatment. Straw, husk and grain samples were taken 64 days after the second treatment.

TRR levels in the 1× treatment group were highest in straw at up to 13 mg eq/kg, followed by forage (day 0 after 1st application) at up to 11 mg eq/kg. In the 5× treatment group TRR was highest in forage (day 0 after 1st application) at up to 76 mg eq/kg, followed by straw at up to 62 mg eq/kg. TRR levels in grain were significantly lower in both treatment group at up to 0.064 mg eq/kg for the 1× treatment and 0.31 mg eq/kg for the 5× treatment.

Samples were sequentially extracted with methanol followed by aqueous ammonia. Kresoxim-methyl conjugates in the extracts were hydrolysed with β -glucosidase and hesperidinase. Extracted radioactivity in all matrices except grain ranged between 90–100% TRR. For grain it was lower with 61–75% TRR.

The predominant residue in all matrices was parent kresoxim-methyl ranging from 17–40% TRR in grain to 64–97% TRR in forage and straw. As a major metabolite, conjugated 490M9 was identified at up to 11% TRR (1.0 mg eq/kg) in straw. Additionally, the Z-isomer of kresoxim-methyl and metabolites 490M1, 490M2 and 490M17 were identified in forage, straw and grain, but none occurred in amounts >3.9% TRR.

In summary, kresoxim-methyl was only moderately metabolised in studies performed with sugar beet, apple, grape and wheat. Parent kresoxim-methyl accounted for most of the residue with 55–98% TRR, which the exception of wheat grain (17–40% TRR). Major identified metabolites were 490M2 (unconjugated and conjugated) in grapes (14% TRR) and 490M9 (conjugated) in straw (11% TRR). All major identified metabolites were also found in the rat.

Animal metabolism

Information was available on the metabolism of kresoxim-methyl in laboratory animals, lactating goats and laying hens. The evaluation of the metabolism studies in rats was carried out by the WHO group.

In <u>lactating goats</u>, the metabolic fate of kresoxim-methyl was investigated using [phenoxy-¹⁴C]- and [phenyl-¹⁴C]radiolabelled kresoxim-methyl. In the first study, [phenyl-¹⁴C]-radiolabelled kresoxim-methyl was administered orally to two lactating goats. Goat A received 7.1 ppm (0.26 mg/kg bw) for five consecutive days, while goat B received 454 ppm (21 mg/kg bw) for eight consecutive days. In a second study [phenoxy-¹⁴C]-radiolabelled kresoxim-methyl was administered for five consecutive days to one lactating goat by gavage at 13.9 ppm feed (1.6 mg/kg bw)

For both labels most of the administered radioactivity was recovered from urine (59–70% AR) and faeces (18–25% AR). In edible tissues residues were low with up to 0.07% AR in the liver. The highest TRRs were found in kidney (0.052–14 mg eq/kg) and liver (0.041–6.8 mg eq/kg). In milk, the radioactive residues ranged between 0.003–2.7 mg eq/kg for both labels.

Residue levels in milk reached a plateau in goat A (dosed with [phenyl-¹⁴C]-kresoxim-methyl) and the [phenoxy-¹⁴C]radiolabelled kresoxim-methyl dosed goat after approximately 3 days. However, for goat B, which received the exaggerated dose of [phenyl-¹⁴C]-kresoxim-methyl, no plateau was reached after 8 days.

Milk and tissue samples of the [phenyl-¹⁴C]-kresoxim-methyl dosed goat B were sequentially extracted with methanol followed by water, while tissues of the [phenoxy-¹⁴C]-radiolabelled kresoxim-methyl dosed goat were extracted with acetonitrile followed by water. Resulting extraction rates for both methods were 101% TRR in milk, 43–63% TRR in liver, 86–98% TRR in kidney, 72% TRR in muscle and 72% TRR in fat.

Identification and characterisation of the radioactivity was done only for the [phenyl-¹⁴C]-kresoxim-methyl dosed goat receiving the exaggerated dose and for the [phenoxy-¹⁴C]-radiolabelled kresoxim-methyl dosed goat (only in liver and kidney). Kresoxim-methyl was not detected in milk and tissues, except for fat at up to 6.6% TRR (0.024 mg eq/kg). However, several metabolites were detected for both labels at significant levels: 490M1 in muscle, fat, liver and kidney ranging between 14–26% TRR (0.059–3.0 mg eq/kg); 490M2 in milk, muscle, fat, liver and kidney ranging between 11–34% TRR (0.032–4.6 mg eq/kg); 490M9 in milk, muscle, liver and kidney ranging between 10–63% TRR (0.023–4.0 mg eq/kg)

In <u>laying hens</u>, the metabolic fate of kresoxim-methyl was investigated using [phenyl-¹⁴C]-radiolabelled kresoxim-methyl. The compound was administered for 6 consecutive days to 2 groups of laying hens by gavage. Group A was dosed daily at 10 ppm (1 mg/kg bw) and group B at 180 ppm (18 mg/kg bw).

Most of the administered radioactivity was recovered from excreta (72-83% AR). For both dosing groups, liver had the highest TRR (0.082 / 7.0 mg eq/kg), followed by kidney (0.065 / 6.4 mg eq/kg).

TRR levels in eggs did not reach a plateau after 5–6 days.

Egg and tissue samples were sequentially extracted with methanol followed by water. Resulting extraction rates were 76% TRR in egg, 65–82% TRR in liver, 84% TRR in muscle, 92% TRR in fat and 66–87% TRR in skin.

Kresoxim-methyl was only detected in significant amounts in skin at up to 11% TRR (0.082 mg eq/kg) and fat at up to 41% TRR (0.31 mg eq/kg).

However, several metabolites were detected at significant levels: 490M9 in liver at 20% TRR (1.4 mg eq/kg); 490M15 (including its glucuronide conjugate 490M28) in fat at 17% TRR (0.12 mg eq/kg) and in liver at 17% TRR (1.1 mg eq/kg); 490M48 in eggs at 14% TRR (0.92 mg eq/kg) and 490M58 in skin at 10% TRR (0.079 mg eq/kg). Metabolites 490M51 (sulphate of 490M46) and 490M66 (sulphate of 490M15) were not chromatographically resolved and were detected in muscle and eggs from 16–20% TRR (0.019–0.035 mg eq/kg);

In summary, strong metabolic degradation of kresoxim-methyl was observed. In lactating goats, the sum of unconjugated metabolites 490M1, 490M2 and 490M9 represented most of the residue. Although, a more complex situation was seen in laying hens, where the pattern of significant metabolites was quantitatively different, the major metabolic pathways were the same as in the goat.

Environmental fate in soil & water

The Meeting received new information for anaerobic and aerobic degradation of kresoxim-methyl in soil. These reports were kinetic evaluations based on laboratory soil degradation studies and field dissipation studies previously submitted in the context of the first JMPR evaluation in 1998.

These studies were not taken into consideration, as uses of kresoxim-methyl only comprise of foliar applications.

The Meeting received two confined rotational crop metabolism studies.

The first study was conducted with [phenyl-14C]-kresoxim-methyl applied at a rate equivalent to 0.3 kg ai/ha to a sandy loam soil. After a plant-back interval (PBI) of 30 days, lettuce, green beans, carrots and wheat were planted/sowed. At harvest, the

highest total radioactive residues were found in the roots of lettuce, beans and wheat, ranging from 0.23–1.1 mg eq/kg. This was followed by bean forage at 0.21 mg eq/kg and wheat straw at 0.15 mg eq/kg. In edible commodities radioactive residues > 0.01 mg eq/kg were only found in lettuce heads (0.02 mg eq/kg).

Samples were extracted with methanol and kresoxim-methyl conjugates in the extracts were hydrolysed with β -glucosidase and hesperidinase. Extracted radioactivity in lettuce (immature), carrot greens, bean forage and wheat straw ranged between 54–82% TRR, except for wheat forage where extractability was lower at 38% TRR.

Due to the low radioactivity, only lettuce (immature), carrot greens, bean forage, wheat forage and straw were further characterised. Only metabolites 490M2 and 490M9 (including their conjugates) were detected at levels >0.01 mg eq/kg in bean forage (up to 0.038 mg eq/kg) and wheat straw (up to 0.013 mg eq/kg).

The second confined rotational crop study was conducted with lettuce, radish and wheat, planted on a sandy loam soil treated with [phenyl-¹⁴C]-kresoxim-methyl at a rate equivalent to 1.5 kg ai/ha after plant-back intervals (PBIs) of 30, 120 and 365 days.

TRR levels found in the model crops generally declined with longer PBIs, ranging between 0.011–0.17 mg eq/kg at 365 days PBI, compared to 0.094–2.0 mg eq/kg at 30 days PBI.

Samples were sequentially extracted with methanol followed by water. At least 49% TRR or more was extracted from immature and mature lettuce, radishes, wheat forage and chaff at 30 days PBI and wheat straw at 30 and 120 days PBI. Extractability was lowest for wheat grain for all PBIs, ranging between 16–17% TRR.

Parent kresoxim-methyl was only found in mature lettuce (30 days PBI) at up to 0.003 mg eq/kg (6.1% TRR) and immature radish (30 days PBI) at up to 0.040 mg eq/kg (9.1% TRR). As a major metabolite, 490M78 was found in immature radish (30 days PBI) and mature radish root and leaves (30 days PBI) at up to 0.19 mg eq/kg (44% TRR). At later PBIs, levels of 490M78 were significantly lower.

Other identified metabolites comprised of 490M06, 490M68/490M69 and 490M76/490M77 at levels ranging between 0.14–0.26 mg eq/kg (8.7–16% TRR). However, these metabolites were identified only in wheat straw at 30 days PBI, but not at later PBIs.

Hydrolytic treatments of the post extraction solids indicated the residues of kresoxim-methyl are further degraded to polar components and became integrated into natural constituents (e.g. starch, lignin).

In summary the Meeting concluded that a significant transfer of kresoxim-methyl residues from soil to succeeding crops is not expected.

Methods of analysis

The Meeting received analytical methods for the determination of kresoxim-methyl and metabolites 490M1, 490M2 and 490M9 in plant matrices.

For matrices of plant origin, the basic principle of most methods employed extraction with methanol or methanol/water, followed by clean-up using liquid-liquid partitioning with isooctane or dichloromethane alone, or in combination with additional clean-up steps, such as SPE on a silica gel, C18 or NH₂ cartridge. Parent kresoxim-methyl was either directly determined by LC-MS/MS, GC-ECD or GC-MS, or hydrolysed (10 M KOH, 1hour) to metabolite 490M1 (kresoxim acid) followed by LC-LC-UV detection. Hence, it should be noted that for the latter method always the sum of kresoxim-methyl and 490M1 is determined. Conjugates of metabolites 490M2 and 490M9 were cleaved by enzymatic hydrolysis (hesperidinase and β -glucosidase) and determined by LC-LC-UV, as well. Although, the limit of quantification for most methods was at 0.01 or 0.05 mg/kg per analyte, one method had higher LOQs of 0.2 and 0.4 mg/kg.

For animal matrices, methods were provided for metabolites 490M1, 490M2 and 490M9, but not for parent kresoximmethyl.

In animal matrices, metabolite 490M1 was determined in tissues, eggs and milk by extraction with methanol without any additional clean-up and LC-MS/MS detection with a LOQ of 0.01 mg/kg. Alternatively, after extraction with methanol, clean-up by partitioning with dichloromethane, SPE on a NH₂-cartridge and preparative HPLC on a RP C18 column, followed by LC-LC-UV analysis with a LOQ of 0.001 mg/kg was employed for metabolites 490M1, 490M2 and 490M9 in tissues. In milk, metabolites 490M2 and 490M9 were determined by extraction with acetone followed by clean-up using liquid-liquid partition with isooctane or dichloromethane, SPE on a NH₂-cartridge and LC-LC-UV analysis with an LOQ of 0.001 mg/kg.

The Meeting concluded that suitable data generation and monitoring methods are available to measure residues of kresoxim-methyl in plants only and/or metabolites 490M1, 490M2 and 490M9 in plant and animal commodities. It was also noted, that a method for the determination of metabolites 490M2 and 490M9 in eggs was missing.

No multi-residue method for the determination of parent kresoxim-methyl or its metabolites was submitted. The Meeting noted that in the European Reference Laboratories (EURL data pool) the QuEChERS method was validated for parent kresoximmethyl in high water content, high acid content and dry matrices with an LOQ of 0.01 mg/kg.

Stability of residues in stored analytical samples

The Meeting received information on the storage stability of kresoxim-methyl and metabolites 490M2 and 490M9 (as glucoside and aglycon) in a variety of plant matrices stored at -10 to -28 °C.

Kresoxim-methyl was stable in high starch and high protein matrices for at least 24 months, while in high water and acid content matrices for least 12 months. In high oil matrices kresoxim-methyl was stable for a maximum of 3 months in soya beans and for at least 6 months in pecan nuts.

Metabolite 490M2 was stable in high starch, high protein and high oil matrices for at least 24 months, while the respective glucoside was stable in high water and acid content matrices for least 12 months and in high oil matrices for at least 6 months.

Metabolite 490M9 was stable in high starch and high protein matrices for at least 24 months, while in high oil matrices (soya bean) 490M9 was not stable (53% after 1 month). The respective glucoside of 490M9 was stable in high water and acid content matrices for least 12 month and in high oil matrices for at least 6 months.

For animal matrices, the Meeting received information on the storage stability of metabolites 490M1, 490M2 and 490M9 in tissues and milk stored at -18 °C.

Metabolites 490M2 and 490M9 in milk and metabolites 490M1, 490M2 and 490M9 in tissues were considered stable for at least 15 months.

Definition of the residue

In the <u>plant metabolism studies</u> conducted on sugar beet, apples, grapes and wheat the predominant residue was parent kresoximmethyl at 74–98% TRR in apples, 55–57% TRR in grapes and 17–40% TRR in wheat grain. In feed matrices, residues of kresoximmethyl ranged from 67–98% TRR in sugar beet leaves and 64–97% TRR in wheat forage and straw.

In <u>confined rotational crops</u> parent kresoxim-methyl was only detected in significant amounts in immature radish at up to 9.1% (0.04 mg eq/kg) at 30 days PBI, while levels in mature crops and/or later PBIs were constantly lower or non-detected. At 30 days PBI, metabolite 490M78 was found in radish root, immature radish and mature radish leaves at up to 44% TRR (0.19 mg eq/kg). At later PBIs, levels of 490M78 declined to around 0.01 mg eq/kg or lower. In summary, a significant transfer of kresoxim-methyl residues from soil to succeeding crops is not expected.

The Meeting concluded that parent kresoxim-methyl is the major residue in primary crops and is a suitable marker compound for compliance with MRLs. Analytical methods are capable of measuring kresoxim-methyl in all plant matrices.

For dietary exposure purposes, the metabolites found in primary metabolism studies at potentially significant levels were 490M2 (free and conjugated) in grapes (14% TRR) and 490M9 (conjugated) in wheat straw (11% TRR).

In supervised field trials metabolite 490M2 occurred in grapes and mango at levels similar or higher (up to 2.5 fold) compared to the levels of kresoxim-methyl. Also, metabolite 490M9 occurred in field trials with grapes at levels similar or higher (up to 2.0 fold) compared to the levels of kresoxim-methyl.

In a nature of residue study during processing, it was demonstrated that parent kresoxim-methyl is hydrolysed to kresoxim acid (490M1) under the conditions of sterilisation at up to 71%.

The Meeting concluded that residues of the plant metabolites 490M2 and 490M9, including their conjugates, may add significantly to the overall dietary exposure to kresoxim-methyl residues. Additionally, metabolite 490M1 (kresoxim acid) was considered relevant since it can be formed during processing.

Kresoxim was toxicologically evaluated by the current Meeting. The Meeting concluded that metabolites 490M1 and 490M9 were covered by the toxicological reference values of parent kresoxim-methyl.

The Meeting noted that as no specific data were available on the toxicity of the metabolite 490M2, the TTC approach could be applied⁴. The estimated exposure based on plant and animal commodities, using worst case assumptions to assess exposure that the estimated exposure is below the applicable threshold of toxicological concern for Cramer Class 3 compounds. The Meeting concluded that no dietary risk from this metabolite can be expected from the uses considered by the current Meeting.

The Meeting decided to include 490M1and 490M9, including their conjugates, into the residue definition for dietary exposure purposes.

⁴ See Toxicology section of the 2018 JMPR Report for further details

In <u>lactating goats</u>, kresoxim-methyl was only detected in fat at 6.6% TRR (0.024 mg eq/kg), following treatment at an exaggerated rate of 454 ppm and a short interval (4 hours) between the last dose and sacrifice. Major metabolites in milk, were 490M2 and 490M9 at 20% (0.039 mg eq/kg) and 63% TRR (0.12 mg eq/kg), respectively. In muscle, fat, liver and kidney, amounts of metabolites accounting for more than 10% TRR were 490M1 ranging between 11–26% TRR (0.006–3.0 mg eq/kg), 490M2 between 11–34% TRR (0.018–4.6 mg eq/kg) and 490M9 between 10–30% TRR (0.023–4.0 mg eq/kg).

A cow feeding study was conducted at treatment rates of 7, 21 and 70 ppm. In milk no residues >LOQ (0.002 mg/kg) were detected in all dosing groups. In tissues, no residues >LOQ (0.01 mg/kg) were detected in the lowest treatment group, except for 490M1 in kidney at 0.03 mg/kg. In the higher treatment groups, residues >LOQ were found for 490M1 in liver (up to 0.32 mg/kg), kidney (up to 0.20 mg/kg) and in fat (up to 0.089 mg/kg), as well as for 490M9 in liver (up to 0.016 mg/kg) and kidney (0.022 mg/kg).

Since parent kresoxim-methyl was only detected in a highly overdosed goat metabolism study, the Meeting concluded that no residues of above 0.01 mg/kg are expected in animal products under actual conditions. Although metabolite 490M2 was detected in significant relative amounts in both goat metabolism studies, only the exaggerated study resulted in significant absolute amounts. Since additionally metabolite 490M2 was not detected in the cow feeding study, the Meeting concluded that no residues above 0.01 mg/kg are expected in animal products.

In <u>laying hens</u>, kresoxim-methyl was found in significant amounts in skin and fat ranging between 11–41% TRR, following administration of 180 ppm kresoxim-methyl in the diet. Additionally several metabolites were detected at significant levels: 490M9 in liver at 20% TRR (1.4 mg eq/kg); 490M15 (including its glucuronide conjugate 490M28 in fat at 17% TRR (0.12 mg eq/kg)and in liver at 17% TRR (1.1 mg eq/kg); 490M48 in eggs at 14% TRR (0.92 mg eq/kg) and 490M58 in skin at 10% TRR (0.079 mg eq/kg). Metabolites 490M51 (sulphate of 490M46) and 490M66 (sulphate of 490M15) were not chromatographically resolved and were detected in muscle and eggs from 16–20% TRR (0.019–0.035 mg eq/kg). Taking into consideration a poultry dietary burden for kresoxim-methyl of maximal 0.35 ppm, the poultry metabolism study is about 500 times overdosed. Hence, no residues above 0.01 mg/kg for these metabolites are expected in animal products under actual conditions and therefore the contribution of these metabolites to the overall dietary exposure was considered as insignificant by the Meeting.

The Meeting concluded that metabolites 490M1 and 490M9 should be included in the residue definition for compliance with the MRL and dietary risk assessment for animal commodities.

In muscle and fat tissues of all animals investigated, residue concentrations of the sum of metabolites 490M1 and 490M9 were of similar proportions. The log P_{ow} of metabolite 490M1 is 0.15. The Meeting decided that residues of the sum of metabolite 490M1 and 490M9 are not fat-soluble.

Definition of the residue for compliance with the MRL for plant commodities: Kresoxim-methyl

Definition of the residue for dietary risk assessment for plant commodities: *Sum of kresoxim-methyl and metabolites (2E)-*(*methoxyimino)*{2-[(2-methylphenoxy)methyl]phenyl}acetic acid (490M1) and (2E)-{2-[(4-hydroxy-2methylphenoxy)methyl]phenyl}(methoxyimino)acetic acid (490M9) including their conjugates expressed as kresoxim-methyl

Definition of the residue for compliance with the MRL and dietary risk assessment for animal commodities: *Sum of metabolites (2E)-(methoxyimino){2-[(2-methylphenoxy)methyl]phenyl}acetic acid (490M1), and (2E)-{2-[(4-hydroxy-2-methylphenoxy)methyl]phenyl}(methoxyimino)acetic acid (490M9) expressed as kresoxim-methyl*

The residue is not fat-soluble.

Results of supervised residue trials on crops

Residues referred to as "total residue" comprises of the sum of kresoxim-methyl and metabolite 490M1, determined as 490M1 plus metabolite 490M9

Citrus fruits

The critical GAP for citrus in Japan allows three outdoor foliar applications of kresoxim-methyl at 25 g ai /hL and a PHI of 14 days, RTI not specified.

Outdoor field trials conducted with mandarins, natsudaidai, kabosu and sudachi in Japan were performed with three foliar applications of kresoxim-methyl at rates of 25 g ai/hL with a 7 ± 1 day interval between applications.

For the subgroup of mandarins, the ranked order of residues following GAP treatment was (n = 2): 2.3, 3.8 mg/kg.

For the subgroup of pummelo and grapefruits (natsudaidai), the ranked order of residues following GAP treatment was (n = 2): 0.92, 1.8 mg/kg.

For the subgroup of lemons and limes (kabosu and sudachi), the ranked order of residues following GAP treatment was (n = 3): < 0.10, 1.5, 4.6 mg/kg.

Based on the lack of data matching the GAP, the Meeting concluded that no maximum residue level could be estimated for kresoxim-methyl in citrus fruits based on the Japanese GAP.

Alternatively, a GAP for citrus in Turkey was provided allowing a maximum of four foliar applications of kresoxim-methyl at 12.5 g ai/hL with an RTI of 12 days and a PHI of 35 days. One trial matching the number of treatments and/or application rate within ±25% of the GAP was submitted.

Residues of kresoxim-methyl in oranges were (n = 1): 0.15 mg/kg. The trial had received three applications instead of four as required in the GAP, but was considered within 25% of the GAP.

Based on the lack of data matching the GAP, the Meeting concluded that no maximum residue level could be estimated for citrus fruits.

The Meeting agreed to withdraw the previous maximum residue level recommendation for kresoxim-methyl in oranges and grapefruit (0.5 mg/kg).

Apple

The GAPs provided for apple were from Japan allowing for three foliar applications of kresoxim-methyl at 33 g ai /hL and a PHI of 1 day, RTI not specified and from Brazil allowing three application at 10 g ai /hL (100 g ai/ha) with an interval of 8–12 days and a PHI of 35 days.

No corresponding supervised field trial data were submitted for either GAP.

The Meeting concluded to withdraw the previous maximum residue level recommendation for kresoxim-methyl in pome fruits (0.2 mg/kg).

Peach

The critical GAP for peach in Japan allows three foliar applications of kresoxim-methyl at 25 g ai /hL and a PHI of 1 day, RTI not specified. Field trials with peach conducted in Europe were performed with 4 foliar applications of kresoxim-methyl at rates of 20 or 30 g ai/hL with an 8–14 day interval between applications. The Meeting noted trials had one additional treatment but concluded that the first application did not contribute significantly to the residue level.

For estimating maximum residue levels of kresoxim-methyl in peach, the ranked order of residues following GAP treatment ($\pm 25\%$) was (n = 6): 0.13, 0.14, 0.25, 0.34, 0.41, 0.66 mg/kg.

For dietary risk assessment, the ranked order of the total residue following GAP treatment (±25%) was (n = 6): 0.18, 0.20, 0.32, 0.42, 0.51, 0.75 mg/kg

The Meeting estimated a maximum residue level of 1.5 mg/kg and a STMR of 0.37 mg/kg for kresoxim-methyl in peach.

Currants, black, red, white

The critical GAP for currant in the United Kingdom allows three foliar applications of kresoxim-methyl at 100 g ai /ha with a RTI of 10 days and a PHI of 14 days. Field trials with currant conducted in Europe were performed with 3 foliar applications of kresoxim-methyl at rates of 100 g ai/ha with a 14 day interval between applications.

For estimating maximum residue levels of kresoxim-methyl in currant, the ranked order of residues following GAP treatment were (n = 5): 0.13, 0.16, 0.18, 0.22 and 0.50 mg/kg.

For dietary risk assessment, the ranked order of the total residues following GAP treatment were (n = 5): 0.18(2), 0.21, 0.23 and 0.57 mg/kg.

The Meeting estimated a maximum residue level of 0.9 mg/kg and a STMR of 0.21 mg/kg for kresoxim-methyl in currants, black, red, white.

Strawberry

The critical GAP for strawberries in the Netherlands allows three outdoor foliar applications of kresoxim-methyl at 150 g ai /ha with a RTI of 10 days and a PHI of 7 days.

None of the trials provided matched the GAPs (e.g. no indoor use on label). Hence, the Meeting concluded that no maximum residue level could be estimated for kresoxim-methyl in strawberry.

Grapes

The critical GAP for grapes in the USA allows four foliar application of kresoxim-methyl at 224 g ai /ha with an RTI of 7 days and a PHI of 14 days. Matching field trials conducted in the USA were performed with four foliar applications of kresoxim-methyl at rates of 224 g ai/ha with a 10±1 day interval between applications.

For estimating maximum residue levels of kresoxim-methyl in grapes, the ranked order of residues following GAP treatment was (n = 18): < 0.05, 0.063, 0.071, 0.11, 0.12(2), 0.25, 0.27, 0.30, 0.31(2), 0.35, 0.36, 0.42(2), 0.61, 0.69, 0.91 mg/kg.

For dietary risk assessment, the ranked order of the total residue following GAP treatment was (n = 18): < 0.10, 0.11, 0.12, 0.17, 0.16, 0.30, 0.32, 0.35, 0.36, 0.37, 0.38, 0.40, 0.41, 0.47(2), 0.66, 0.74, 0.96 mg/kg.

The Meeting estimated a STMR value of 0.365 mg/kg and a maximum residue level of 1.5 mg/kg for kresoxim-methyl in grapes. The latter replaces the previous recommendation (1.0 mg/kg).

Olives

The critical GAP for olives in France allows three foliar applications of kresoxim-methyl at 100 g ai/ha and a PHI of 30 days. One application is performed when the fruits are present while the remaining two are performed between harvest and flowering.

Field trials with olives conducted in Europe were performed with one foliar application at BBCH 79 (fruit size about 90% of final size) to BBCH 85 (increasing of specific fruit colouring) of kresoxim-methyl at rates of 100 g ai/ha. The Meeting noted that despite a lower number of applications, trials were acceptable as the application of the two additional treatments included in the GAP occur when no fruits were present. Also, kresoxim-methyl is not systemic and no translocation into olive fruits from treatments before fruit formation are expected.

For estimating maximum residue levels of kresoxim-methyl in olives, the ranked order of residues following GAP treatment ($\pm 25\%$) was (n = 5): < 0.05(3), 0.11, 0.23 mg/kg.

For dietary risk assessment, the ranked order of the total residue following GAP treatment ($\pm 25\%$) was (n = 5): < 0.10(3), 0.14 and 0.16 mg/kg

The Meeting estimated a maximum residue level of 0.2 mg/kg and a STMR of 0.10 mg/kg for kresoxim-methyl in olives.

Mango

The critical GAP for mango in Brazil allows two foliar applications of kresoxim-methyl at 120 g ai /ha with a RTI of 15 days and a PHI of 7 days. Matching field trials conducted in Brazil were performed with two foliar applications of kresoxim-methyl at rates of 120 g ai/ha with a 14 day interval between applications.

For estimating maximum residue levels of kresoxim-methyl in mango, the ranked order of residues following GAP treatment was (n = 5): < 0.010(2), 0.014, 0.041, 0.049 mg/kg.

For dietary risk assessment, the ranked order of the total residue following GAP treatment was (n = 5): < 0.020(2), 0.024, 0.055, 0.059 mg/kg.

The Meeting estimated a maximum residue level of 0.1 mg/kg and a STMR of 0.024 mg/kg in mango.

Bulb onion

Kresoxim-methyl is registered for the use on shallots in Taiwan, Province of China, with two foliar applications of kresoxim-methyl at a rate of 400 g ai/ha with a 7 day interval between applications and 10 day PHI and in the Netherlands for bulb onion with 3 foliar applications at 200 g ai/ha with a 7 day interval between applications and the PHI covered by the growth stage.

None of the trials provided matched the GAPs. The Meeting concluded that no maximum residue level could be estimated for kresoxim-methyl in onion.

Garlic

The critical GAP for garlic in Brazil allows four foliar applications of kresoxim-methyl at 70 g ai/ha with a RTI of 10 days and a 7 day PHI. Available trials matched the critical GAP for garlic in Brazil allowing four application at 70 g ai /ha with a RTI of 7 days and a 7 day PHI.

For estimating maximum residue levels of kresoxim-methyl in garlic, the ranked order of residues following GAP treatment was (n = 4): < 0.01(4) mg/kg.

For dietary risk assessment, the ranked order of the total residue following GAP treatment was (n = 4): < 0.02(4) mg/kg.

858

The Meeting noted that garlic falls under category 3 of the minor crop classification, requiring a minimum of five supervised field trials to estimate maximum residue levels. However, the Meeting considered four trials were sufficient, as no residues > 0.01 mg/kg were detected. The Meeting estimated a maximum residue level of 0.01 mg/kg and a STMR of 0.02 mg/kg.

Leek

The critical GAP for leek in the Netherlands allows three foliar applications of kresoxim-methyl at 375 g ai /ha with a RTI of 10 days and a PHI of 14 days. Field trials with leek conducted in Europe were performed with three foliar applications of kresoxim-methyl at rates of 375 g ai/ha with a 9–14 day interval between applications and a 14 day PHI.

For estimating maximum residue levels of kresoxim-methyl in leek the ranked order of residues following GAP treatment was (n = 8): 2.7, 2.8, 3.1(2), 3.3(2), 3.4, 4.5 mg/kg.

For dietary risk assessment, the ranked order of the total residues following GAP treatment was (n = 8): 2.7, 2.8, 3.1(2), 3.3(2), 3.4, 4.5 mg/kg.

The Meeting estimated a maximum residue level of 10 mg/kg and a STMR of 3.2 mg/kg in leek.

Fruiting vegetables, Cucurbits – Cucumber and Summer Squashes

The critical GAP for cucumber and summer squashes in the USA allows four foliar application of kresoxim-methyl at 168 g ai /ha with a RTI of 7 days and a PHI of 0 days. Field trials with cucumber and summer squash conducted in the USA were performed with six foliar applications of kresoxim-methyl at rates of 196 g ai/ha with a 7 ± 1 day interval between applications. Although a higher number of applications in connection with a higher application rate were applied in the supervised field trials from the USA, the Meeting noted that cucumbers and summer squashes have a short period between flowering and harvest. When taking into account the intervals of 7 ± 1 days, the fruits did not receive all six applications until harvest. Since kresoxim-methyl is non-systemic, additional treatments before the formation of the edible part of the crop will not affect the terminal residue, making the USA trials suitable for an assessment.

Cucumber

For estimating maximum residue levels of kresoxim-methyl in cucumber, the ranked order of residues was (n = 8): < 0.05(4), 0.06(3), 0.11 mg/kg

For dietary risk assessment of cucumber, the ranked order of the total residue was (n = 8): < 0.10(4), 0.11(3), 0.17 mg/kg

Summer squash

For estimating maximum residue levels of kresoxim-methyl in summer squash, the ranked order of residues was (n = 5): < 0.05(4), 0.22 mg/kg.

For dietary risk assessment of summer squash, the ranked order of the total residue was (n = 5): < 0.10(4), 0.27 mg/kg

Melon

The critical GAP for melon in the USA allows four foliar applications of kresoxim-methyl at 168 g ai /ha with a RTI of 7 days and a PHI of 0 days. Available field trials with melon from France and Spain matching the USA GAP, received four foliar applications at 150 g ai/ ha with a 10-day interval between applications.

For estimating maximum residue levels of kresoxim-methyl in melon, the ranked order of residues was (n = 5): 0.05, 0.06, 0.13, 0.19, 0.20 mg/kg.

For dietary risk assessment of melon, the ranked order of the total residue was (n = 5): 0.10, 0.11, 0.18, 0.24, 0.25 mg/kg

The Meeting noted that the GAP in the USA is for cucurbits, including cucurbits with edible and in-edible peel. Since median residues of cucumbers, summer squash and melons were within a 5-fold range, the Meeting considered estimating a maximum residue level for the group. It was recognised that the residue population from trials on cucumber, summer squash and melon were not significantly different according to the Kruskal-Wallis H-test. Therefore, the Meeting decided to combine the data to estimate a maximum residue level and STMR for the group of fruiting vegetables, cucurbits.

For estimating a maximum residue level, the combined residues of kresoxim-methyl in cucumber, summer squash and melon in ranked order were (n = 18): < 0.05(8), 0.05, 0.06(4), 0.11, 0.13, 0.19, 0.20, 0.22 mg/kg.

For dietary risk assessment, the combined residues of kresoxim-methyl in cucumber, summer squash and melon in ranked order were (n = 18): < 0.10(8), 0.10, 0.11(4), 0.17, 0.18, 0.24, 0.25, 0.27 mg/kg.

The Meeting estimated a STMR of 0.105 mg/kg and a maximum residue level of 0.5 mg/kg for the group of fruiting vegetables, cucurbits. The latter replaces the previous recommendation for cucumber (0.05 mg/kg).

Sweet pepper

The critical GAP for sweet pepper in Brazil allows four foliar applications of kresoxim-methyl at 100 g ai/ha with a RTI of 10 days and a PHI of 3 days. Matching field trials with pepper conducted in Brazil were performed with 4 foliar applications of kresoxim-methyl at rates of 97 g ai/ha with a 7 ± 1 day interval between applications.

Ranked residues of kresoxim-methyl in sweet pepper from Brazil were (n = 4): < 0.01, 0.02, 0.03, 0.04 mg/kg.

The data on sweet pepper from Brazil are insufficient for an assessment. However, the Meeting noted that additional field trials with sweet pepper performed outdoors in Spain are available, which received four foliar applications at 250 g ai/ ha with a 10 day interval between applications.

Ranked residues of kresoxim-methyl in sweet pepper from Europe were (n = 2): 0.16, 0.44 mg/kg.

Since the European trials were overdosed, the Meeting decided that the proportionality principle could be applied in this case, as the application rate is not higher than $4 \times of$ the GAP rate. Therefore, a scaling factor of 0.4 was applied to residues >LOQ from Europe, resulting in a total residue population of (n = 6): < 0.01, 0.02, 0.03, 0.04, 0.064^{scaled}, 0.18^{scaled} mg/kg.

For dietary risk assessment, the corresponding ranked order of the total residue of kresoxim-methyl in pepper was (n = 6): < 0.02, 0.03, 0.04, 0.05, 0.084^{scaled}, 0.20^{scaled} mg/kg.

The Meeting estimated a maximum residue level of 0.3 mg/kg and a STMR of 0.045 mg/kg in sweet pepper.

Tomato

The critical GAP for tomato in Brazil allows two foliar applications of kresoxim-methyl at 200 g ai/ha with a RTI of 7 days and a PHI of 3 days.

None of the trials provided matched the GAPs. Hence, the Meeting concluded that no maximum residue level could be estimated for kresoxim-methyl in tomato.

Grape leaves

The critical GAP for grape leaves in Turkey allows three foliar applications of kresoxim-methyl at 30 g ai /ha with a RTI of 10 days and a PHI of 14 days. Field trials with grape leaves conducted in Europe were performed with three foliar applications of kresoxim-methyl at rates of 150 g ai/ha with an 8–10 day interval between applications.

None of the trials provided matched the GAPs. As a result the Meeting concluded that no maximum residue level could be estimated for kresoxim-methyl in grape leaves.

Sugar beets, beet roots and turnip

The critical GAP for beet root in Germany allows two foliar applications of kresoxim-methyl at 125 g ai /ha with a RTI of 10 days and a PHI of 28 days. For sugar beet and turnip, the GAP in Germany allows for one application at 125 g ai/ha with a PHI of 28 days. Field trials with sugar beet conducted in France and Germany were performed with two foliar applications of kresoxim-methyl at rates of 125 g ai/ha with a 21 day interval between applications.

For estimating maximum residue levels of kresoxim-methyl in sugar beets, the ranked order of residues following GAP treatment was (n = 10): < 0.05 (10) mg/kg.

For dietary risk assessment, the ranked order of the total residue following GAP treatment was (n = 10): < 0.10 (10)mg/kg.

The Meeting estimated a maximum residue level of 0.05(*) mg/kg and a STMR of 0 mg/kg in beet root, supported by evidence from a metabolism study performed with sugar beet, and decided to extrapolate its estimations to sugar beets and turnips.

Wheat

The critical GAP for wheat, rye and triticale in the United Kingdom allows two foliar applications of kresoxim-methyl at 125 g ai /ha (RTI not given) and the PHI is covered by conditions of use (last application up to BBCH 65). Field trials with wheat conducted in Europe were performed with two foliar applications of kresoxim-methyl at rates of 125 g ai/ha with an 11–24 day interval between applications and the last application at BBCH 69.

For estimating maximum residue levels of kresoxim-methyl in wheat grain, the ranked order of residues following GAP treatment was (n = 12): < 0.01(9), 0.01(2), 0.04 mg/kg.

For dietary risk assessment, the ranked order of residues following GAP treatment was (n = 12): $\leq 0.02(9)$, 0.02(2), 0.05 mg/kg.

The Meeting estimated a maximum residue level of 0.05 mg/kg and a STMR of 0.02 mg/kg. The Meeting decided to withdraw its previous recommendation of a maximum residue level for wheat of 0.05 mg/kg, to be replaced by a maximum residue level of 0.05 mg/kg for the subgroup of wheat grain.

Barley

The critical GAP for barley and oat in the United Kingdom allows two foliar applications of kresoxim-methyl at 125 g ai /ha (RTI not given) and the PHI is covered by conditions of use (last application up to BBCH 59). Field trials with barley conducted in Europe were performed with two foliar applications of kresoxim-methyl at rates of 125 g ai/ha with a 14–25 day interval between applications and the last application at BBCH 69 or 71.

For estimating maximum residue levels of kresoxim-methyl in barley grain, the ranked order of residues following GAP treatment was (n = 10): 0.01, 0.02(4), 0.03(2), 0.04, 0.06, 0.08 mg/kg.

For dietary risk assessment, the ranked order of residues following GAP treatment was (n = 10): 0.02, 0.03(4), 0.04(2), 0.05, 0.07, 0.09 mg/kg.

The Meeting estimated a maximum residue level of 0.15 mg/kg and a STMR of 0.035 mg/kg. The Meeting decided to withdraw its previous recommendation of a maximum residue level for barley of 0.1 mg/kg, to be replaced by a maximum residue level of 0.15 mg/kg for the subgroup of barley grain.

Pecan nuts

The critical GAP for pecan nuts in the USA allows three foliar applications of kresoxim-methyl at 168 g ai /ha with a RTI of 14 days and a PHI of 45 days. Field trials with pecan nuts conducted in the USA were performed with 8 foliar applications of kresoxim-methyl at rates of 224 g ai/ha with a 14–21 day interval between applications.

For estimating maximum residue levels of kresoxim-methyl in pecan nuts, the ranked order of residues following GAP treatment was (n = 6): < 0.05(6) mg/kg.

For dietary risk assessment, the ranked order of residues following GAP treatment was (n = 6): < 0.10(6) mg/kg.

Although all trials were overdosed, the Meeting concluded that recommendations could be given since all residues were <LOQ and estimated a maximum residue level of 0.05(*) mg/kg and a STMR of 0.10 mg/kg in pecan nuts.

Animal feeds

Sugar beet tops

The critical GAP for sugar beet in Germany allows one foliar applications of kresoxim-methyl at 125 g ai /ha and a PHI of 28 days. Field trials with sugar beet conducted in Europe were performed with two applications of kresoxim-methyl at rates of 125 g ai/ha with a 21 day interval between applications.

None of the trials provided matched the GAP for sugar beet.

Cereal forage

Data was provided for cereal whole plant with the last application performed at BBCH 69–71 and 0 day PHI. The Meeting concluded that the growth stage of the last treatment was not at the forage stage anymore and decided not to consider cereal forage for the livestock dietary burden calculation.

Cereal straw

The critical GAP for wheat and barley in the United Kingdom allows two foliar applications of kresoxim-methyl at 125 g ai /ha (RTI not given) and the PHI is covered by conditions of use (last application up to BBCH 65 and 59 for wheat and barley, respectively). Field trials with wheat conducted in Europe were performed with two foliar applications of kresoxim-methyl at rates of 125 g ai/ha with an 11–24 day interval between applications.

Residues of total kresoxim-methyl in wheat and barley straw following GAP treatment (±25%) were (n = 22): 0.05, 0.08, 0.15, 0.17(2), 0.22, 0.27, 0.30, 0.33, 0.37, 0.48, 0.52, 0.55, 0.59, 0.62, 0.63, 0.68, 0.76, 0.89, 1.3, 1.6 and 2.3 mg/kg as received.

The Meeting estimated a highest residue of 2.3 mg/kg (as received) for total kresoxim-methyl in cereal straw, a median residue of 0.50 mg/kg (as received) and a maximum residue level of 3 mg/kg (DM, based on 90% DM content). The latter replaces the previous recommendation for straw and fodder (dry) of cereal grains (5 mg/kg).

Fate of residues during processing

The Meeting received information on the hydrolysis of ¹⁴C-labelled-kresoxim-methyl as well as processing studies using unlabelled kresoxim-methyl on apples, grapes, gherkin and olive for oil production.

In a hydrolysis study using radiolabelled kresoxim-methyl typical processing conditions were simulated (pH 4,5 and 6 with 90 °C, 100 °C and 120 °C for 20, 60 and 20 minutes). Significant hydrolysis of kresoxim-methyl to kresoxim acid (490M1) was observed at the conditions of sterilisation at up to 71%, while 24% of parent kresoxim-methyl remained.

The Meeting concluded that kresoxim-methyl is stable under the conditions of pasteurisation, boiling, baking and brewing, but not under condition of sterilisation.

For the estimation of maximum residue levels, processing factor according to the residue definition (*kresoxim-methyl*) are summarised below.

| Raw commodity | Processed commodity | Individual processing factors | Median or best estimate processing factor | RAC MRL (mg/kg) | RAC MRL × PF (mg/kg) |
|------------------|---------------------|----------------------------------|--|--------------------|-------------------------|
| Grapes | Raisins | 1.6, <u>1.6</u> , 2.2 | 1.6 | 1.5 | 2.4 |
| Olives | Virgin oil | 4.5 | 4.5 | 0.2 | 0.9 |

The Meeting estimated a maximum residue level of 1 mg/kg for virgin oil and 3 mg/kg in raisins. The latter replaces the previous recommendation for raisin (2 mg/kg).

For the estimation of dietary intake of processed commodities, processing factor according to the residue definition (*Sum of kresoxim-methyl and metabolites 490M1and 490M9 including their conjugates expressed as kresoxim-methyl*) are summarised below.

| Raw commodity | Processed commodity | Individual processing factors | Median or best estimate processing factor | STMR _{RAC} (mg/kg) | STMR-P = STMR _{RAC} × PF (mg/kg) |
|------------------|---------------------|-------------------------------|---|--------------------------------|--|
| | Wine | 0.12, 0.20, 0.31, 0.46 | 0.26 | 0.365 | 0.095 |
| | Juice | 0.10, 0.48, 0.68 | 0.48 | 0.365 | 0.18 |
| Grapes | Must, cold | 0.27, 0.30, 0.31, 0.64 | 0.31 | 0.365 | 0.11 |
| | Raisins | 1.5, 1.6, 1.8 | 1.6 | 0.365 | 0.58 |
| | Wet pomace | 0.94, 2.0, 2.4, 2.6 | 2.2 | 0.365 | 0.80 |
| Olives | Virgin oil | 3.4 | 3.4 | 0.10 | 0.34 |

Residues in animal commodities

Farm animal feeding studies

The Meeting received one feeding study involving kresoxim-methyl on lactating cows. No poultry feeding study was submitted.

The study was conducted with parent kresoxim-methyl at treatment rates of 7, 21 and 70 ppm. In milk, skim milk and cream residues of metabolites 490M2 and 490M9 were <LOQ (0.002 mg/kg) in all dosing groups throughout the study.

In the 7 ppm treatment group no residues >LOQ (0.01 mg/kg) were detected in tissues, except for 490M1 in kidney at up to 0.034 mg/kg (mean: 0.030 mg/kg).

In the 21 ppm treatment group residues were < 0.01 mg/kg in muscle. However, in liver, 490M1 and 490M9 were found at up to 0.037 mg/kg (mean: 0.027 mg/kg) and 0.020 mg/kg (mean: 0.016 mg/kg), respectively. In kidney, 490M1 and 490M9 were found at up to 0.16 mg/kg (mean: 0.010 mg/kg) and 0.019 mg/kg (mean: 0.014 mg/kg), respectively. In fat, 490M1 was found at up to 0.041 mg/kg (mean: 0.030 mg/kg).

In the 70 ppm treatment group residues were < 0.01 mg/kg in muscle. In liver, 490M1 and 490M9 were found at up to 0.040 mg/kg (mean: 0.032 mg/kg) and 0.021 mg/kg (mean: 0.015 mg/kg), respectively. In kidney, 490M1 and 490M9 were found at up to 0.39 mg/kg (mean: 0.020 mg/kg) and 0.047 mg/kg (mean: 0.022 mg/kg), respectively. In fat only, 490M1 was found at up to 0.13 mg/kg (mean: 0.089 mg/kg).

It was noted that 490M9 was not measured in muscle. However, since in the lactating goat metabolism studies 490M9 occurred in muscle at levels half of 490M1, it is assumed that 490M9 in the feeding study is < 0.01 mg/kg as well.

Estimated maximum and mean dietary burdens of livestock

Dietary burdens were calculated for beef cattle, dairy cattle, broilers and laying poultry based on feed items evaluated by the JMPR. The dietary burdens, estimated using the OECD diets listed in Appendix IX of the 2016 edition of the FAO manual, are presented in Annex 6 and summarised below

862

| Livestock dietary bur | den, Sum of kre | soxim-methyl, | 490M1and 490 | IM9, ppm of dry | matter diet | | | |
|-----------------------|-----------------|---------------|-------------------|--------------------|-------------------|------------------|-------|-------|
| | US-Canad | US-Canada | | EU | | Australia | | |
| | max. | Mean | max. | mean | max. | Mean | max. | Mean |
| Beef cattle | 0.28 | 0.077 | 0.87 | 0.20 | 3.2 ^a | 1.5 ^b | 0.028 | 0.028 |
| Dairy cattle | 0.31 | 0.075 | 0.86 | 0.19 | 3.1 ^{ab} | 1.5 ^b | 0.15 | 0.044 |
| Poultry – broiler | 0.030 | 0.030 | 0.061 | 0.028 | 0.025 | 0.025 | 0.004 | 0.004 |
| Poultry – layer | 0.030 | 0.030 | 0.33 ^c | 0.093 ^d | 0.015 | 0.015 | none | none |

^{a)} Highest maximum beef or dairy cattle burden suitable for maximum residue level estimates for mammalian tissues

^{b)} Highest maximum dairy cattle burden suitable for maximum residue level estimates for mammalian milk

c) Highest mean beef or dairy cattle burden suitable for STMR estimates for mammalian meat and milk

^{d)} Highest maximum broiler or laying hen burden suitable for maximum residue level estimates for poultry products and eggs

e) Highest mean broiler or laying hen burden suitable for STMR estimates for poultry products and eggs

none no relevant feed items

Animal commodities maximum residue levels

For <u>beef and dairy cattle</u>, a maximum and mean dietary burden of 3.2 ppm and 1.5 ppm were estimated, respectively. The estimated dietary burdens are evaluated against a lactating cow feeding study involving administration of kresoxim-methyl at 7, 21 and 70 ppm.

For maximum residue level estimation, the high residues in the tissues were calculated by taking the maximum dietary burden (3.2 ppm) as a proportion of the lowest feeding level (7 ppm) multiplied with the highest tissue concentrations from individual animals within this feeding group.

The STMR values for the tissues were calculated by taking the mean dietary burden (1.5 ppm) as a proportion of the lowest feeding level (7 ppm) multiplied by the feeding-level mean residue.

| Maximum residue level beef or dairy | Feed level (ppm) for milk | Sum of 490M1 and 490M9 as parent equivalents in | Feed level (ppm) for tissue | Sum of 490M1 and 490M9 as parent equivalents ^a (mg/kg) | | | |
|--|------------------------------|---|--------------------------------|---|--------|--------|--------|
| cattle | residues | milk (mg/kg) | residues | Liver | Kidney | Muscle | Fat |
| Feeding study | 7.0 | < 0.004 | 7.0 | < 0.02 | 0.044 | < 0.02 | < 0.02 |
| Dietary burden and | 3.2 | < 0.002 | 3.2 | < 0.01 | 0.020 | < 0.01 | < 0.01 |
| highest residue | | | | | | | |

^{a)} Metabolite 490M9 in muscle was estimated at < 0.01mg/kg.

| STMR beef or dairy cattle | Feed level (ppm) for milk | Sum of 490M1 and 490M9Feed levelas parent equivalents in(ppm) for tiss | | Sum of 490M1 and 490M9 as parent e equivalents ^a (mg/kg) | | | |
|------------------------------------|------------------------------|--|----------|--|--------|---------|---------|
| | residues | milk (mg/kg) | residues | Liver | Kidney | Muscle | Fat |
| Feeding study | 7.0 | < 0.004 | 7.0 | < 0.02 | 0.040 | < 0.02 | < 0.02 |
| Dietary burden and mean residue | 1.5 | < 0.001 | 1.5 | < 0.004 | 0.009 | < 0.004 | < 0.004 |

^{a)} Metabolite 490M9 in muscle was estimated at < 0.01 mg/kg.

The Meeting concluded that residues >0.01 mg/kg are expected in kidney and estimated maximum residues levels of 0.05 mg/kg and a STMR of 0.009 mg/kg edible offal (mammalian). For all other tissues and milk the Meeting concluded that no residues > 0.01 mg/kg are expected and estimated maximum residue levels of 0.02(*) mg/kg for mammalian meat, milks, fat and as well as STMR value of 0.

For <u>poultry</u> a maximum and mean dietary burden of 0.33 ppm and 0.093 ppm were estimated, respectively. However, no farm animal feeding studies were provided. Laying hen metabolism studies involved administration of 180 ppm kresoxim-methyl in the diet, which is about 500 times overdosed compared to the expected maximum dietary burden. Extrapolation of the highest residue found in poultry tissues and egg (metabolite 490M9 at 1.4 mg eq/kg) to the maximum dietary burden would result in residues at up to 0.003 mg/kg. Therefore, the Meeting concluded that no residues > 0.01 mg/kg are expected in eggs and poultry tissues and estimated maximum residues levels of 0.02(*) mg/kg for poultry meat, eggs, fat and edible offal of as well as STMR value of 0.

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 assessments.

Definition of the residue for compliance with the MRL for plant commodities: Kresoxim-methyl

Definition of the residue for dietary risk assessment for plant commodities: *Sum of kresoxim-methyl and metabolites (2E)-*(*methoxyimino*){2-[(2-methylphenoxy)methyl]phenyl}acetic acid (490M1) and (2E)-{2-[(4-hydroxy-2methylphenoxy)methyl]phenyl}(methoxyimino)acetic acid (490M9) including their conjugates expressed as kresoxim-methyl

Definition of the residue for compliance with the MRL and dietary risk assessment for animal commodities: *Sum of metabolites (2E)-(methoxyimino){2-[(2-methylphenoxy)methyl]phenyl}acetic acid (490M1), and (2E)-{2-[(4-hydroxy-2-methylphenoxy)methyl]phenyl}(methoxyimino)acetic acid (490M9) expressed as kresoxim-methyl*

The residue is not fat-soluble.

The Meeting concluded that if future uses of kresoxim-methyl result in an increase of the exposure for metabolite 490M2, a reconsideration of the residue definition for dietary exposure purposes may become necessary.

Maximum residue levels and dietary exposure

| Commodity | | Recommended maximum residue level, mg/kg | | STMR or STMR-P, mg/kg | HR or highest residue, mg/kg |
|-----------|--|--|----------|--------------------------|------------------------------|
| CCN | Name | New | Previous | | |
| GC 0640 | Barley | W | 0.1 | | |
| GC 2087 | Barley, subgroup of (includes all commodities in this subgroup) | 0.15 | - | 0.035 | |
| VR 0574 | Beet root | 0.05* | - | 0 | |
| VC 0424 | Cucumber | W | 0.05* | | |
| FB 0021 | Currants, Black, Red, White | 0.9 | - | 0.21 | |
| DF 0269 | Dried grapes (=currants, raisins and sultanas) | 3 | 2 | 0.58 | |
| MO 0105 | Edible offal (Mammalian) | 0.05 | 0.05* | 0.009 | |
| PE 0112 | Eggs | 0.02* | - | 0 | |
| VC 0045 | Fruiting vegetables, Cucurbits, Group of (includes all commodities in this group) | 0.5 | - | 0.105 | |
| VA 0381 | Garlic | 0.01 | - | 0.02 | |
| FB 0269 | Grape | 1.5 | 1 | 0.365 | |
| FC 0203 | Grapefruit | W | 0.5 | | |
| VA 0384 | Leek | 10 | - | 3.2 | |
| MF 0100 | Mammalian fats (except milk fats) | 0.02* | 0.05* | 0 | |
| FI 0345 | Mango | 0.1 | - | 0.024 | |
| MM 0095 | Meat (from mammals other than marine mammals) | 0.02* | 0.05* | 0 | |
| ML 0106 | Milks | 0.02* | 0.01* | 0 | |
| OC 0305 | Olive oil, Virgin | 1 | - | 0.34 | |
| SO 0305 | Olives for oil production | 0.2 | - | 0.10 | |
| FC 0004 | Oranges, Sweet, Sour (including Orange- like hybrids): several cultivars | W | 0.5 | | |
| FS 0247 | Peach | 1.5 | - | 0.37 | |
| TN 0672 | Pecan nuts | 0.05* | - | 0.10 | |
| VO 0445 | Peppers, sweet | 0.3 | - | 0.045 | |

| Commodity | 5 | | Recommended maximum residue level, mg/kg | | HR or highest residue, mg/kg |
|-----------|--|--------|---|-------------------|------------------------------|
| CCN | Name | New | Previous | | |
| FP 0009 | Pome fruits | W | 0.2 | | |
| PF 0111 | Poultry fats | 0.02* | - | 0 | |
| PM 0110 | Poultry meat | 0.02* | 0.05* | 0 | |
| P0 0111 | Poultry, Edible offal of | 0.02* | - | 0 | |
| AS 0081 | Straw and fodder (dry) of cereal grains | 3 (DM) | 5 | Median: 0.50 (as) | Highest: 2.3 (as) |
| VR 0596 | Sugar beet | 0.05* | - | 0 | |
| FT 0305 | Table olives | 0.2 | - | 0.10 | |
| VR 0497 | Turnip | 0.05* | - | 0 | |
| GC 0654 | Wheat | W | 0.05* | | |
| GC 2086 | Wheat, subgroup of (includes all commodities in this subgroup) | 0.05 | - | 0.02 | |

Dietary exposure and feed burden only

| Commodity | | | | STMR or STMR-P, mg/kg | HR or highest residue, mg/kg |
|-----------|--------------------|---|---|--------------------------|------------------------------|
| | Grapes, wet pomace | - | - | 0.80 | - |
| | Grape Wine | - | - | 0.095 | |
| | Grape Juice | - | - | 0.18 | |
| | Grape Must | - | - | 0.11 | |

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The ADI for kresoxim-methyl is 0–0.3 mg/kg bw. The International Estimated Daily Intakes (IEDIs) for kresoxim-methyl were estimated for the 17 GEMS/Food Consumption Cluster Diets using the STMR or STMR-P values estimated by the JMPR. The results are shown in Annex 3 of the 2018 JMPR Report. The IEDIs ranged from 0–0.4% of the maximum ADI.

The Meeting concluded that long-term dietary exposure to residues of kresoxim-methyl from uses considered by the JMPR is unlikely to present a public health concern.

Acute dietary exposure

The 2018 JMPR decided that an ARfD for kresoxim-methyl is unnecessary. The Meeting therefore concluded that the acute dietary exposure to residues of kresoxim-methyl from the uses considered is unlikely to present a public health concern.

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| Kresoxim_044 | Wayman, L.H. | 1994 | Validation of the method of analysis for determination of residual concentration of |
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| Kresoxim 047 | Rabe, U., | 1996 | Validation of BASF Analytical Method No. 350/3: Determination of BAS 490 F |
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869

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| Kresoxim_053 | Goecer, M., Class, T. | 2011 | Validation of BASF method L0177/01: Determination of BF 490-1 (Reg.No. 262 |
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| Kresoxim_062 | Mackenroth, Ch., | 1994d | Storage stability of BAS 490 F in apple |
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| Kresoxim_089 | Oxspring, S. | 2013 | Study on the residue behaviour of Kresoxim-methyl (BAS 490 F) in strawberry after |
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| | | | Eurofins Agroscience Services, Melbourne Derbyshire DE73 8AG, United Kingdom |
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| | | | 2000/1014856 |
| | | | BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed.Rep. |
| | | | 2000/1014856 |
| | | | GLP: yes |
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