# FLUPYRADIFURONE (285)

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## EXPLANATION

Flupyradifurone (fpd) is an insecticide belonging to the chemical class of butenolides. It acts as an agonist of the nicotinic acetylcholine receptor. It was scheduled for evaluation as a new compound by the 2015 JMPR at the 46th session of the CCPR (2014). It was evaluated for toxicology in 2015. An ADI of 0–0.08 mg/kg bw and an ARfD of 0.2 mg/kg bw were established. It was evaluated for residues in 2016 at which time 52 MRLs and a large number of dietary parameters were proposed. Flupyradifurone was scheduled by the 48th session of the CCPR meeting in 2016 for the evaluation of residues data for additional crops by the JMPR.

The residue definition for compliance with the MRL for plant commodities is flupyradifurone. The residue definition for estimation of dietary intake for plant commodities is the sum of flupyradifurone, difluoroacetic acid and 6-chloronicotinic acid, expressed as parent equivalents.

The residue definition for compliance with the MRL and for estimation of dietary intake for animal commodities is the sum of flupyradifurone and difluoroacetic acid, expressed as parent equivalents.

For this submission the manufacturer supplied information on an analytical method, storage stability information, a registered use pattern for stone fruit, supervised residue trials on cherries, peaches and plums and fate of residues in processing studies for peaches and plums. A product label was made available from the United States of America.

## RESIDUE ANALYSIS

### *Analytical methods*

*Plant commodities*

The analytical method 01304 and the amendment RV-001-P10-02, for the determination of the residues of parent flupyradifurone, difluoroacetic acid (DFA), difluoroethyl-amino-furanone (DFEAF) and 6-chloronicotinic acid (6-CNA) in plant commodities by HPLC-MS/MS, using stable isotopically labelled internal standards, were described in the previous residues evaluation of flupyradifurone (JMPR 2016).

In the peach processing study (Noss and Teubner 2012, 10-3216) and the storage stability study (Netzband 2015, RARVP046-2), the residues of parent flupyradifurone, DFA, DFEAF and 6-CNA in stone fruit (cherries, peaches and plums) were determined according to analytical method RV-0001-P10-02.

In the stone fruit residue trials (Beedle and Jerkins 2015, RARVY014) and in the plum processing study (Lam and Jerkins 2015, RARVY040), residue method RV-001-P10-02 (Method 01304) was used with modifications. The updated method was reported as method RV-001-P10-03 (Li 2012, RV-001-P10-03). This was not reported in the previous evaluation and is described below.

The method description RV-001-P10-03 summarises all modifications of Method RV-001-P10-02. Flupyradifurone and its metabolites DFA, DFEAF and 6-CNA are extracted twice from plant material with acetonitrile/water (4/1, v/v) with 2.2 mL/L formic acid, followed by dilution. For an optional clean-up (analysis of parent flupyradifurone), an aliquot is evaporated to near dryness, reconstituted, and purified through a C-18 solid phase extraction (SPE) column. The column is diluted and amended with a mixture of stable, isotopically labelled internal standards. It is then analysed by HPLC-MS/MS. For the analysis of DFA, a clean-up process using a cationic resin can be used. An aliquot of the sample extract is shaken with cationic resin AG-50W-X8, acetonitrile and a mixture of

stable, isotopically labelled internal standards. After centrifugation, the supernatant is analysed by HPLC-MS/MS. Alternative chromatographic conditions are described for matrices difficult to analyse such as hay, hops, instant coffee or dried orange pomace.

The responses of the HPLC-MS/MS system to parent flupyradifurone, DFA, DFEAF and 6-CNA were linear over the range 0.002 mg/L to 2.50 mg/L (in parent equivalents) using 9 different concentration levels, for all compounds. The correlation coefficients of the 1/× weighted linear regression were > 0.99 in all cases.

The limits of quantitation (LOQ) for parent flupyradifurone, and its metabolites 6-CNA and DFEAF are generally 0.01 mg/kg (as parent equivalents) while the LOQ of DFA ranges between 0.02 and 0.05 mg/kg (as parent equivalents).

Data on the extraction efficiency of method 010304 was evaluated by the JMPR in 2016. Since the extraction procedure for Bayer Method RV-001-P10-03 is identical with the previous extraction procedure, no additional discussion is required.

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

*Plant matrices*

To determine the freezer storage stability of the residues of flupyradifurone in plant materials, individual 5-g control samples of homogenised orange fruit (high acid content), spinach leaves and tomato fruit (high water content), wheat grain (high starch content), bean seed (high protein content), coffee bean and soybean seed (high oil content) and sugar cane were separately fortified with 5.0 µg of either BYI 02960 parent compound, DFA or DFEAF (Netzband 2015, RARVP046-2). This resulted in a fortification level of 1.0 µg/g (1 mg/kg) of each analyte. Except for the day-0 analysis, samples were stored in glass containers in a freezer at an average temperature of -23 °C.

For day-0 analysis, three treated samples of each material were chosen, as well as one control sample of each. Samples were then also analysed after nominal intervals of 1, 2½, 5–6, 12, 18, 35 and 52 months. At each of these intervals, two treated samples of each material were removed from storage and analysed, as well as a control sample and two samples for concurrent recovery. Analyses of parent compound, DFA and DFEAF were carried out using the data gathering method (01304).

Note: The JMPR 2016 reported these data up to the 18 months storage time (Netzband, Timberlake and Harbin 2012, RARVP046-1). The newly submitted data are shown below in bold.

The storage stability data for parent flupyradifurone and its metabolites are summarised below in Table 1.

Table 1 Summary of stability data for deep frozen samples fortified at 1 mg/kg with Parent, DFA or DFEAF

| Matrix | Storage period (d) | Parent | | DFA | | DFEAF | |
|---|---|---|---|---|---|---|---|
| | | Concurrrent recovery (%) | Recovery in stored sample (%) | Concurrrent recovery (%) | Recovery in stored sample (%) | Concurrrent recovery (%) | Recovery in stored sample (%) |
| sugar cane | 0 | (100) | 100 | (100) | 98 | (100) | 110 |
| | 29 | 101 | 92 | 97 | 96 | 89 | 81 |
| | 77 | 91 | 85 | 97 | 92 | 93 | 88 |
| | 149 | 98 | 94 | 86 | 100 | 117 | 112 |
| | 372 | 87 | 93 | 97 | 99 | 87 | 93 |
| | 559 | 89 | 92 | 93 | 137 | 83 | 94 |
| | 1038 | 104 | 103 | 107 | 99 | 101 | 92 |
| | 1568 | 89 | 86 | 87 | 81 | 102 | 82 |
| coffee bean (green) | 0 | (100) | 94 | (100) | 90 | (100) | 101 |
| | 33 | 97 | 93 | 90 | 89 | 100 | 94 |
| | 81 | 81 | 81 | 77 | 76 | 89 | 88 |
| | 152 | 85 | 104 | 80 | 94 | 118 | 100 |
| | 370 | 93 | 83 | 73 | 79 | 90 | 86 |
| | 560 | 91 | 94 | 85 | 108 | 88 | 89 |

| Matrix | Storage period (d) | Parent | | DFA | | DFEAF | |
|---|---|---|---|---|---|---|---|
| | | Concurrrent recovery (%) | Recovery in stored sample (%) | Concurrrent recovery (%) | Recovery in stored sample (%) | Concurrrent recovery (%) | Recovery in stored sample (%) |
| | 1043 | 98 | 95 | 86 | 86 | 98 | 96 |
| | 1572 | 86 | 82 | 82 | 77 | 96 | 89 |
| orange fruit | 0 | (100) | 96 | (100) | 95 | (100) | 104 |
| | 28 | 89 | 84 | 98 | 100 | 100 | 109 |
| | 77 | 86 | 95 | 89 | 99 | 98 | 94 |
| | 148 | 104 | 84 | 87 | 93 | 119 | 110 |
| | 365 | 102 | 80 | 94 | 98 | 97 | 92 |
| | 556 | 80 | 90 | 92 | 119 | 97 | 85 |
| | 1045 | 114 | 111 | 110 | 106 | 111 | 90 |
| | 1567 | 92 | 89 | 93 | 88 | 99 | 82 |
| soybean seed | 0 | (100) | 93 | (100) | 79 | (100) | 107 |
| | 28 | 93 | 93 | 89 | 71 | 99 | 106 |
| | 75 | 93 | 96 | 93 | 82 | 103 | 94 |
| | 148 | 91 | 97 | 75 | 98 | 117 | 117 |
| | 371 | 96 | 98 | 73 | 79 | 84 | 85 |
| | 558 | 95 | 89 | 79 | 87 | 97 | 87 |
| | 1045 | 102 | 108 | 82 | 77 | 99 | 98 |
| | 1567 | 89 | 79 | 72 | 63 | 96 | 81 |
| navy bean | 0 | (100) | 111 | (100) | 96 | (100) | 102 |
| | 26 | 91 | 107 | 96 | 99 | 109 | 102 |
| | 75 | 94 | 110 | 98 | 108 | 107 | 93 |
| | 148 | 100 | 123 | 79 | 100 | 116 | 103 |
| | 364 | 113 | 127 | 80 | 78 | 93 | 88 |
| | 558 | 100 | 94 | 99 | 128 | 114 | 104 |
| | 1043 | 103 | 127 | 93 | 105 | 101 | 101 |
| | 1556 | 98 | 104 | 85 | 80 | 100 | 90 |
| tomato fruit | 0 | (100) | 95 | (100) | 101 | (100) | 110 |
| | 28 | 90 | 92 | 99 | 96 | 96 | 116 |
| | 76 | 86 | 89 | 94 | 94 | 103 | 100 |
| | 148 | 107 | 105 | 84 | 98 | 118 | 101 |
| | 370 | 89 | 95 | 98 | 105 | 97 | 84 |
| | 558 | 102 | 94 | 102 | 134 | 111 | 89 |
| | 1047 | 102 | 101 | 108 | 103 | 103 | 88 |
| | 1566 | 88 | 88 | 94 | 90 | 101 | 79 |
| spinach | 0 | (100) | 91 | (100) | 100 | (100) | 106 |
| | 26 | 96 | 97 | 98 | 97 | 107 | 109 |
| | 75 | 90 | 85 | 90 | 87 | 101 | 96 |
| | 147 | 94 | 104 | 85 | 100 | 114 | 109 |
| | 364 | 119 | 97 | 70 | 74 | 105 | 95 |
| | 557 | 98 | 100 | 107 | 147 | 102 | 95 |
| | 1046 | 102 | 104 | 104 | 99 | 102 | 95 |
| | 1566 | 84 | 83 | 97 | 93 | 93 | 78 |
| wheat grain | 0 | (100) | 94 | (100) | 92 | (100) | 103 |
| | 27 | 93 | 90 | 97 | 98 | 101 | 106 |
| | 76 | 89 | 82 | 88 | 94 | 96 | 91 |
| | 186 | 97 | 109 | 78 | 98 | 104 | 99 |
| | 362 | 92 | 77 | 72 | 71 | 92 | 90 |
| | 557 | 97 | 91 | 88 | 116 | 100 | 87 |
| | 1046 | 96 | 81 | 85 | 77 | 97 | 86 |
| | 1566 | 85 | 83 | 81 | 74 | 89 | 76 |

At day 0, average residue recoveries of parent flupyradifurone ranged from 91–111% of nominal, of DFEAF from 101–110% of nominal and of DFA from 79–101% of nominal. In samples analysed after approximately 52 months of frozen storage, storage stability recoveries, ranged from 79–104% for flupyradifurone, 76–90% for DFEAF and 63–93% for DFA. Storage stability after approximately 52 months of frozen storage adjusted for concurrent recoveries, were 89–106% for flupyradifurone, 78–93% for DFEAF and 88–96% for DFA.

At all sampling dates and in all sample materials (sugarcane, green coffee bean, orange fruit, soybean seed, navy bean, tomato fruit, spinach and wheat grain), residues of flupyradifurone, DFEAF and DFA were above 70% (except one sample of DFA, soybean seed at 1567 days). Even in the case of the lower values in the given ranges, there was no evidence of any continued degradation of any of the analytes in any of the sample materials. Thus, all analytes can be considered stable in all relevant plant matrix types for a period of at least 52 months (1556 to 1572 days).

## USE PATTERN

Information on registered uses made available to this Meeting is shown in Table 2.

Table 2 Registered use of flupyradifurone on stone fruits

| Crop | Country | Formulation | | Application | | | | PHI [days] |
|---|---|---|---|---|---|---|---|---|
| | | g ai/L or [g ai/kg] | Type | Method | Timing [Interval – days] | Rate [g ai/ ha] | Season Max. [g ai/ ha/year] or (no. per crop) | |
| Stone Fruit | | | | | | | | |
| Crop Group 12-12 | USA | 200 | SL | Foliar | 10 | 205 | [410] (2) | 14 |

Crops of Crop Group 12-12 Including:

Apricot (including Japanese apricot), Capulin, Cherry (including Black cherry, Nanking cherry, Sweet cherry, and Tart cherry), Chinese Jujube, Nectarine, Peach, Plum (including American plum, Beach plum, Canada plum, Cherry plum, Chickasaw plum, Damson plum, Japanese plum, Kiamath plum, and Prune plum), Plumcot, Sloe, and cultivars, varieties, and/or hybrids of these commodities

## RESULTS OF SUPERVISED RESIDUE TRIALS ON CROPS

The Meeting received information on supervised trials for the uses of flupyradifurone on stone fruits (cherry, peach and plum).

Trials were well documented with laboratory and field reports. The former included method validation including recoveries with spiking at residue levels similar to those occurring in samples from the supervised trials. Dates of analyses or duration of sample storage were also provided. Samples were collected and stored frozen immediately or soon after sampling. Although trials included control plots, no control data are recorded in the Tables because, residues in control samples did not exceed the LOQ. Residues are unadjusted for recoveries.

Residues from the trials conducted according to maximum GAP have been used for the estimation of maximum residue levels and dietary risk assessment and are underlined. If a higher residue level was observed at a longer PHI than the GAP, the higher value has been used in MRL setting and dietary risk assessment.

Where parent or DFA residues were not detected or were less than the LOQ (*i.e.* generally < 0.01 mg/kg for parent or 0.05 mg/kg for DFA) the LOQ value was utilised for maximum residue estimation and dietary intake assessment. For 6-CNA, values less than the LOQ were not added for calculation of total residues of flupyradifurone.

The following table shows how residues in the trials were added to give total residues of flupyradifurone.

| Parent | DFA | 6-CNA | Total |
|---|---|---|---|
| < 0.01 | 0.05 | 0.01 | 0.07 |
| 0.01 | < 0.05 | 0.01 | 0.07 |
| < 0.01 | < 0.05 | < 0.01 | < 0.06 |
| 0.01 | 0.05 | < 0.01 | 0.06 |
| 0.01 | 0.05 | 0.01 | 0.07 |

For multiple trials on a crop from the same location, the result from the trial yielding the highest residue was utilised for maximum residue level estimation and dietary intake assessment. In this case the trials are separated with a dotted line. Residues were added as unrounded values.

| Group | Commodity | Country/ Countries | Table |
|---|---|---|---|
| FS Stone Fruits | Cherry | USA and Canada | 3 |
| | Peach | USA | 4 |
| | Plum | USA and Canada | 5 |

The results of these supervised trials are shown in the following tables:

*Stone fruits*

Supervised trials were carried out on sour and sweet cherries (five trials on sour cherries, four decline and one harvest; four on sweet cherries, three decline and one harvest – Table 3), peaches (12 trials, seven decline and five harvest – Table 4) and plums (eight trials, seven decline and one harvest – Table 5) in the USA and, for cherries and plums only, Canada, during the 2012 and 2014 growing seasons (Beedle and Jerkins 2015, RARVY014) to determine residues in stone fruit following either two concentrated airblast applications (TRTDC) or two dilute airblast applications (TRTDD) of a flupyradifurone 200 SL (soluble concentrate) formulation. Applications were made to plots using ground-based equipment.

Across all trials individual application rates ranged from 0.19 to 0.22 kg ai/ha and total seasonal application rates ranged from 0.40 to 0.42 kg ai/ha. The first application ranged between BBCH growth stages 72 and 81 (stone fruit BBCH 72: green ovary surrounded by dying sepal crown, sepals beginning to fall; BBCH 81: beginning of fruit colouring). The interval between applications was 8 to 11 days, except for one trial that had a 14 day interval between applications. The spray volumes ranged for the TRTDC plots ranged from 190–484 litres per hectare and for the TRTDD plots 1879–2878 litres per hectare.

Adjuvant was added in all applications; either Non-Ionic Surfactant (NIS) at 0.25% v/v (RV003-12DA, RV006-12DA, RV009-12DC, RV012-12DA, RV015-12DA, RV018-12HC, RV021-12DA, RV027-12DA, RV030-12DA and RV033-12DA); or Methylated Seed Oil (MSO) at 0.25% v/v (RV004-12DA, RV007-12HA, RV010-12HA, RV013-12DB, RV016-12DA, RV019-12DA, RV022-12HA, RV025-12DA, RV028-12DC and RV034-12DA; or Crop Oil Concentrate (COC) at 1.0% v/v (RV008-12DA, RV011-12DA, RV014-12HB, RV017-12HA, RV020-12DA, RV023-12HA, RV026-12DA, RV029-12HA and RV032-12DA).

Potental residue reduction was investigated in two sweet cherry trials (RV003-12DA and RV007-12HA) and three peach trials (RV014-12HB, RV018-12HC and RV022-12HA).

Residues of flupyradifurone, difluoroacetic acid (DFA), difluoroethylaminofuranone (DFEAF) and 6-chloronicotinic acid (6-CNA) in cherries, peaches and plums were determined by LC-MS/MS method 01304 using stable isotopically labelled internal standards. Acceptable method and concurrent recovery data were obtained for all analytes in all fruit (including washed, cooked and peeled).

Table 3 Residues from the foliar application of flupyradifurone to cherries in the USA and Canada (Beedle and Jerkins 2015, RARVY014)

| Trial No., Location, Year (Type-Variety) | Application | | | | Sample | DALA | Residues as parent (mg/kg) | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | No. (RTI, days) | Growth Stage | Rate (g ai/ha) | Volume (L/ha) | | | Parent | DFA | DFEAF | 6-CNA | Parent + DFA + 6-CNA |
| GAP, USA, Stone fruit Foliar | 2 (10) | | 205 | | | 14 | | | | | |
| RV003-12DA-TRTDC Hart, | 2 (10) | 77 | 205 | 458 | Fruit (RAC) | 0 | 0.64 | 0.089 | 0.010 | 0.019 | 0.74 |
| | | 78 | 204 | 418 | | 7 | 0.73 | 0.083 | 0.013 | 0.022 | 0.83 |
| | | | | | | 13 | 0.25 | 0.13 | < 0.01 | 0.013 | 0.39 |

| Trial No., Location, Year (Type-Variety) | Application No. (RTI, days) | Growth Stage | Rate (g ai/ha) | Volume (L/ha) | Sample | DALA | Residues as parent (mg/kg) Parent | DFA | DFEAF | 6-CNA | Parent + DFA + 6-CNA |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Michigan, USA, 2012 (Sweet-Baby Gold) | | | | | | 13 | 0.27 | 0.21 | 0.011 | 0.022 | 0.50 |
| | | | | | | | 0.26 | 0.22 | < 0.01 | 0.019 | 0.50 |
| | | | | | | | 0.25 | 0.23 | < 0.01 | 0.018 | 0.51 |
| | | | | | | Mean | 0.26 | 0.22 | < 0.01 | 0.020 | 0.50 |
| | | | | | | 21 | 0.11 | 0.072 | < 0.01 | < 0.01 | 0.18 |
| | | | | | | 28 | 0.27 | 0.14 | < 0.01 | 0.015 | 0.42 |
| | | | | | | 35 | 0.26 | 0.18 | < 0.01 | 0.020 | 0.46 |
| | | | | | Fruit, washed | 13 | 0.27 | 0.24 | < 0.01 | 0.023 | 0.53 |
| | | | | | | | 0.24 | 0.23 | < 0.01 | 0.020 | 0.48 |
| | | | | | | | 0.24 | 0.22 | < 0.01 | 0.021 | 0.48 |
| | | | | | | mean | 0.25 | 0.23 | < 0.01 | 0.021 | 0.49 |
| | | | | | Fruit, cooked | 13 | 0.066 | < 0.05 | < 0.01 | < 0.01 | 0.12 |
| | | | | | | | 0.071 | 0.052 | < 0.01 | 0.010 | 0.12 |
| | | | | | | | 0.065 | 0.054 | < 0.01 | < 0.01 | 0.12 |
| | | | | | | mean | 0.067 | 0.052 | < 0.01 | < 0.01 | 0.12 |
| RV003-12DA-TRTDD Hart, Michigan, USA, 2012 (Sweet-Baby Gold) | 2 (10) | 77 78 | 205 207 | 2109 1879 | Fruit | 0 | 0.36 | < 0.05 | < 0.01 | 0.010 | 0.42 |
| | | | | | | 7 | 0.61 | 0.066 | 0.021 | 0.026 | 0.70 |
| | | | | | | 13 | 0.33 | 0.14 | 0.013 | 0.023 | 0.49 |
| | | | | | | 21 | 0.25 | 0.15 | 0.012 | 0.017 | 0.41 |
| | | | | | | 28 | 0.36 | 0.18 | 0.014 | 0.023 | 0.57 |
| | | | | | | 35 | 0.14 | 0.092 | < 0.01 | 0.011 | 0.25 |
| RV004-12DA,-TRTDC Perry, Utah USA, 2012 (Sweet-Bing) | 2 (10) | 79 85 | 203 204 | 478 362 | Fruit | 0 | 0.72 | < 0.05 | < 0.01 | < 0.01 | 0.77 |
| | | | | | | 5 | 0.64 | 0.057 | < 0.01 | < 0.01 | 0.69 |
| | | | | | | 13 | 0.45 | 0.11 | < 0.01 | 0.014 | 0.57 |
| | | | | | | 19 | 0.42 | 0.17 | < 0.01 | 0.017 | 0.61 |
| | | | | | | 26 | 0.37 | 0.25 | < 0.01 | 0.016 | 0.64 |
| | | | | | | 33 | 0.31 | 0.36 | < 0.01 | 0.019 | 0.69 |
| RV004-12DA,-TRTDD Perry, Utah USA, 2012 (Sweet-Bing) | 2 (10) | 79 85 | 204 207 | 2151 2206 | Fruit | 0 | 1.3 | < 0.05 | 0.010 | < 0.01 | 1.4 |
| | | | | | | 5 | 0.90 | 0.081 | 0.015 | 0.013 | 1.0 |
| | | | | | | 13 | 0.62 | 0.14 | < 0.01 | 0.014 | 0.77 |
| | | | | | | 19 | 0.55 | 0.25 | < 0.01 | 0.016 | 0.82 |
| | | | | | | 26 | 0.39 | 0.31 | < 0.01 | 0.015 | 0.71 |
| | | | | | | 33 | 0.29 | 0.35 | < 0.01 | 0.013 | 0.65 |
| RV006-12DA-TRTDC Ephrata, Washington USA, 2012 (Sweet-Bing) | 2 (9) | 75 77 | 203 204 | 420 416 | Fruit | 0 | 1.1 | < 0.05 | 0.010 | < 0.01 | 1.1 |
| | | | | | | 7 | 0.94 | < 0.05 | 0.021 | 0.023 | 1.0 |
| | | | | | | 14 | 0.61 | 0.15 | 0.012 | 0.023 | 0.79 |
| | | | | | | 21 | 0.49 | 0.23 | < 0.01 | 0.025 | 0.74 |
| | | | | | | 28 | 0.50 | 0.31 | < 0.01 | 0.024 | 0.84 |
| | | | | | | 35 | 0.25 | 0.42 | < 0.01 | 0.022 | 0.69 |
| RV006-12DA-TRTDD Ephrata, Washington USA, 2012 (Sweet-Bing) | 2 (9) | 75 77 | 209 209 | 2330 2326 | Fruit | 0 | 0.91 | < 0.05 | < 0.01 | < 0.01 | 0.96 |
| | | | | | | 7 | 0.82 | 0.13 | 0.016 | 0.020 | 0.97 |
| | | | | | | 14 | 0.46 | 0.17 | < 0.01 | 0.020 | 0.65 |
| | | | | | | 21 | 0.94 | 0.13 | 0.021 | 0.023 | 1.1 |
| | | | | | | 28 | 0.39 | 0.32 | < 0.01 | 0.023 | 0.73 |
| | | | | | | 35 | 0.30 | 0.35 | < 0.01 | 0.027 | 0.68 |
| RV007-12HA-TRTDC Ephrata, Washington, USA, 2012 (Sweet-Skeena) | 2 (10) | 75 78 | 204 204 | 422 419 | Fruit | 14 | 0.42 | 0.17 | < 0.01 | 0.019 | 0.61 |
| | | | | | Fruit RAC | 14 | 0.38 | 0.22 | < 0.01 | 0.023 | 0.63 |
| | | | | | | | 0.41 | 0.24 | < 0.01 | 0.024 | 0.68 |
| | | | | | | | 0.40 | 0.24 | < 0.01 | 0.021 | 0.66 |
| | | | | | | mean | 0.40 | 0.23 | < 0.01 | 0.022 | 0.65 |
| | | | | | Fruit washed | 14 | 0.40 | 0.23 | < 0.01 | 0.030 | 0.65 |
| | | | | | | | 0.40 | 0.22 | < 0.01 | 0.031 | 0.65 |
| | | | | | | | 0.36 | 0.20 | < 0.01 | 0.022 | 0.58 |
| | | | | | | mean | 0.38 | 0.22 | < 0.01 | 0.027 | 0.63 |
| | | | | | Fruit, cooked | 14 | 0.19 | 0.090 | < 0.01 | 0.014 | 0.29 |
| | | | | | | | 0.19 | 0.086 | < 0.01 | 0.012 | 0.29 |
| | | | | | | | 0.22 | 0.094 | < 0.01 | 0.011 | 0.32 |
| | | | | | | mean | 0.20 | 0.090 | < 0.01 | 0.012 | 0.30 |
| RV007-12HA- | 2 | 75 | 205 | 2338 | Fruit | 14 | 0.30 | 0.15 | < 0.01 | 0.016 | 0.47 |

| Trial No., Location, Year (Type-Variety) | Application | | | | Sample | DALA | Residues as parent (mg/kg) | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | No. (RTI, days) | Growth Stage | Rate (g ai/ha) | Volume (L/ha) | | | Parent | DFA | DFEAF | 6-CNA | Parent + DFA + 6-CNA |
| TRTDD Ephrata, Washington, USA, 2012 (Sweet-Skeena) | (10) | 78 | 204 | 2320 | | | | | | | |
| RV008-12DA-TRTDC Alton, New York, USA, 2012 (Sour-Montmorency) | 2 (14) | 76 85 | 208 205 | 483 471 | Fruit | 0 | 0.69 | < 0.05 | 0.022 | 0.020 | 0.76 |
| | | | | | | 6 | 0.94 | 0.13 | 0.021 | 0.033 | 1.1 |
| | | | | | | 14 | 0.26 | 0.10 | 0.029 | 0.047 | 0.41 |
| | | | | | | 21 | 0.18 | 0.14 | 0.030 | 0.067 | 0.38 |
| | | | | | | 28 | 0.078 | 0.17 | 0.029 | 0.075 | 0.33 |
| | | | | | | 35 | 0.034 | 0.19 | 0.020 | 0.094 | 0.32 |
| RV008-12DA-TRTDD Alton, New York, USA, 2012 (Sour-Montmorency) | 2 (14) | 76 85 | 206 205 | 1882 1879 | Fruit | 0 | 0.61 | < 0.05 | 0.023 | 0.024 | 0.68 |
| | | | | | | 6 | 0.56 | 0.088 | 0.029 | 0.021 | 0.67 |
| | | | | | | 14 | 0.30 | 0.15 | 0.042 | 0.048 | 0.49 |
| | | | | | | 21 | 0.24 | 0.16 | 0.039 | 0.059 | 0.45 |
| | | | | | | 28 | 0.064 | 0.17 | 0.023 | 0.052 | 0.28 |
| | | | | | | 35 | 0.36 | 0.060 | 0.027 | 0.033 | 0.45 |
| RV009-12DC-TRTDC Branchton, Ontario, Canada, 2014 (Sour-North Star) | 2 (9) | 72 75 | 204 207 | 199 201 | Fruit | 0 | 3.5 | 0.13 | 0.040 | 0.022 | 3.6 |
| | | | | | | 7 | 1.7 | 0.24 | 0.077 | 0.035 | 2.0 |
| | | | | | | 14 | 0.18 | 0.25 | 0.030 | 0.022 | 0.45 |
| | | | | | | 20 | 0.051 | 0.36 | 0.013 | 0.022 | 0.44 |
| | | | | | | 27 | 0.012 | 0.39 | < 0.01 | 0.016 | 0.42 |
| | | | | | | 34 | < 0.01 | 0.51 | < 0.01 | 0.017 | 0.53 |
| RV009-12DC-TRTDD Branchton, Ontario, Canada, 2014 (Sour-North Star) | 2 (9) | 72 75 | 204 211 | 2594 2678 | Fruit | 0 | 3.1 | 0.12 | 0.048 | 0.024 | 3.3 |
| | | | | | | 7 | 1.8 | 0.26 | 0.066 | 0.045 | 2.1 |
| | | | | | | 14 | 0.25 | 0.26 | 0.039 | 0.026 | 0.54 |
| | | | | | | 20 | 0.068 | 0.31 | 0.017 | 0.023 | 0.40 |
| | | | | | | 27 | 0.019 | 0.35 | < 0.01 | 0.021 | 0.39 |
| | | | | | | 34 | 0.012 | 0.41 | < 0.01 | 0.014 | 0.44 |
| RV010-12HA-TRTDC Conklin, Michigan, USA, 2012 (Sour-Montmorency) | 2 (10) | 81 87 | 203 204 | 389 406 | Fruit | 14 | 0.17 | 0.26 | 0.032 | 0.078 | 0.50 |
| RV010-12HA-TRTDD Conklin, Michigan, USA, 2012 (Sour-Montmorency) | 2 (10) | 81 87 | 205 207 | 2272 2368 | Fruit | 14 | 0.051 | 0.29 | 0.030 | 0.068 | 0.41 |
| RV011-12DA-TRTDC Perry, Utah, USA, 2012 (Sour-Montmorency) | 2 (10) | 81 85 | 205 203 | 327 322 | Fruit | 0 | 1.6 | 0.078 | 0.034 | 0.051 | 1.8 |
| | | | | | | 7 | 0.69 | 0.14 | 0.062 | 0.11 | 0.94 |
| | | | | | | 14 | 0.36 | 0.27 | 0.040 | 0.19 | 0.82 |
| | | | | | | 21 | 0.10 | 0.38 | 0.018 | 0.19 | 0.68 |
| | | | | | | 28 | 0.038 | 0.43 | < 0.01 | 0.14 | 0.60 |
| | | | | | | 35 | 0.021 | 0.46 | < 0.01 | 0.13 | 0.61 |
| RV011-12DA-TRTDD Perry, Utah, USA, 2012 (Sour-Montmorency) | 2 (10) | 81 85 | 206 203 | 1981 1955 | Fruit | 0 | 2.3 | 0.071 | 0.041 | 0.056 | 2.4 |
| | | | | | | 7 | 1.1 | 0.13 | 0.075 | 0.11 | 1.4 |
| | | | | | | 14 | 0.58 | 0.28 | 0.050 | 0.20 | 1.1 |
| | | | | | | 21 | 0.15 | 0.40 | 0.025 | 0.19 | 0.74 |
| | | | | | | 28 | 0.12 | 0.57 | 0.027 | 0.19 | 0.88 |
| | | | | | | 35 | 0.032 | 0.55 | 0.012 | 0.15 | 0.74 |
| RV012-12DA-TRTDC Saskatoon, Saskatchewan, Canada, 2012 (Sour-Valentine) | 2 (8) | 81 85 | 215 200 | 368 377 | Fruit | 0 | 0.34 | 0.089 | 0.023 | 0.022 | 0.45 |
| | | | | | | 6 | 0.044 | 0.13 | 0.028 | 0.036 | 0.21 |
| | | | | | | 12 | 0.014 | 0.19 | 0.014 | 0.028 | 0.24 |
| | | | | | | 21 | < 0.01 | 0.23 | < 0.01 | 0.023 | 0.26 |
| | | | | | | 27 | < 0.01 | 0.19 | < 0.01 | 0.027 | 0.23 |
| | | | | | | 35 | < 0.01 | 0.24 | < 0.01 | 0.018 | 0.27 |

| Trial No., Location, Year (Type-Variety) | Application | | | | Sample | DALA | Residues as parent (mg/kg) | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | No. (RTI, days) | Growth Stage | Rate (g ai/ha) | Volume (L/ha) | | | Parent | DFA | DFEAF | 6-CNA | Parent + DFA + 6-CNA |
| RV012-12DA-TRTDD Saskatoon, Saskatchewan, Canada, 2012 (Sour-Valentine) | 2 (8) | 81 85 | 206 205 | 2257 2303 | Fruit | 0 | 0.30 | 0.082 | 0.028 | 0.022 | 0.41 |
| | | | | | | 6 | 0.087 | 0.21 | 0.034 | 0.045 | 0.34 |
| | | | | | | 12 | 0.011 | 0.21 | 0.012 | 0.019 | 0.24 |
| | | | | | | 21 | < 0.01 | 0.36 | < 0.01 | 0.024 | 0.39 |
| | | | | | | 27 | < 0.01 | 0.31 | < 0.01 | 0.018 | 0.33 |
| | | | | | | 35 | < 0.01 | 0.37 | < 0.01 | 0.021 | 0.40 |

LOQ is 0.01 mg/kg for each of parent flupyradifurone and the metabolites DFEAF and 6-CNA and 0.05 mg/kg for DFA (parent equivs.)

Table 4 Residues from the foliar application of flupyradifurone to peaches in the USA (Beedle and Jerkins 2015, RARVY014)

| Trial No., Location, Year (Variety) | Application | | | | Sample | DALA | Residues as parent (mg/kg) | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | No. (RTI, days) | Growth Stage | Rate (g ai/ha) | Volume (L/ha) | | | Parent | DFA | DFEAF | 6-CNA | Parent + DFA + CNA |
| GAP, USA, Stone fruit Foliar | 2 (10) | | 205 | | | 14 | | | | | |
| RV013-12DB-TRTDC Orefield, Pennsylvania, USA, 2012 (Glen Glow) | 2 (8) | 79 79 | 200 204 | 422 431 | Fruit | 0 | 0.62 | < 0.025 | < 0.005 | < 0.005 | 0.64 |
| | | | | | | 7 | 0.31 | 0.038 | < 0.005 | < 0.005 | 0.34 |
| | | | | | | 14 | 0.33 | 0.10 | < 0.005 | < 0.005 | 0.43 |
| | | | | | | 20 | 0.21 | 0.13 | < 0.005 | < 0.005 | 0.34 |
| | | | | | | 27 | 0.18 | 0.16 | < 0.005 | < 0.005 | 0.34 |
| | | | | | | 30 | 0.084 | 0.13 | < 0.005 | < 0.005 | 0.21 |
| RV013-12DB-TRTDD Orefield, Pennsylvania, USA, 2012 (Glen Glow) | 2 (8) | 79 79 | 210 207 | 2509 2478 | Fruit | 0 | 0.62 | < 0.025 | < 0.005 | < 0.005 | 0.65 |
| | | | | | | 7 | 0.27 | 0.044 | < 0.005 | 0.0061 | 0.32 |
| | | | | | | 14 | 0.27 | 0.11 | < 0.005 | < 0.005 | 0.37 |
| | | | | | | 20 | 0.23 | 0.15 | < 0.005 | < 0.005 | 0.38 |
| | | | | | | 27 | 0.15 | 0.15 | < 0.005 | < 0.005 | 0.29 |
| | | | | | | 30 | 0.091 | 0.17 | < 0.005 | < 0.005 | 0.26 |
| RV014-12HB,-TRTDC Byron, Georgia USA, 2012 (Sun-Prince) | 2 (9) | 81 87 | 206 206 | 406 414 | Fruit | 13 | 0.22 | 0.12 | < 0.005 | < 0.005 | 0.34 |
| | | | | | Fruit, RAC | 13 | 0.27 | 0.14 | < 0.005 | < 0.005 | 0.41 |
| | | | | | | | 0.24 | 0.15 | < 0.005 | < 0.005 | 0.38 |
| | | | | | | | 0.26 | 0.14 | < 0.005 | < 0.005 | 0.41 |
| | | | | | | mean | 0.26 | 0.14 | < 0.005 | < 0.005 | 0.40 |
| | | | | | Whole fruit, washed | 13 | 0.17 | 0.13 | 0.005 | 0.0051 | 0.31 |
| | | | | | | | 0.19 | 0.12 | < 0.005 | < 0.005 | 0.32 |
| | | | | | | | 0.21 | 0.13 | < 0.005 | < 0.005 | 0.34 |
| | | | | | | mean | 0.19 | 0.13 | < 0.005 | < 0.005 | 0.32 |
| | | | | | Fruit, cooked | 13 | 0.076 | 0.073 | < 0.005 | 0.0061 | 0.15 |
| | | | | | | | 0.078 | 0.067 | < 0.005 | < 0.005 | 0.14 |
| | | | | | | | 0.077 | 0.069 | < 0.005 | < 0.005 | 0.15 |
| | | | | | | mean | 0.077 | 0.069 | < 0.005 | < 0.005 | 0.15 |
| | | | | | Fruit, peeled | 13 | 0.086 | 0.14 | < 0.005 | < 0.005 | 0.23 |
| | | | | | | | 0.085 | 0.15 | < 0.005 | < 0.005 | 0.23 |
| | | | | | | | 0.095 | 0.16 | < 0.005 | < 0.005 | 0.26 |
| | | | | | | mean | 0.089 | 0.15 | < 0.005 | < 0.005 | 0.24 |
| RV014-12HB,-TRTDD Byron, Georgia USA, 2012 (Sun-Prince) | 2 (9) | 81 87 | 206 204 | 2143 2179 | Fruit | 13 | 0.73 | 0.38 | 0.0078 | 0.0051 | 1.1 |
| RV015-12DA-TRTDC Cana, Virginia USA, 2012 | 2 (8) | 78 85 | 204 211 | 347 364 | Fruit | 0 | 0.60 | 0.039 | 0.0068 | < 0.005 | 0.64 |
| | | | | | | 7 | 1.1 | 0.12 | 0.024 | 0.011 | 1.2 |
| | | | | | | 14 | 0.39 | 0.34 | 0.0093 | 0.0087 | 0.74 |
| | | | | | | 21 | 0.20 | 0.45 | < 0.005 | < 0.005 | 0.66 |
| | | | | | | 27 | 0.19 | 0.54 | < 0.005 | < 0.005 | 0.73 |

| Trial No., Location, Year (Variety) | Application | | | | Sample | DALA | Residues as parent (mg/kg) | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | No. (RTI, days) | Growth Stage | Rate (g ai/ha) | Volume (L/ha) | | | Parent | DFA | DFEAF | 6-CNA | Parent + DFA + CNA |
| (Autumn Prince) | | | | | | 35 | 0.084 | 0.45 | < 0.005 | < 0.005 | 0.53 |
| RV015-12DA-TRTDD Cana, Virginia USA, 2012 (Autumn Prince) | 2 (8) | 78 85 | 207 203 | 2022 2163 | Fruit | 0 | 1.4 | 0.036 | 0.016 | < 0.005 | 1.4 |
| | | | | | | 7 | 0.58 | 0.17 | 0.013 | 0.013 | 0.76 |
| | | | | | | 14 | 0.72 | 0.22 | 0.015 | < 0.005 | 0.94 |
| | | | | | | 21 | 0.26 | 0.22 | < 0.005 | < 0.005 | 0.48 |
| | | | | | | 27 | 0.33 | 0.28 | < 0.005 | < 0.005 | 0.61 |
| | | | | | | 35 | 0.23 | 0.33 | < 0.005 | < 0.005 | 0.56 |
| RV016-12DA-TRTDC Plains, Georgia USA, 2012 (Redskin) | 2 (8) | 75 NA | 202 199 | 403 408 | Fruit | 0 | 0.27 | < 0.025 | < 0.005 | < 0.005 | 0.30 |
| | | | | | | 7 | 0.27 | 0.079 | < 0.005 | < 0.005 | 0.35 |
| | | | | | | 14 | 0.12 | 0.13 | < 0.005 | < 0.005 | 0.25 |
| | | | | | | 21 | 0.034 | 0.058 | < 0.005 | < 0.005 | 0.092 |
| | | | | | | 28 | 0.074 | 0.23 | < 0.005 | < 0.005 | 0.30 |
| | | | | | | 35 | 0.038 | 0.24 | < 0.005 | < 0.005 | 0.28 |
| RV016-12DA-TRTDD Plains, Georgia USA, 2012 (Redskin) | 2 (8) | 75 NA | 205 202 | 2504 2024 | Fruit | 0 | 0.28 | < 0.025 | < 0.005 | < 0.005 | 0.30 |
| | | | | | | 7 | 0.25 | 0.096 | < 0.005 | < 0.005 | 0.35 |
| | | | | | | 14 | 0.14 | 0.10 | < 0.005 | < 0.005 | 0.24 |
| | | | | | | 21 | 0.070 | 0.14 | < 0.005 | < 0.005 | 0.21 |
| | | | | | | 28 | 0.12 | 0.23 | < 0.005 | < 0.005 | 0.35 |
| | | | | | | 35 | 0.092 | 0.21 | < 0.005 | < 0.005 | 0.30 |
| RV017-12HA-TRTDC Shelby, Michigan USA, 2012 (Baby Gold) | 2 (10) | 79 79 | 205 206 | 454 401 | Fruit | 15 | 0.13 | < 0.025 | < 0.005 | < 0.005 | 0.16 |
| RV017-12HA-TRTDD Shelby, Michigan USA, 2012 (Baby Gold) | 2 (10) | 78 79 | 205 206 | 2108 2032 | Fruit | 15 | 0.13 | < 0.025 | < 0.005 | < 0.005 | 0.15 |
| RV018-12HC-TRTDC Ringwood, Oklahoma, USA, 2014 (John Boy) | 2 (11) | 79 85 | 207 202 | 385 376 | Fruit | 13 | 0.14 | 0.10 | < 0.005 | < 0.005 | 0.24 |
| | | | | | Fruit RAC | 13 | 0.096 | 0.079 | < 0.005 | < 0.005 | 0.17 |
| | | | | | | | 0.089 | 0.074 | < 0.005 | < 0.005 | 0.16 |
| | | | | | | | 0.090 | 0.075 | < 0.005 | < 0.005 | 0.16 |
| | | | | | | mean | 0.090 | 0.076 | < 0.005 | < 0.005 | 0.17 |
| | | | | | Fruit washed | 13 | 0.081 | 0.067 | < 0.005 | < 0.005 | 0.15 |
| | | | | | | | 0.087 | 0.070 | < 0.005 | < 0.005 | 0.16 |
| | | | | | | | 0.080 | 0.067 | < 0.005 | < 0.005 | 0.15 |
| | | | | | | mean | 0.083 | 0.068 | < 0.005 | < 0.005 | 0.15 |
| | | | | | Fruit, cooked | 13 | 0.030 | 0.035 | < 0.005 | < 0.005 | 0.066 |
| | | | | | | | 0.026 | 0.031 | < 0.005 | < 0.005 | 0.057 |
| | | | | | | | 0.026 | 0.032 | < 0.005 | < 0.005 | 0.058 |
| | | | | | | mean | 0.028 | 0.033 | < 0.005 | < 0.005 | 0.060 |
| | | | | | Fruit, peeled | 13 | 0.069 | 0.079 | < 0.005 | < 0.005 | 0.15 |
| | | | | | | | 0.063 | 0.067 | < 0.005 | < 0.005 | 0.13 |
| | | | | | | | < 0.005 | 0.078 | < 0.005 | < 0.005 | 0.083 |
| | | | | | | mean | 0.046 | 0.075 | < 0.005 | < 0.005 | 0.12 |
| RV018-12HC-TRTDD Ringwood, Oklahoma, USA, 2014 (John Boy) | 2 (11) | 79 85 | 206 213 | 2189 2582 | Fruit | 13 | 0.28 | 0.11 | < 0.005 | < 0.005 | 0.39 |
| RV019-12DA-TRTDC Hart, Michigan, USA, 2012 (PF7A) | 2 (9) | 75 78 | 205 205 | 418 401 | Fruit | 0 | 0.62 | 0.045 | < 0.005 | < 0.005 | 0.67 |
| | | | | | | 7 | 0.32 | 0.087 | < 0.005 | < 0.005 | 0.41 |
| | | | | | | 14 | 0.18 | 0.15 | < 0.005 | < 0.005 | 0.33 |
| | | | | | | 21 | 0.085 | 0.13 | < 0.005 | < 0.005 | 0.22 |
| | | | | | | 28 | 0.056 | 0.12 | < 0.005 | < 0.005 | 0.18 |
| RV019-12DA- | 2 | 75 | 205 | 2424 | Fruit | 0 | 0.45 | 0.031 | < 0.005 | < 0.005 | 0.48 |

| Trial No., Location, Year (Variety) | Application No. (RTI, days) | Growth Stage | Rate (g ai/ha) | Volume (L/ha) | Sample | DALA | Residues as parent (mg/kg) Parent | DFA | DFEAF | 6-CNA | Parent + DFA + CNA |
|---|---|---|---|---|---|---|---|---|---|---|---|
| TRTDD Hart, Michigan, USA, 2012 (PF7A) | (9) | 78 | 205 | 2323 | | 7 | 0.52 | 0.098 | 0.0052 | 0.0062 | 0.61 |
| | | | | | | 14 | 0.20 | 0.12 | < 0.005 | < 0.005 | 0.32 |
| | | | | | | 21 | 0.15 | 0.14 | < 0.005 | < 0.005 | 0.29 |
| | | | | | | 28 | 0.10 | 0.14 | < 0.005 | < 0.005 | 0.24 |
| | | | | | | 35 | 0.12 | 0.18 | < 0.005 | 0.0052 | 0.30 |
| RV020-12DA-TRTDC Hart, Michigan, USA, 2012 (Red Haven) | 2 (10) | 76 78 | 205 207 | 407 398 | Fruit | 0 | 0.66 | < 0.025 | < 0.005 | < 0.005 | 0.69 |
| | | | | | | 7 | 0.48 | 0.049 | 0.0062 | < 0.005 | 0.53 |
| | | | | | | 14 | 0.19 | 0.079 | < 0.005 | < 0.005 | 0.27 |
| | | | | | | 21 | 0.077 | 0.11 | < 0.005 | < 0.005 | 0.18 |
| | | | | | | 28 | 0.051 | 0.13 | < 0.005 | < 0.005 | 0.18 |
| | | | | | | 35 | 0.037 | 0.14 | < 0.005 | < 0.005 | 0.17 |
| RV020-12DA-TRTDD Hart, Michigan, USA, 2012 (Red Haven) | 2 (10) | 76 78 | 206 206 | 2367 2309 | Fruit | 0 | 0.74 | < 0.025 | 0.0088 | < 0.005 | 0.76 |
| | | | | | | 7 | 0.23 | 0.031 | 0.0050 | < 0.005 | 0.26 |
| | | | | | | 14 | 0.25 | 0.083 | 0.0055 | < 0.005 | 0.34 |
| | | | | | | 21 | 0.17 | 0.091 | < 0.005 | < 0.005 | 0.26 |
| | | | | | | 28 | 0.057 | 0.090 | < 0.005 | < 0.005 | 0.15 |
| | | | | | | 35 | 0.056 | 0.086 | < 0.005 | < 0.005 | 0.14 |
| RV021-12DA-TRTDC Enid, Oklahoma, USA, 2012 (Loring) | 2 (11) | 74 76 | 199 215 | 438 468 | Fruit | 0 | 1.3 | 0.11 | 0.016 | < 0.005 | 1.4 |
| | | | | | | 6 | 0.61 | 0.19 | 0.015 | 0.0056 | 0.80 |
| | | | | | | 13 | 0.31 | 0.25 | < 0.005 | < 0.005 | 0.56 |
| | | | | | | 21 | 0.19 | 0.39 | < 0.005 | < 0.005 | 0.57 |
| | | | | | | 28 | 0.12 | 0.40 | < 0.005 | < 0.005 | 0.52 |
| | | | | | | 35 | 0.13 | 0.41 | < 0.005 | < 0.005 | 0.54 |
| RV021-12DA-TRTDD Enid, Oklahoma, USA, 2012 (Loring) | 2 (11) | 74 76 | 202 206 | 1979 2163 | Fruit | 0 | 1.3 | 0.11 | 0.013 | 0.0061 | 1.4 |
| | | | | | | 6 | 0.50 | 0.15 | 0.012 | < 0.005 | 0.64 |
| | | | | | | 13 | 0.26 | 0.22 | 0.0055 | < 0.005 | 0.48 |
| | | | | | | 21 | 0.15 | 0.30 | < 0.005 | < 0.005 | 0.45 |
| | | | | | | 28 | 0.14 | 0.36 | < 0.005 | < 0.005 | 0.50 |
| | | | | | | 35 | 0.20 | 0.41 | < 0.005 | < 0.005 | 0.62 |
| RV022-12HA-TRTDC Madera, California, USA, 2012 (Maycrest) | 2 (10) | 74 87 | 209 207 | 384 378 | Fruit | 14 | 0.30 | 0.059 | < 0.005 | < 0.005 | 0.36 |
| | | | | | Fruit RAC | 14 | 0.43 | 0.073 | 0.0059 | < 0.005 | 0.50 |
| | | | | | | | 0.42 | 0.080 | 0.0060 | < 0.005 | 0.50 |
| | | | | | | | 0.42 | 0.073 | 0.0063 | < 0.005 | 0.49 |
| | | | | | | mean | 0.42 | 0.075 | 0.0061 | < 0.005 | 0.50 |
| | | | | | Fruit washed | 14 | 0.29 | 0.095 | < 0.005 | < 0.005 | 0.38 |
| | | | | | | | 0.28 | 0.10 | 0.0050 | < 0.005 | 0.38 |
| | | | | | | | 0.27 | 0.087 | < 0.005 | < 0.005 | 0.35 |
| | | | | | | mean | 0.28 | 0.095 | < 0.005 | < 0.005 | 0.37 |
| | | | | | Fruit, cooked | 14 | 0.13 | < 0.025 | < 0.005 | < 0.005 | 0.15 |
| | | | | | | | 0.11 | < 0.025 | < 0.005 | < 0.005 | 0.14 |
| | | | | | | | 0.11 | 0.026 | < 0.005 | < 0.005 | 0.14 |
| | | | | | | mean | 0.12 | < 0.025 | < 0.005 | < 0.005 | 0.14 |
| | | | | | Fruit, peeled | 14 | 0.27 | 0.046 | < 0.005 | < 0.005 | 0.31 |
| | | | | | | | 0.26 | 0.055 | < 0.005 | < 0.005 | 0.31 |
| | | | | | | | 0.26 | 0.054 | < 0.005 | < 0.005 | 0.31 |
| | | | | | | mean | 0.26 | 0.051 | < 0.005 | < 0.005 | 0.31 |
| RV022-12HA-TRTDD Madera, California, USA, 2012 (Maycrest) | 2 (10) | 74 87 | 207 209 | 2366 2380 | Fruit | 14 | 0.62 | 0.092 | 0.0077 | < 0.005 | 0.71 |
| RV023-12HA-TRTDC Selma, California, USA, 2012 (September Sun) | 2 (9) | 79 78 | 202 202 | 279 280 | Fruit | 14 | 0.061 | < 0.025 | < 0.005 | < 0.005 | 0.086 |
| RV023-12HA-TRTDD | 2 (9) | 79 78 | 207 204 | 2836 2798 | Fruit | 14 | 0.31 | 0.061 | 0.0074 | < 0.005 | 0.37 |

| Trial No., Location, Year (Variety) | Application | | | | Sample | DALA | Residues as parent (mg/kg) | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | No. (RTI, days) | Growth Stage | Rate (g ai/ha) | Volume (L/ha) | | | Parent | DFA | DFEAF | 6-CNA | Parent + DFA + CNA |
| Selma, California, USA, 2012 (September Sun) | | | | | | | | | | | |
| RV025-12DA-TRTDC Hood River, Oregon, USA, 2012 (Red Haven) | 2 (10) | 78 79 | 203 214 | 402 408 | Fruit | 0 | 0.13 | < 0.025 | < 0.005 | < 0.005 | 0.16 |
| | | | | | | 6 | 0.12 | < 0.025 | < 0.005 | < 0.005 | 0.14 |
| | | | | | | 14 | 0.11 | 0.026 | < 0.005 | < 0.005 | 0.13 |
| | | | | | | 21 | 0.081 | < 0.025 | < 0.005 | < 0.005 | 0.11 |
| | | | | | | 27 | 0.058 | 0.031 | < 0.005 | < 0.005 | 0.089 |
| RV025-12DA-TRTDD Hood River, Oregon, USA, 2012 (Red Haven) | 2 (10) | 78 79 | 204 203 | 2218 2309 | Fruit | 0 | 0.12 | < 0.025 | < 0.005 | < 0.005 | 0.14 |
| | | | | | | 6 | 0.19 | 0.028 | < 0.005 | < 0.005 | 0.21 |
| | | | | | | 14 | 0.22 | 0.037 | < 0.005 | < 0.005 | 0.25 |
| | | | | | | 21 | 0.093 | 0.044 | < 0.005 | < 0.005 | 0.14 |
| | | | | | | 27 | 0.049 | 0.035 | < 0.005 | < 0.005 | 0.083 |

LOQ is 0.005 mg/kg for each of parent flupyradifurone and the metabolites DFEAF and 6-CNA and 0.025 mg/kg for DFA (parent equivs.)

Table 5 Residues from the foliar application of flupyradifurone to plums in the USA and Canada (Beedle and Jerkins 2015, RARVY014)

| Trial No., Location, Year (Variety) | Application | | | | Sample | DALA | Residues as parent (mg/kg) | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | No. (RTI, days) | Growth Stage | Rate (g ai/ha) | Volume (L/ha) | | | Parent | DFA | DFEAF | 6-CNA | Parent + DFA + CNA |
| GAP, USA, Stone fruit Foliar | 2 (10) | | 205 | | | 14 | | | | | |
| RV026-12DA-TRTDC Williamstown, New York USA, 2013 (Ozark and Starking Delicious) | 2 (10) | 78 85 | 207 205 | 426 422 | Fruit | 0 | 0.14 | < 0.05 | < 0.01 | < 0.01 | 0.19 |
| | | | | | | 7 | 0.072 | 0.059 | < 0.01 | < 0.01 | 0.13 |
| | | | | | | 13 | 0.070 | 0.085 | < 0.01 | < 0.01 | 0.15 |
| | | | | | | 21 | 0.053 | 0.13 | < 0.01 | < 0.01 | 0.19 |
| | | | | | | 28 | 0.031 | 0.11 | < 0.01 | < 0.01 | 0.14 |
| | | | | | | 35 | 0.038 | 0.19 | < 0.01 | < 0.01 | 0.23 |
| RV026-12DA-TRTDD Williamstown, New York USA, 2013 (Ozark and Starking Delicious) | 2 (10) | 78 85 | 208 208 | 2139 2138 | Fruit | 0 | 0.25 | < 0.05 | < 0.01 | < 0.01 | 0.30 |
| | | | | | | 7 | 0.18 | 0.058 | < 0.01 | < 0.01 | 0.23 |
| | | | | | | 13 | 0.15 | 0.060 | < 0.01 | < 0.01 | 0.21 |
| | | | | | | 21 | 0.097 | 0.081 | < 0.01 | < 0.01 | 0.18 |
| | | | | | | 28 | 0.10 | 0.14 | < 0.01 | 0.014 | 0.26 |
| | | | | | | 35 | 0.063 | 0.13 | < 0.01 | 0.012 | 0.21 |
| RV027-12DA-TRTDC Hart, Michigan USA, 2012 (Stanley) | 2 (10) | 77 79 | 207 207 | 375 403 | Fruit | 0 | 0.089 | < 0.05 | < 0.01 | < 0.01 | 0.14 |
| | | | | | | 7 | 0.063 | < 0.05 | < 0.01 | < 0.01 | 0.11 |
| | | | | | | 14 | 0.031 | < 0.05 | < 0.01 | < 0.01 | 0.081 |
| | | | | | | 21 | 0.014 | < 0.05 | < 0.01 | < 0.01 | 0.064 |
| | | | | | | 28 | 0.014 | < 0.05 | < 0.01 | < 0.01 | 0.064 |
| | | | | | | 35 | < 0.01 | < 0.05 | < 0.01 | < 0.01 | < 0.060 |
| RV027-12DA-TRTDD Hart, Michigan USA, 2012 (Stanley) | 2 (10) | 77 78 | 211 206 | 2136 2017 | Fruit | 0 | 0.16 | < 0.05 | < 0.01 | < 0.01 | 0.21 |
| | | | | | | 7 | 0.038 | < 0.05 | < 0.01 | < 0.01 | 0.088 |
| | | | | | | 14 | 0.037 | < 0.05 | < 0.01 | < 0.01 | 0.087 |
| | | | | | | 21 | 0.027 | < 0.05 | < 0.01 | < 0.01 | 0.077 |
| | | | | | | 28 | 0.030 | < 0.05 | < 0.01 | < 0.01 | 0.080 |
| | | | | | | 35 | 0.029 | < 0.05 | < 0.01 | < 0.01 | 0.079 |
| RV028-12DC- | 2 | NR | 206 | 202 | Fruit | 0 | 0.26 | 0.053 | < 0.01 | < 0.01 | 0.31 |

| Trial No., Location, Year (Variety) | Application | | | | Sample | DALA | Residues as parent (mg/kg) | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | No. (RTI, days) | Growth Stage | Rate (g ai/ha) | Volume (L/ha) | | | Parent | DFA | DFEAF | 6-CNA | Parent + DFA + CNA |
| TRTDC Branchton, Ontario Canada, 2014 (German) | (11) | NR | 193 | 190 | | 7 | 0.19 | 0.13 | < 0.01 | < 0.01 | 0.32 |
| | | | | | | 14 | 0.12 | 0.20 | < 0.01 | < 0.01 | 0.33 |
| | | | | | | 21 | 0.14 | 0.33 | < 0.01 | < 0.01 | 0.47 |
| | | | | | | 28 | 0.15 | 0.44 | < 0.01 | < 0.01 | 0.59 |
| | | | | | | 35 | 0.12 | 0.45 | < 0.01 | < 0.01 | 0.57 |
| RV028-12DC-TRTDD Branchton, Ontario Canada, 2014 (German) | 2<br>(11) | NR<br>NR | 200<br>196 | 2542<br>2483 | Fruit | 14 | 0.21 | 0.14 | < 0.01 | < 0.01 | 0.36 |
| | | | | | | 21 | 0.26 | 0.24 | < 0.01 | < 0.01 | 0.50 |
| | | | | | | 28 | 0.19 | 0.24 | < 0.01 | < 0.01 | 0.43 |
| | | | | | | 35 | 0.20 | 0.24 | < 0.01 | < 0.01 | 0.45 |
| RV029-12HA-TRTDC Madera, California USA, 2012 (Fortune) | 2<br>(10) | 81<br>81 | 209<br>210 | 388<br>387 | Fruit | 14 | 0.086 | 0.11 | < 0.01 | < 0.01 | 0.20 |
| RV029-12HA-TRTDD Madera, California USA, 2012 (Fortune) | 2<br>(10) | 81<br>81 | 209<br>207 | 2401<br>2390 | Fruit | 14 | 0.098 | 0.062 | < 0.01 | < 0.01 | 0.16 |
| RV030-12DA-TRTDC Selma, California, USA, 2012 (Howard Sun) | 2<br>(10) | 79<br>81 | 200<br>204 | 277<br>283 | Fruit | 0 | 0.047 | < 0.05 | < 0.01 | < 0.01 | 0.097 |
| | | | | | | 6 | 0.011 | < 0.05 | < 0.01 | < 0.01 | 0.061 |
| | | | | | | 14 | < 0.01 | 0.14 | < 0.01 | < 0.01 | 0.15 |
| | | | | | | 21 | < 0.01 | 0.15 | < 0.01 | < 0.01 | 0.16 |
| | | | | | | 28 | < 0.01 | 0.27 | < 0.01 | < 0.01 | 0.28 |
| | | | | | | 35 | < 0.01 | 0.33 | < 0.01 | < 0.01 | 0.34 |
| RV030-12DA-TRTDD Selma, California, USA, 2012 (Howard Sun) | 2<br>(10) | 79<br>81 | 206<br>207 | 2824<br>2829 | Fruit | 0 | 0.11 | < 0.05 | < 0.01 | < 0.01 | 0.16 |
| | | | | | | 6 | 0.059 | 0.076 | < 0.01 | < 0.01 | 0.13 |
| | | | | | | 14 | 0.046 | 0.17 | < 0.01 | < 0.01 | 0.22 |
| | | | | | | 21 | 0.029 | 0.23 | < 0.01 | < 0.01 | 0.26 |
| | | | | | | 28 | 0.027 | 0.34 | < 0.01 | < 0.01 | 0.37 |
| | | | | | | 35 | 0.013 | 0.39 | < 0.01 | < 0.01 | 0.40 |
| RV032-12DA-TRTDC Lindsay, California, USA, 2012 (Angelina's) | 2<br>(10) | 81<br>85 | 205<br>200 | 466<br>420 | Fruit | 0 | 0.15 | < 0.05 | < 0.01 | < 0.01 | 0.20 |
| | | | | | | 6 | 0.17 | 0.055 | < 0.01 | < 0.01 | 0.22 |
| | | | | | | 14 | 0.11 | < 0.05 | < 0.01 | < 0.01 | 0.16 |
| | | | | | | 20 | 0.11 | 0.081 | < 0.01 | < 0.01 | 0.19 |
| | | | | | | 28 | 0.14 | 0.13 | < 0.01 | < 0.01 | 0.27 |
| | | | | | | 33 | 0.12 | 0.13 | < 0.01 | < 0.01 | 0.24 |
| RV032-12DA-TRTDC Lindsay, California, USA, 2012 (Angelina's) | 2<br>(10) | 81<br>85 | 209<br>203 | 2477<br>2522 | Fruit | 0 | 0.11 | < 0.05 | < 0.01 | < 0.01 | 0.16 |
| | | | | | | 6 | 0.11 | < 0.05 | < 0.01 | < 0.01 | 0.16 |
| | | | | | | 14 | 0.066 | < 0.05 | < 0.01 | < 0.01 | 0.12 |
| | | | | | | 20 | 0.085 | 0.068 | < 0.01 | < 0.01 | 0.15 |
| | | | | | | 28 | 0.065 | 0.12 | < 0.01 | < 0.01 | 0.19 |
| | | | | | | 33 | 0.051 | 0.11 | < 0.01 | < 0.01 | 0.16 |
| RV033-12DA-TRTDC Weiser, Idaho, USA, 2012 (Empress) | 2<br>(9) | 81<br>81 | 206<br>207 | 421<br>424 | Fruit | 0 | 0.13 | < 0.05 | < 0.01 | < 0.01 | 0.18 |
| | | | | | | 7 | 0.046 | < 0.05 | < 0.01 | < 0.01 | 0.096 |
| | | | | | | 14 | 0.051 | 0.059 | < 0.01 | < 0.01 | 0.11 |
| | | | | | | 21 | 0.021 | 0.084 | < 0.01 | < 0.01 | 0.10 |
| | | | | | | 27 | 0.025 | 0.12 | < 0.01 | < 0.01 | 0.15 |
| | | | | | | 35 | 0.019 | 0.13 | < 0.01 | < 0.01 | 0.15 |
| RV033-12DA-TRTDD Weiser, Idaho, USA, 2012 (Empress) | 2<br>(9) | 81<br>81 | 207<br>208 | 1959<br>1970 | Fruit | 0 | 0.081 | < 0.05 | < 0.01 | < 0.01 | 0.13 |
| | | | | | | 7 | 0.070 | < 0.05 | < 0.01 | < 0.01 | 0.12 |
| | | | | | | 14 | 0.068 | 0.062 | < 0.01 | < 0.01 | 0.13 |
| | | | | | | 21 | 0.042 | 0.098 | < 0.01 | < 0.01 | 0.14 |
| | | | | | | 27 | 0.039 | 0.13 | < 0.01 | < 0.01 | 0.16 |
| | | | | | | 35 | 0.027 | 0.13 | < 0.01 | < 0.01 | 0.15 |

| Trial No., Location, Year (Variety) | Application | | | | Sample | DALA | Residues as parent (mg/kg) | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | No. (RTI, days) | Growth Stage | Rate (g ai/ha) | Volume (L/ha) | | | Parent | DFA | DFEAF | 6-CNA | Parent + DFA + CNA |
| RV034-12DA-TRTDC Newberg, Oregon, USA, 2012 (Italian) | 2 (9) | 79 81 | 204 205 | 377 371 | Fruit | 0 | 0.11 | < 0.05 | < 0.01 | < 0.01 | 0.16 |
| | | | | | | 7 | 0.061 | < 0.05 | < 0.01 | < 0.01 | 0.11 |
| | | | | | | 14 | 0.061 | < 0.05 | < 0.01 | < 0.01 | 0.11 |
| | | | | | | 21 | 0.089 | < 0.05 | < 0.01 | < 0.01 | 0.14 |
| | | | | | | 28 | 0.069 | 0.052 | < 0.01 | < 0.01 | 0.12 |
| | | | | | | 35 | 0.041 | < 0.05 | < 0.01 | < 0.01 | 0.091 |
| RV034-12DA-TRTDD Newberg, Oregon, USA, 2012 (Italian) | 2 (9) | 79 81 | 203 204 | 2862 2878 | Fruit | 0 | 0.16 | < 0.05 | < 0.01 | < 0.01 | 0.21 |
| | | | | | | 7 | 0.11 | < 0.05 | < 0.01 | < 0.01 | 0.16 |
| | | | | | | 14 | 0.082 | < 0.05 | < 0.01 | < 0.01 | 0.13 |
| | | | | | | 21 | 0.070 | < 0.05 | < 0.01 | < 0.01 | 0.12 |
| | | | | | | 28 | 0.065 | 0.065 | < 0.01 | < 0.01 | 0.13 |
| | | | | | | 35 | 0.058 | 0.079 | < 0.01 | < 0.01 | 0.14 |

LOQ is 0.01 mg/kg for each of parent flupyradifurone and the metabolites DFEAF and 6-CNA and 0.05 mg/kg for DFA (parent equivs.)

## FATE OF RESIDUES IN STORAGE AND PROCESSING

### *Residues after processing*

The fate of flupyradifurone residues during processing of raw agricultural commodities was investigated in stone fruit (plums and peaches).

As a measure for the transfer of residues into processed products, a processing factor was used, which is defined as:

$$TF = \frac{\text{Residue in processed products (mg/kg)}}{\text{Residue in raw agricultural commodity)(mg/kg)}}$$

*Plums*

The effect of processing (laboratory scale) on residues of flupyradifurone in plums (Table 6) was investigated in two trials carried out in the 2011 growing season in the USA (Lam and Jerkins 2015, RARVY040).

Plums with incurred residues were obtained where trees were sprayed with two foliar sprays of BYI 02960 200 SL (200 g ai/L) at exaggerated application rates (992–1008 g ai/ha), with a 9 or 10-day retreatment interval. The spray volumes for the treated plots were 1830–1960 L/ha. All applications were made with ground-based equipment. NIS tank adjuvant was added at 0.20% v/v. One control and one treated bulk plum whole fruit sample were harvested 13 or 14 days after the last application. Bulk plum samples (one control and one treated for each trial) were processed into plum commodity samples (pitted fruit, washed and pitted fruit, dried and pitted fruit and washings).

The sample was sorted and any leaves, stems and other debris along with rotten or otherwise damaged fruit were removed. An aliquot of sorted fresh plums was reserved for pitting prior to washing. After pitting (removal of stones), a representative sample of unwashed pitted plums was removed, packaged, labelled and placed in frozen storage for the required sample fraction.

The remaining fresh plums were washed and a sample of wash water was removed, packaged, labelled and placed in frozen storage for the required sample fraction. A portion of the remaining washed fruit was placed in each of three drying trays; the remaining balance was discarded. The drying trays were heated at 74 °C and were periodically removed from the airdryer and weighed. The

trays were periodically rotated to ensure even drying. The fruit were removed when an average plum moisture content of 33.8–36.5% was achieved.

The plums were allowed to cool then were sliced with a knife and the pits removed. The cooled, pitted plums were packaged, labelled and placed in frozen storage for the required sample fraction.

Residues of flupyradifurone and metabolites in plum RAC and processed commodities were quantitated using analytical methods RV-001-P10-02 and RV-001-P10-03 by LC-MS/MS using stable isotopically labelled internal standards (Method 01304). No residues above the LOQ were found in the control samples. Acceptable concurrent recovery data for plum commodities were obtained for each analyte.

The maximum storage period of frozen samples before analysis was approximately 15 months (447 days for whole fruits (RAC) and washings, 442 days for pitted fruits, 448 days for washed and pitted fruits and 449 days for the dried and pitted fruit) which is covered by the available storage stability data.

Table 6 Residues in plum processed fractions from the foliar application of flupyradifurone to plums in the USA (Lam and Jerkins 2015, RARVY040)

| Trial No., Location, Year (Variety) | Sample | DALA | Residues as parent (mg/kg) | | | | | Processing Factor (Parent + DFA + 6-CNA) |
|---|---|---|---|---|---|---|---|---|
| | | | Parent | DFA | DFEAF | 6-CNA | Parent + DFA + 6-CNA | |
| GAP, USA, Stone fruits<br>2 applications<br>at 205 g ai/ha<br>with 10 day RTI | | 14 | | | | | | |
| RV237-11PA-TRT5X, | Fruit | 13 | 0.42 | 0.088 | 0.015 | 0.020 | 0.53 | |
| Hart, | (RAC) | | 0.44 | 0.085 | 0.018 | 0.017 | 0.55 | |
| Michigan, | | | 0.44 | 0.078 | < 0.01 | < 0.01 | 0.52 | |
| USA, 2011 | | **mean** | **0.43** | **0.084** | **0.014** | **0.016** | **0.53** | **NA** |
| (Stanley) | Fruit, pitted | 13 | 1.1 | 0.17 | < 0.01 | < 0.01 | 1.3 | |
| | | | 0.74 | 0.12 | < 0.01 | < 0.01 | 0.86 | |
| 2 applications | | | 0.78 | 0.12 | < 0.01 | < 0.01 | 0.90 | |
| 10 day RTI | | **mean** | **0.87** | **0.14** | **< 0.01** | **< 0.01** | **1.0** | **1.9** |
| Growth Stages 81 and 85 | Fruit, | 13 | 0.83 | 0.13 | < 0.01 | < 0.01 | 0.96 | |
| 998 and 1008 g ai/ha | washed, | | 0.89 | 0.14 | < 0.01 | < 0.01 | 1.0 | |
| 1960 and 1950 L/ha | Pitted | | 0.71 | 0.13 | < 0.01 | < 0.01 | 0.84 | |
| | | **mean** | **0.81** | **0.13** | **< 0.01** | **< 0.01** | **0.94** | **1.8** |
| | Fruit, | 13 | 3.1 | 0.45 | < 0.01 | 0.049 | 3.6 | |
| | dried and | | 3.4 | 0.45 | < 0.01 | 0.056 | 3.9 | |
| | Pitted | | 3.2 | 0.45 | < 0.01 | 0.049 | 3.7 | |
| | (prunes) | **mean** | **3.2** | **0.45** | **< 0.01** | **0.051** | **3.7** | **7.0** |
| | Washings | 13 | < 0.01 | < 0.05 | < 0.01 | < 0.01 | < 0.06 | |
| | | | < 0.01 | < 0.05 | < 0.01 | < 0.01 | < 0.06 | |
| | | | < 0.01 | < 0.05 | < 0.01 | < 0.01 | < 0.06 | |
| | | **Mean** | **< 0.01** | **< 0.05** | **< 0.01** | **< 0.01** | **< 0.06** | **< 0.11** |
| RV238-11PA-TRT5X, | Fruit | 14 | 0.25 | 0.098 | < 0.01 | < 0.01 | 0.35 | |
| Kerman, California | (RAC) | | 0.28 | 0.089 | < 0.01 | < 0.01 | 0.37 | |
| USA, 2011 | | | 0.28 | 0.099 | < 0.01 | < 0.01 | 0.38 | |
| (French prune) | | **Mean** | **0.27** | **0.095** | **< 0.01** | **< 0.01** | **0.37** | **NA** |
| | Fruit, pitted | 14 | 0.21 | 0.077 | < 0.01 | < 0.01 | 0.29 | |
| 2 applications | | | 0.22 | 0.077 | < 0.01 | < 0.01 | 0.30 | |
| 9 day RTI | | | 0.31 | 0.099 | < 0.01 | < 0.01 | 0.41 | |
| Growth Stage 79 and 85 | | **Mean** | **0.25** | **0.084** | **< 0.01** | **< 0.01** | **0.33** | **0.89** |
| 992 and 997 g ai/ha | Fruit, | 14 | 0.39 | 0.099 | < 0.01 | < 0.01 | 0.49 | |
| 1830 and 1850 L/ha | washed, | | 0.26 | 0.090 | < 0.01 | < 0.01 | 0.35 | |
| | Pitted | | 0.20 | 0.084 | < 0.01 | < 0.01 | 0.28 | |
| | | **Mean** | **0.28** | **0.091** | **< 0.01** | **< 0.01** | **0.37** | **1.0** |
| | Fruit, | 14 | 0.92 | 0.25 | < 0.01 | 0.043 | 1.2 | |
| | dried and | | 0.79 | 0.24 | < 0.01 | 0.039 | 1.1 | |
| | Pitted | | 0.85 | 0.23 | < 0.01 | 0.034 | 1.1 | |
| | (prunes) | **Mean** | **0.85** | **0.24** | **< 0.01** | **0.039** | **1.1** | **3.0** |
| | Washings | 14 | < 0.01 | < 0.05 | < 0.01 | < 0.01 | < 0.06 | |
| | | | < 0.01 | < 0.05 | < 0.01 | < 0.01 | < 0.06 | |

| Trial No., Location, Year (Variety) | Sample | DALA | Residues as parent (mg/kg) | | | | | Processing Factor (Parent + DFA + 6-CNA) |
|---|---|---|---|---|---|---|---|---|
| | | | Parent | DFA | DFEAF | 6-CNA | Parent + DFA + 6-CNA | |
| | | | < 0.01 | < 0.05 | < 0.01 | < 0.01 | < 0.06 | |
| | | **Mean** | **< 0.01** | **< 0.05** | **< 0.01** | **< 0.01** | < 0.06 | **< 0.16** |

LOQ is 0.01 mg/kg for each of parent and the metabolites DFEAF and 6-CNA and 0.05 mg/kg for DFA (parent equivs.) for all processed commodities

For processing, three sub-samples out of a single sample collected from the treated trial were used for each processing step (RAC, fruit pitted, fruit washed and pitted, fruit pitted and dried, washings). Mean residues are shown in bold.

*Peaches*

The effect of processing (laboratory scale) on residues of flupyradifurone in peaches (Table 7) was investigated in two trials carried out in the 2010 growing season in the EU (Noss and Teubner 2012, RARVL062).

Peaches with incurred residues were obtained where trees were sprayed with two foliar sprays at 127.5 or 150 g ai/ha with a 14-day retreatment interval. The spray volumes for the treated plots were 850 or 1000 L/ha. Peach bulk RAC samples were harvested 14 days after the last application. Bulk peach samples (one control and one treated for each trial) were processed into peach commodity samples (washed fruit, peel, peeled fruit, preserve and washings). Samples were stored deep-frozen within 24 hours of sampling.

The processing of peach samples was designed to simulate procedures of peach fruit into preserved (= canned) fruit. Peach fruit was washed, peeled, and the stone was removed. The pitted fruit was sterilized at 92–94 °C for approximately 1–2 minutes. The total time of the sterilisation cycles, including heating and cooling, was 21–29 minutes. After processing, samples were stored in a freezer until analysis.

Residues of flupyradifurone and metabolites in peach RAC and processed commodities were quantitated using LC-MS/MS Method 01304. No residues above the LOQ were found in the control samples. Acceptable concurrent recovery data for peach commodities were obtained for each analyte.

The storage period of frozen samples before analysis was 230 or 328 days, which is covered by the available storage stability data.

Table 7 Residues in peach processed fractions from the foliar application of flupyradifurone to peaches in the EU (Noss and Teubner 2012, RARVL062)

| Trial No., Location, Year (Variety) | Sample | DALA | Residues as parent (mg/kg) | | | | | Processing Factor (Parent + DFA + 6-CNA) |
|---|---|---|---|---|---|---|---|---|
| | | | Parent | DFA | DFEAF | 6-CNA | Parent + DFA + 6-CNA | |
| GAP, USA, Stone fruits<br>2 applications at 205 g ai/ha<br>with 10 day RTI | | 14 | | | | | | |
| 10-3216-02,<br>E-41320 Cantillana, Sevilla<br>Spain, 2010<br>(Transvalia)<br>2 applications<br>14 day RTI<br>Growth Stages 75 and 81<br>127.5 (×2) g ai/ha<br>850 (×2) L/ha | Fruit (RAC) | 14 | 0.09 | 0.04 | < 0.01 | < 0.01 | 0.13 | NA |
| | Fruit, washed | 14 | 0.05 | 0.05 | < 0.01 | < 0.01 | 0.10 | 0.77 |
| | Washings | 14 | 0.01 | < 0.02 | < 0.01 | < 0.01 | < 0.03 | < 0.23 |
| | Peel | 14 | 0.16 | 0.06 | < 0.01 | < 0.01 | 0.22 | 1.7 |
| | Fruit, peeled | 14 | 0.02 | 0.03 | < 0.01 | < 0.01 | 0.05 | 0.38 |
| | Preserve (canned) | 14 | 0.03 | 0.03 | < 0.01 | < 0.01 | 0.06 | 0.46 |
| 10-3216-03,<br>I-44124 San Marino,<br>Italy, 2010<br>(Flaminia)<br>2 applications<br>14 day RTI | Fruit (RAC) | 14 | 0.20 | 0.05 | < 0.01 | < 0.01 | 0.25 | NA |
| | Fruit, washed | 14 | 0.21 | 0.06 | < 0.01 | < 0.01 | 0.27 | 1.1 |
| | Washings | 14 | 0.03 | < 0.02 | < 0.01 | < 0.01 | < 0.05 | < 0.20 |
| | Peel | 14 | 0.84 | 0.07 | < 0.01 | < 0.01 | 0.91 | 3.6 |
| | Fruit, peeled | 14 | 0.08 | 0.05 | < 0.01 | < 0.01 | 0.13 | 0.52 |
| | Preserve (canned) | 14 | 0.07 | 0.03 | < 0.01 | < 0.01 | 0.10 | 0.40 |

| Trial No., Location, Year (Variety) | Sample | DALA | Residues as parent (mg/kg) | | | | | Processing Factor (Parent + DFA + 6-CNA) |
|---|---|---|---|---|---|---|---|---|
| | | | Parent | DFA | DFEAF | 6-CNA | Parent + DFA + 6-CNA | |
| Growth Stages 76 and 81<br>150 (×2) g ai/ha<br>1000 (×2) L/ha | | | | | | | | |

LOQ is 0.01 mg/kg for each of parent and the metabolites DFEAF and 6-CNA and 0.02 mg/kg for DFA (parent equivs.) for all processed commodities

The results of the processing factors are summarised in Table 8 below:

Table 8 Summary of processing factors for flupyradifurone residues

| Raw Agricultural Commodity (RAC) | Processed Commodity | Calculated processing factors (parent only) | Best estimate processing factor (parent only) | Calculated Processing factors (Parent + DFA + 6-CNA) | Best Estimate Processing Factor (Parent + DFA + 6-CNA) |
|---|---|---|---|---|---|
| Peach | Fruit, washed | 0.56, 0.67, 0.73, 0.92, 1.0 | 0.73 | 0.74, 0.77, 0.80, 0.88, 1.1 | 0.80 |
| | Washings | 0.11, 0.15 | 0.13 | 0.20, 0.23 | 0.215 |
| | Peel | 1.8, 4.2 | 3.0 | 1.7, 3.6 | 2.65 |
| | Fruit, peeled | 0.22, 0.34, 0.40, 0.51, 0.62 | 0.40 | 0.38, 0.52, 0.60, 0.62, 0.71 | 0.60 |
| | Fruit, cooked | 0.29, 0.30, 0.31 | 0.30 | 0.28, 0.35, 0.38 | 0.35 |
| | Canned peaches (peeled fruit with stone removed) | 0.33, 0.35 | 0.34 | 0.40, 0.46 | 0.43 |
| Cherries | Fruit, washed | 0.95, 0.96 | 0.955 | 0.97, 0.98 | 0.975 |
| | Fruit, cooked | 0.26, 0.50 | 0.38 | 0.24, 0.46 | 0.35 |
| Plum | Fruit pitted (stones removed) | 0.93, 2.0 | 1.5 | 0.89, 1.9 | 1.40 |
| | Fruit, washed, pitted | 1.0, 1.9 | 1.5 | 1.0, 1.8 | 1.4 |
| | Fruit, dried and pitted (prunes) | 3.1, 7.4 | 5.3 | 3.0, 7.0 | 5.0 |
| | Washings | 0.02, 0.04 | 0.03 | 0.11, 0.16 | 0.135 |

## APPRAISAL

Flupyradifurone was first evaluated for toxicology and residues by the 2015 JMPR and 2016 JMPR respectively. The 2015 JMPR established an ADI for flupyradifurone of 0–0.08 mg/kg bw/day and an ARfD of 0.2 mg/kg bw. At the 48th Session of the CCPR (2016), flupyradifurone was listed for additional MRLs by the 2017 JMPR.

The manufacturer supplied information on an analytical method, storage stability information, a registered use pattern for stone fruit, supervised residue trials on cherries, peaches and plums and fate of residues in processing studies for peaches and plums. A product label was made available from the United States of America.

The residue definition for compliance with the MRL for plant commodities is flupyradifurone. The residue definition for estimation of dietary intake for plant commodities is the sum of flupyradifurone, difluoroacetic acid (DFA) and 6-chloronicotinic acid (6-CNA), expressed as parent equivalents.

The residue definition for compliance with the MRL and for estimation of dietary intake for animal commodities is the sum of flupyradifurone and difluoroacetic acid, expressed as parent equivalents.

The residue is not fat soluble.

### *Methods of analysis*

The Meeting received an analytical method (RV-001-P10-03) suitable for the determination of the active substance flupyradifurone and the metabolites DFA, DFEAF and 6-CNA in plant matrices. This method involves minor modifications to the clean-up steps for method RV-001-P10-2 considered by the 2016 JMPR and has an LOQ of 0.01 mg/kg for parent, difluoroethyl-amino-furanone (DFEAF), and 6-CNA (as parent equivalents) and between 0.02–0.05 mg/kg (as parent equivalents and depending on the matrix) for DFA.

Method RV-001-P10-02 was used in the storage stability study and in the peach processing study, while the modified method RV-001-P10-03 was used in the stone fruit residue trials and in the plum processing study.

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

The Meeting received information on the freezer storage stability of flupyradifurone in plant commodities. This is an extension to longer storage intervals of a study which was evaluated at JMPR 2016.

Residue trial data are supported by the supplied storage stability study which showed that flupyradifurone, DFEAF and DFA are stable for at least 52 months in high water, high acid, high oil, high protein, and high starch content matrices, when stored frozen at approximately -18 °C. The storage periods in the storage stability studies cover the storage intervals in the residue trials.

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

The Meeting received supervised trial data for application of flupyradifurone on stone fruit (cherries, peaches and plums).

For maximum residue level estimation (compliance), residues of flupyradifurone parent have been considered. For dietary intake assessment (risk assessment), residues of flupyradifurone, DFA and 6-chloronicotinic acid expressed as parent equivalents (referred to as total residues of flupyradifurone), have been considered.

Where parent or DFA residues were not detected or were less than the LOQ (*i.e.* generally < 0.01 mg/kg for parent or 0.05 mg/kg for DFA) the LOQ value was utilised for maximum residue estimation and dietary intake assessment. For 6-CNA, values less than the LOQ were not added for calculation of total residues of flupyradifurone.

The following table shows how residues in the trials were added to give total residues of flupyradifurone.

| Parent | DFA | 6-CNA | Total |
|---|---|---|---|
| < 0.01 | 0.05 | 0.01 | 0.07 |
| 0.01 | < 0.05 | 0.01 | 0.07 |
| < 0.01 | < 0.05 | < 0.01 | < 0.06 |
| 0.01 | 0.05 | < 0.01 | 0.06 |
| 0.01 | 0.05 | 0.01 | 0.07 |

*Stone Fruits – cherries, peaches and plums*

The USA GAP for stone fruit is two foliar applications at 205 g ai/ha with a 10 day minimum retreatment interval and a 14-day PHI.

Residue data for cherries (sweet and sour), peaches and plums that match the US GAP have been submitted. Foliar applications were made using either concentrated or dilute sprays. The highest residue observations have been selected from each trial for estimation of maximum residue levels and for dietary intake purposes.

The Meeting noted that the use in the USA is for the stone fruit group (Crop Group 12-12). Although the median residues for each fruit differed by a factor of less than five, the Meeting decided to recommend maximum residue levels for the individual sub-groups of stone fruit, as there are sufficient trials for each sub-group.

For the estimation of the maximum residue level the ranked order of residues of flupyradifurone in cherries from supervised trials according to the GAP in the USA was 0.014, 0.17, 0.25, 0.36, 0.36, 0.58, 0.62 and 0.94 mg/kg.

For the estimation of dietary intake the ranked order of total residues of flupyradifurone in cherries from supervised trials according to the GAP in the USA was 0.40, 0.49, 0.50, 0.54, 0.57, 0.82, 1.1 and 1.1 mg/kg.

The Meeting estimated a maximum residue level, an STMR and an HR for the subgroup of cherries of 2, 0.555 and 1.1 mg/kg respectively.

For the estimation of the maximum residue level the ranked order of residues of flupyradifurone in peaches from supervised trials according to the GAP in the USA was 0.13, 0.14, 0.22, 0.25, 0.28, 0.31, 0.31, 0.33, 0.62, 0.72 and 0.73 mg/kg.

The ranked order of total residues of flupyradifurone in peaches from supervised trials according to the GAP in the USA was 0.16, 0.25, 0.34, 0.35, 0.37, 0.39, 0.43, 0.62, 0.71, 0.94 and 1.1 mg/kg.

The Meeting estimated a maximum residue level, an STMR and an HR for the subgroup of peaches (including nectarine and apricots) of 1.5, 0.39 and 1.1 mg/kg respectively.

For the estimation of the maximum residue level the ranked order of residues of flupyradifurone in plums from supervised trials according to the GAP in the USA was 0.037, 0.046, 0.068, 0.089, 0.098, 0.14, 0.15 and 0.26 mg/kg.

For the estimation of dietary intake the ranked order of total residues of flupyradifurone in plums from supervised trials according to the GAP in the USA was 0.087, 0.14, 0.16, 0.20, 0.26, 0.27, 0.40 and 0.59 mg/kg.

The Meeting estimated a maximum residue level, an STMR and an HR for the subgroup of plums (including fresh prunes) of 0.4, 0.23 and 0.59 mg/kg respectively.

***Fate of residues during processing***

The Meeting received processing studies for peaches and plums. Additionally, some processing data was generated for peaches and cherries as part of the residue trials.

Based on the best estimate flupyradifurone processing factor (parent only, i.e. the residue definition for enforcement) of 5.3 for prunes and the plums maximum residue level of 0.4 mg/kg, the calculated expected highest residues in prunes are 2.1 mg/kg. The Meeting estimated a maximum residue level for flupyradifurone in prunes of 3 mg/kg.

All other processing factors for parent compound only (with the exceptions of peach peel, which is not of significance as either a processed food commodity or a by-product for animal feeding and pitted fresh plums, for which maximum residue levels are not set) are < 1.

The table below summarises STMR-P and HR-P values calculated from the processing factors determined for total residues of flupyradifurone (residue definition for dietary risk assessment).

| RAC | Processed Commodity | Best Estimate Processing Factor (total residues) | RAC STMR (total residues) | Processed commodity STMR-P (total residues) | RAC HR (total residues) | Processed commodity HR-P (total residues) |
|---|---|---|---|---|---|---|
| Peach | Fruit, washed | 0.80 | 0.39 | 0.31 | 1.1 | 0.88 |
| | Fruit, peeled | 0.60 | | 0.23 | | 0.66 |
| | Fruit, cooked | 0.35 | | 0.14 | | 0.38 |
| | Canned peaches (peeled fruit with stone removed) | 0.43 | | 0.17 | | 0.47 |
| Cherry | Fruit, washed | 0.975 | 0.555 | 0.54 | 1.1 | 1.07 |
| | Fruit, cooked | 0.35 | | 0.19 | | 0.38 |
| Plum | Fruit pitted (stone removed) | 1.40 | 0.23 | 0.32 | 0.59 | 0.83 |
| | Fruit, washed, pitted | 1.4 | | 0.32 | | 0.83 |
| | Fruit, pitted and dried (prunes) | 5.0 | | 1.15 | | 2.95 |

### *Farm animal dietary burden*

*Estimated maximum and mean dietary burdens of farm animals*

No stone fruit commodities are used as livestock feeds. Therefore the livestock dietary burden is unchanged from that previously calculated.

### *Animal commodity maximum residue levels*

As the additional livestock dietary burden for flupyradifurone is nil, no changes are required to animal commodity MRLs or dietary parameters.

## RECOMMENDATIONS

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

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

Definition of the residue (for estimation of dietary intake for plant commodities): Sum of flupyradifurone, difluoroacetic acid (DFA) and 6-chloropyridine-3-carboxylic acid (6-CNA), expressed as parent equivalents

Definition of the residue (for compliance with the MRL and for estimation of dietary intake for animal commodities): *Sum of flupyradifurone and difluoroacetic acid (DFA), expressed as parent equivalents*

*The residue is not fat soluble*

| Commodity | | MRL, mg/kg | STMR or STMR-P, mg/kg | HR or HR-P, mg/kg |
|---|---|---|---|---|
| CCN | Name | | | |
| FS 0013 | Subgroup of Cherries (includes all commodities in this subgroup) | 2 | 0.555 | 1.1 |
| FS 2001 | Subgroup of Peaches (including Nectarine and Apricots) (includes all commodities in this subgroup) | 1.5 | 0.39 | 1.1 |
| FS 0014 | Subgroup of Plums (including fresh Prunes) (includes all commodities in this subgroup) | 0.4 | 0.23 | 0.59 |
| DF 0014 | Prunes | 3 | 1.15 | 2.95 |
| | | | | |
| | Canned peaches | | 0.17 | 0.47 |
| | Cooked cherries (figures used in dietary intake calculations for cherry jam) | | 0.19 | 0.38 |

## DIETARY RISK ASSESSMENT

### *Long-term dietary exposure*

The 2015 JMPR established an ADI of 0–0.08 mg/kg bw for flupyradifurone. The International Estimated Daily Intakes (IEDIs) of flupyradifurone were calculated for the 17 GEMS/Food cluster diets using STMRs/STMR-Ps estimated by the current and previous Meetings. The results are shown in Annex 3 of the 2017 JMPR report. The calculated IEDIs were 6–20% of the maximum ADI. The Meeting concluded, on the basis of the information provided to the current and previous Meetings, that the long-term dietary exposure to residues of flupyradifurone is unlikely to present a public health concern.

### *Short-term dietary exposure*

The 2015 JMPR established an ARfD of 0.2 mg/kg bw for flupyradifurone. The International Estimated Short Term Intakes (IESTIs) of flupyradifurone were calculated for food commodities using the HRs/HR-Ps or STMRs/STMR-Ps estimated by the current Meeting. The results are shown in Annex 4 in the 2017 JMPR report. The IESTIs were 10% of the ARfD for the general population and 30% of the ARfD for children. The Meeting concluded that the short-term dietary exposure to residues of flupyradifurone, resulting from the uses considered by the Meeting, are unlikely to present a public health concern.

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