

DIFLUBENZURON (130)

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EXPLANATION

Diflubenzuron is an agricultural insect growth regulator. It was originally evaluated by the JMPR in 1981 and re-evaluated for residues several times up to 1988.

Diflubenzuron was evaluated under the Periodic Review Program for toxicology in 2001 and for residues in 2002 respectively.

The original ADI 0–0.02 mg/kg bw was re-confined and the acute reference dose was considered unnecessary. The residue definition is diflubenzuron both for compliance with MRLs and dietary intake. The residue is fat-soluble. The maximum residue levels for citrus fruit, pome fruit, rice, mushroom, rice straw and fodder were recommended, and the previous maximum residue level for apple, pear, plum, Brussels sprouts, head cabbage, soya bean and tomato were withdrew.

This Meeting received information on the residue analysis, storage stability, use patterns and supervised field trials for peaches, plums, peppers, mustard green, barley, wheat, almond, pecan and peanut.

Plant metabolism studies with diflubenzuron on maize, soybean, cabbage, cotton, and apple were reviewed by the 2002 JMPR. The JMPR concluded that in plants, most of the resultant residue from the use of diflubenzuron was a surface residue and that the parent diflubenzuron was the major component of the residue. In accordance with JMPR and OECD Guidelines, these metabolism studies are sufficient to cover all crops for which additional MRLs are proposed. No additional plant metabolism studies are submitted to this meeting.

RESIDUE ANALYSIS***Analytical methods***

The present meeting received the analytical methods of diflubenzuron, CPU and PCA in peaches, plums, peppers, mustard green, barley, wheat, almond, pecan and peanut which are primarily based on the methods previously reviewed by JMPR in 2002, with some modifications to minimize matrix interference depending on the matrices. The results are summarized below, including the commodities, for which the methods were validated, analytes and their limit of quantization (LOQ), determination technique and a brief description of the method.

Diflubenzuron was extracted from homogenized samples using dichloromethane (peaches, plums, peppers) ethyl acetate (mustard greens, pepper method B, almonds, and peanuts) or acetonitrile (wheat, barley). For the GC-ECD method involving derivatisation (peaches, plums, pepper, almonds, and peanuts) extracts were cleaned-up on a florisil column and derivatised with heptafluorobutyric anhydride prior to analysis by GC-ECD. Typical LOQs were 0.05 mg/kg. For the HPLC-UV methods (mustard greens, pepper method B, almonds, peanuts) extracts were cleaned-up by liquid-liquid partitioning and also using a florisil column. Quantitation of diflubenzuron residues was by HPLC with UV detection. Typical LOQs were 0.005 to 0.05 mg/kg. In the case of wheat and barley, extracts were cleaned-up by liquid-liquid partitioning followed by elution from C18 and silica gel solid-phase extraction columns. Quantification was by HPLC with UV detection. The reported LOQ ranged between 0.037–0.08 mg/kg.

Table 1 Summary of method validation data for various matrices

| Matrix | Analyte | Fortification (mg/kg) | Recoveries (%) | Mean (%) | RSD (%) | Reference |
|--------|---------------|-----------------------|-----------------------------|----------|---------|-----------|
| Peach | Diflubenzuron | 0.05(n = 7) | 89, 96, 103, 94, 95, 97, 88 | 95 | 5 | GRL-12274 |
| | | 0.07(n = 3) | 95, 95, 96 | 95 | 1 | |

| Matrix | Analyte | Fortification (mg/kg) | Recoveries (%) | Mean (%) | RSD (%) | Reference |
|----------------|---------------|---------------------------|----------------------------------|---------------|---------|------------------------|
| | CPU | 0.1(n = 3) | 89, 94, 93 | 92 | 3 | |
| | | 0.01(n = 6) | 73, 87, 88, 101, 86, 84 | 87 | 12 | |
| | | 0.05(n = 3) | 117, 98, 89 | 101 | 4 | |
| | PCA | 0.1(n = 3) | 105, 99, 97 | 100 | 3 | |
| | | 0.01(n = 6) | 109, 107, 101, 97, 97, 100 | 102 | 5 | |
| | | 0.05(n = 3) | 105, 117, 94 | 105 | 11 | |
| Plum | Diflubenzuron | 0.1(n = 3) | 101, 102, 101 | 101 | 0.3 | GRL-12274 |
| | | 0.05 (n = 7) | 98, 91, 91, 90, 107, 98, 101 | 97 | 7 | |
| | | 0.07 (n = 3) | 99, 102, 97 | 100 | 3 | |
| | CPU | 0.1 (n = 3) | 93, 99, 94 | 96 | 3 | |
| | | 0.01 (n = 8) | 82, 108, 104, 98, 85, 81, 93, 85 | 92 | 11 | |
| | | 0.05 (n = 3) | 109, 93, 104 | 102 | 8 | |
| PCA | 0.1 (n = 3) | 95, 104, 97 | 95 | 10 | | |
| | 0.005 (n = 6) | 93, 92, 97, 102, 112, 113 | 101 | 9 | | |
| | 0.05 (n = 3) | 101, 105, 100 | 102 | 3 | | |
| Mustard greens | Diflubenzuron | 0.1 (n = 3) | 87, 105, 109 | 100 | 12 | PR 08031/ 2005-059 |
| | | 0.05(n = 3) | 67, 80, 88 | 78 | 14 | |
| | | 0.5(n = 3) | 90, 91, 88 | 90 | 2 | |
| | CPU | 5(n = 3) | 81, 86, 79 | 82 | 4 | |
| | | 0.01(n = 3) | 80, 90, 90 | 87 | 7 | |
| | PCA | 0.10(n = 3) | 88, 93, 88 | 90 | 3 | |
| 0.005(n = 3) | | 112, 118, 108 | 113 | 4 | | |
| Pepper | Diflubenzuron | 0.05(n = 3) | 112, 120 | 116 | ----- | RP-97016 |
| | | 0.05(n = 6) | 85, 85, 73, 85, 66, 85 | 80 | 10 | |
| | | 0.5(n = 6) | 87, 98, 94, 93, 104, 97 | 96 | 6 | |
| | CPU | 1(n = 6) | 92, 83, 84, 84, 82, 88 | 86 | 4 | |
| | | 0.01(n = 6) | 92, 82, 94, 76, 100, 80 | 87 | 11 | |
| | | 0.1(n = 1) | 94, 100, 84, 100, 81 | 92 | 10 | |
| | PCA | 0.2(n = 5) | 84 | ----- | ----- | |
| | | 0.6(n = 6) | 110, 81, 109, 80, 107, 83 | 95 | 16 | |
| | | 0.005(n = 6) | 104, 93, 97, 94, 101, 99 | 98 | 4 | |
| | | 0.010(n = 6) | 102, 96, 106, 99, 106, 98 | 101 | ----- | |
| | Diflubenzuron | 0.05(n = 6) | 117, 93, 106, 97, 80, 95 | 98 | ----- | RP-03007/ GRL-12164 |
| | | 0.005(n = 3) | 82, 95, 97 | 92 | 8.8 | |
| | | 0.05(n = 3) | 70, 77, 85 | 77 | 9.4 | |
| | CPU | 0.5(n = 3) | 81, 78, 76 | 78 | 2.8 | |
| | | 0.005(n = 3) | 95, 101, 110 | 102 | 7.2 | |
| | | 0.05(n = 3) | 100, 80, 88 | 89 | 11 | |
| | PCA | 0.5(n = 3) | 83, 92, 119 | 98 | 19 | |
| | | 0.005(n = 3) | 103, 110, 107 | 107 | 3.3 | |
| | | 0.05(n = 3) | 101, 106, 101 | 102 | 3 | |
| | Wheat grain | Diflubenzuron | 0.5(n = 3) | 112, 100, 105 | 106 | |
| 0.05 | | | 98, 96, 94 | 96 | 2 | |
| Wheat hay | Diflubenzuron | 0.5 | 94, 87, 101 | 94 | 7 | |
| | | 0.05 | 86, 112, 94 | 97 | 13 | |
| Wheat straw | Diflubenzuron | 0.5 | 82, 91, 84 | 86 | 6 | |
| | | 0.05 | 80, 78, 104 | 87 | 15 | |
| Wheat flour | Diflubenzuron | 0.5 | 81, 91, 78 | 83 | 7 | |
| | | 0.05 | 98, 102, 98 | 99 | 2 | |
| Wheat bran | Diflubenzuron | 0.5 | 98, 96, 84 | 93 | 8 | |
| | | 0.05 | 84, 70, 74 | 76 | 9 | |
| Wheat germ | Diflubenzuron | 0.5 | 82, 85, 91 | 86 | 5 | |
| | | 0.05 | 70, 62, 84 | 72 | 11 | |
| Wheat grain | CPU | 0.5 | 83, 75, 83 | 80 | 6 | |
| | | 0.005 | 120, 100, 80 | 100 | 20 | |
| Barley hay | CPU | 0.05 | 58, 80, 72 | 70 | 11 | |
| | | 0.005 | 80, 80, 80 | 80 | 0 | |
| Barley straw | CPU | 0.05 | 92, 98, 96 | 95 | 3 | |
| | | 0.005 | 80, 80, 80 | 80 | 0 | |
| | | 0.05 | 88, 88, 90 | 89 | 1 | |

| Matrix | Analyte | Fortification (mg/kg) | Recoveries (%) | Mean (%) | RSD (%) | Reference |
|----------------|---------------|-----------------------|----------------|------------|---------|------------------------|
| Wheat flour | CPU | 0.005 | 100, 80, 100 | 93 | 12 | |
| | | 0.05 | 102, 108, 102 | 104 | 3 | |
| Wheat bran | CPU | 0.005 | 100, 100, 100 | 100 | 0 | |
| | | 0.05 | 84, 82, 94 | 87 | 6 | |
| Wheat germ | CPU | 0.005 | 80, 80, 80 | 80 | 0 | |
| | | 0.05 | 106, 100, 94 | 100 | 6 | |
| Wheat grain | PCA | 0.005 | 112, 112, 114 | 113 | 1 | |
| | | 0.05 | 119, 117, 116 | 117 | 2 | |
| | | 0.10 | 117, 112, 116 | 115 | 3 | |
| Wheat hay | PCA | 0.005 | 106, 106, 104 | 105 | 1 | |
| | | 0.05 | 110, 108, 108 | 109 | 1 | |
| Wheat straw | PCA | 0.005 | 94, 98, 97 | 96 | 2 | |
| | | 0.05 | 103, 113, 103 | 106 | 6 | |
| Wheat flour | PCA | 0.005 | 100, 112, 104 | 105 | 6 | |
| | | 0.05 | 100, 101, 100 | 100 | 1 | |
| Wheat bran | PCA | 0.005 | 102, 104, 100 | 102 | 2 | |
| | | 0.05 | 105, 105, 107 | 106 | 1 | |
| Wheat germ | PCA | 0.005 | 110, 102, 100 | 104 | 5 | |
| | | 0.05 | 104, 107, 103 | 105 | 2 | |
| Almond hulls | Diflubenzuron | 0.005(n = 3) | 91, 78, 96 | 88 | 10 | |
| | | 0.05(n = 3) | 83, 82, 75 | 80 | 5.3 | |
| | | 0.5(n = 3) | 82, 74, 84 | 80 | 6.4 | |
| | | 5(n = 3) | 104, 73, 78 | 85 | 20 | |
| | CPU | 0.005(n = 3) | 106, 80, 104 | 97 | 15 | |
| | | 0.01(n = 3) | 87, 100, 101 | 96 | 8 | |
| | | 0.5(n = 3) | 118, 76, 117 | 104 | 23 | |
| | PCA | 0.005(n = 3) | 74, 113, 96 | 94 | 21 | |
| | | 0.01(n = 3) | 114, 96, 99 | 103 | 9.3 | |
| 0.3(n = 3) | 118, 93, 80 | 97 | 20 | | | |
| Almond nutmeat | Diflubenzuron | 0.05(n = 2) | 76, 80 | 78 | ----- | RP-98003/ PTRL 723W |
| | | 0.1(n = 3) | 70, 80, 81 | 77 | 8 | |
| | | 0.5(n = 3) | 81, 85, 85 | 84 | 3 | |
| | CPU | 0.005(n = 3) | 84, 94, 86 | 88 | 6 | |
| | | 0.01(n = 3) | 96, 97, 95 | 96 | 1 | |
| | | 0.001(n = 3) | 97, 60, 79 | 79 | 24 | |
| | PCA | 0.005(n = 3) | 71, 88, 69 | 76 | 14 | |
| | | 0.01(n = 3) | 80, 84, 98 | 87 | 11 | |
| | Almond hulls | Diflubenzuron | 0.05(n = 3) | 84, 85, 78 | 82 | |
| 0.1(n = 3) | | | 93, 79, 85 | 86 | 8 | |
| 0.5(n = 3) | | | 78, 70, 74 | 74 | 5 | |
| CPU | | 0.01(n = 3) | 85, 98, 110 | 98 | 12 | |
| PCA | | 0.005(n = 3) | 82, 97, 100 | 93 | 10 | |
| | | 0.01(n = 3) | 102, 99, 83 | 95 | 11 | |
| Peanut nutmeat | diflubenzuron | 0.05(n = 3) | 116, 102, 106 | 108 | 7 | 2005-006/ PR07737 |
| | | 0.5(n = 3) | 85, 84, 85 | 85 | 1 | |
| | | 1.0(n = 3) | 98, 97, 81 | 92 | 10 | |
| | CPU | 0.005(n = 3) | 80, 100, 100 | 93 | 12 | |
| | | 0.05(n = 3) | 98, 100, 96 | 98 | 2 | |
| | | 0.1(n = 3) | 93, 91, 92 | 92 | 1 | |
| | PCA | 0.005(n = 3) | 104, 120, 100 | 108 | 10 | |
| | | 0.05(n = 3) | 112, 113, 111 | 112 | 1 | |
| | | 0.1(n = 3) | 112, 115, 110 | 112 | 2 | |
| Peanut hay | diflubenzuron | 0.5(n = 3) | 89, 88, 91 | 89 | 2 | |
| | | 1.0(n = 3) | 104, 103, 98 | 102 | 43 | |
| | | 20(n = 3) | 87, 85, 92 | 88 | | |
| | CPU | 0.01(n = 3) | 50, 100, 110 | 83 | 35 | |
| | | 0.1(n = 3) | 93, 93, 100 | 95 | 4 | |
| | PCA | 0.005(n = 3) | 108, 106, 110 | 108 | 2 | |
| | | 0.05(n = 3) | 112, 100, 115 | 109 | 7 | |
| | | 0.1(n = 3) | 103, 118, 96 | 106 | 11 | |
| Peanut meal | diflubenzuron | 0.5(n = 3) | 79, 78, 82 | 80 | 3 | |
| | | 1.0(n = 3) | 85, 79, 84 | 83 | 4 | |

| Matrix | Analyte | Fortification (mg/kg) | Recoveries (%) | Mean (%) | RSD (%) | Reference |
|------------|---------------|-----------------------|----------------|----------|---------|-----------|
| | CPU | 0.02(n = 3) | 95, 95, 95 | 95 | 0 | |
| | | 0.05(n = 3) | 92, 90, 78 | 87 | 9 | |
| | | 0.1(n = 3) | 86, 72, 92 | 83 | 12 | |
| | PCA | 0.005(n = 3) | 94, 94, 90 | 93 | 2 | |
| | | 0.05(n = 3) | 104, 103, 98 | 102 | 3 | |
| | | 0.1(n = 3) | 102, 104, 105 | 104 | 1 | |
| Peanut oil | diflubenzuron | 0.05(n = 3) | 84, 80, 78 | 81 | 4 | |
| | | 0.5(n = 3) | 77, 79, 79 | 78 | 1 | |
| | | 1.0(n = 3) | 81, 83, 81 | 82 | 1 | |
| | CPU | 0.005(n = 3) | 100, 100, 80 | 93 | 12 | |
| | | 0.05(n = 3) | 74, 86, 78 | 79 | 8 | |
| | | 0.1(n = 3) | 84, 93, 48 | 75 | 32 | |
| | PCA | 0.005(n = 3) | 96, 96, 102 | 98 | 4 | |
| | | 0.05(n = 3) | 102, 106, 104 | 104 | 2 | |
| | | 0.1(n = 3) | 98, 111, 101 | 103 | 7 | |

STORAGE STABILITY TESTS

Analytical issues with studies on the stability of diflubenzuron in rice, lettuce, turnip root and wheat commodities did not enable the 2002 JMPR to determine whether or not residues declined on frozen storage. Studies with peppers demonstrated residues of diflubenzuron are stable for at least 12 months frozen storage.

The present meeting received the additional storage stability of residue samples from trials on almonds, peanut, peach, pepper, wheat matrices. The lowest freezer temperature was -24 °C. The average freezer temperature was -18 °C. All of the storage stability trial results are summarized in Table 2. The results indicate that under the frozen conditions of storage, residues of diflubenzuron was stable over the study period in matrices of peaches, mustard green, peppers, barley, wheat, almonds and peanuts.

Table 2 Frozen storage stability of diflubenzuron, CPU and PCA in various matrices

| Analyte | Fortification level (mg/kg) | Storage interval (days) | Procedural recovery (%) | % remaining | Mean %remaining | Reference |
|---------------|-----------------------------|-------------------------|-------------------------|-------------|-----------------|-----------|
| Peach | | | | | | RP-00012 |
| Diflubenzuron | 0.1 | 0 | 77, 68 | - | - | |
| | | 33 | 81, 99 | 81, 71 | 76 | |
| | | 95 | 81, 99 | 39, 58 | 49 | |
| | | 209 | 80, 79 | 72, 75 | 74 | |
| | | 297 | 57, 64 | 61, 57 | 59 | |
| | | 411 | 72, 81 | 74, 65 | 69 | |
| CPU | 0.1 | 0 | 63, 64 | - | - | |
| | | 34 | 74, 73 | 47, 63 | 55 | |
| | | 103 | 104, 103 | 68, 69 | 69 | |
| | | 189 | 85, 86 | 66, 68 | 67 | |
| | | 279 | 67, 72 | 52, 55 | 54 | |
| | | 391 | 63, 66 | 61, 64 | 63 | |
| PCA | 0.1 | 0 | 109, 106 | - | - | |
| | | 35 | 103, 106 | 17, 12 | 15 | |
| | | 103 | 103, 105 | 10, 11 | 11 | |
| | | 187 | 107, 108 | 17, 17 | 17 | |
| | | 265 | 109, 109 | 13, 12 | 13 | |
| | | 376 | 102, 103 | 12, 12 | 12 | |
| Mustard green | | | | | | |
| Diflubenzuron | 0.5 | 422 | 88 | 75, 72 | 74 | |
| CPU | 0.1 | 520 | 132 | 120, 114 | 117 | |
| Peppers | | | | | | RP-97016 |
| Diflubenzuron | 0.1 | 0 | 73, 70 | - | - | |
| | | 23 | 117, 137 | 138, 131 | 135 | |
| | | 86 | 77, 77 | 72, 63, 74 | 70 | |

| Analyte | Fortification level (mg/kg) | Storage interval (days) | Procedural recovery (%) | % remaining | Mean %remaining | Reference |
|---------------|-----------------------------|-------------------------|-------------------------|-------------|-----------------|----------------------|
| | | 174 | 80, 78 | 81, 76, 81 | 79 | |
| | | 369 | 81, 87 | 74, 84, 79 | 79 | |
| CPU | 0.1 | 0 | 94, 89 | - | - | |
| | | 31 | 71 | 63, 65 | 64 | |
| | | 89 | 109, 103 | 78, 69, 84 | 77 | |
| | | 186 | 91, 82 | 64, 66, 66 | 65 | |
| | | 367 | 69, 83 | 55, 57, 54 | 55 | |
| PCA | 0.1 | 0 | 96, 73 | - | - | |
| | | 32 | 92, 97 | 57, 59, 53 | 56 | |
| | | 90 | 101, 101 | 46, 46, 52 | 48 | |
| | | 183 | 75, 76 | 32, 31, 31 | 31 | |
| | | 366 | 87, 87 | 18, 20, 19 | 19 | |
| Wheat flour | | | | | | 2006-050 2008-002 |
| Diflubenzuron | 0.5 | 0 | 83, 92, 100 | - | - | |
| | | 30 | 94, 88 | 88, 84 | 86 | |
| | | 90 | 104, 112 | 81, 83 | 82 | |
| | | 180 | 92, 97 | 89, 86 | 88 | |
| CPU | 0.05 | 0 | 80, 80, 80 | - | - | |
| | | 30 | 98, 100 | 38, 44 | 41 | |
| | | 90 | 64, 72 | 28, 26 | 27 | |
| | | 180 | 72, 62 | 22, 34 | 23 | |
| PCA | 0.05 | 0 | 100, 104, 106 | - | - | |
| | | 30 | 102, 104 | 94, 92 | 93 | |
| | | 90 | 104, 106 | 90, 90 | 90 | |
| | | 180 | 112, 110 | 80, 84 | 82 | |
| Wheat germ | | | | | | |
| Diflubenzuron | 0.5 | 0 | 95, 92, 94 | - | - | |
| | | 90 | 90, 86 | 74, 62 | 68 | |
| | | 180 | 107, 88 | 111, 110 | 111 | |
| | | 365 | 77, 79 | 62, 70 | 66 | |
| CPU | 0.05 | 0 | 82, 78, 74 | - | - | |
| | | 90 | 76, 96 | 48, 52 | 50 | |
| | | 180 | 88, 82 | 36, 40 | 38 | |
| | | 240 | 94, 96 | 32, 30 | 31 | |
| PCA | 0.05 | 0 | 116, 110, 110 | - | - | |
| | | 90 | 108, 104 | 82, 82 | 82 | |
| | | 180 | 116, 114 | 72, 72 | 72 | |
| | | 365 | 110, 114 | 58, 60 | 59 | |
| Barley grain | | | | | | PR 08024 |
| Diflubenzuron | 0.5 | 0 | 108,106 | - | - | |
| | | 182 | 96,99 | 94, 81 | 88 | |
| | | 296 | 86,86 | 108, 102 | 105 | |
| CPU | 0.5 | 0 | 80,100 | - | - | |
| | | 195 | 67,71 | 67, 87 | 77 | |
| | | 348 | 80,80 | 74, 96 | 85 | |
| PCA | 0.05 | 0 | 108,106 | - | - | |
| | | 118 | 85,89 | 37, 57 | 47 | |
| | | 293 | 106,102 | 31, 35 | 33 | |
| Barley straw | | | | | | |
| Diflubenzuron | 0.5 | 0 | 84,76 | - | - | |
| | | 134 | 72,74 | 100, 108 | 104 | |
| | | 301 | 81,61 | 117, 118 | 118 | |
| CPU | 0.5 | 0 | 120,120 | - | - | |
| | | 233 | 77,104 | 44, 74 | 59 | |
| | | 299 | 60,90 | 19, 49 | 34 | |
| PCA | 0.05 | 0 | 108,110 | - | - | |
| | | 168 | 104,108 | 88, 87 | 88 | |
| | | 302 | 102,108 | 88, 91 | 90 | |
| Wheat forage | | | | | | |
| Diflubenzuron | 0.5 | 0 | 98,84 | - | - | |
| | | 245 | 84,99 | 78, 88 | 83 | |
| | | 422 | 71,64 | 79, 99 | 89 | |

Diflubenzuron

| Analyte | Fortification level (mg/kg) | Storage interval (days) | Procedural recovery (%) | % remaining | Mean %remaining | Reference | |
|----------------|-----------------------------|-------------------------|-------------------------|-------------|-----------------|-----------|--|
| CPU | 0.5 | 0 | 81,84 | - | - | | |
| | | 267 | 105,101 | 90,78 | 84 | | |
| PCA | 0.05 | 0 | 98,100 | - | - | | |
| | | 194 | 104,109 | 50,56 | 53 | | |
| | | 345 | 100,100 | 38,40 | 39 | | |
| Wheat hay | | | | | | | |
| Diflubenzuron | 0.5 | 0 | 98,84 | - | - | | |
| | | 140 | 85,86 | 105,99 | 102 | | |
| | | 337 | 80,71 | 105,101 | 103 | | |
| CPU | 0.5 | 0 | 100,101 | - | - | | |
| | | 251 | 80,85 | 61,69 | 65 | | |
| | | 355 | 80,80 | 26,34 | 30 | | |
| PCA | 0.05 | 0 | 102,106 | - | - | | |
| | | 212 | 104,107 | 67,70 | 69 | | |
| | | 359 | 110,104 | 75,77 | 76 | | |
| Almond nutmeat | | | | | | | |
| Diflubenzuron | 0.1 | 0 | 84,87 | - | - | RP-00010 | |
| | | 32 | 79,84 | 79,76 | 78 | | |
| | | 103 | 85,91 | 81,78 | 80 | | |
| | | 180 | 81,82 | 76,88 | 82 | | |
| | | 321 | 87,84 | 81,86 | 84 | | |
| | | 379 | 87,92 | 78,69 | 74 | | |
| CPU | 0.1 | 0 | 66,74 | - | - | | |
| | | 34 | 79,83 | 63,64 | 64 | | |
| | | 110 | 77,79 | 68,73 | 71 | | |
| | | 208 | 101,82 | 86,60 | 73 | | |
| | | 278 | 88,77 | 83,82 | 83 | | |
| | | 378 | 68,77 | 77,61 | 69 | | |
| PCA | 0.1 | 0 | 111,116 | - | - | | |
| | | 34 | 104,108 | 103,102 | 103 | | |
| | | 110 | 104,103 | 81,81 | 81 | | |
| | | 182 | 104,105 | 90,89 | 90 | | |
| | | 273 | 107,108 | 86,90 | 88 | | |
| | | 384 | 99,104 | 86,80 | 83 | | |
| Almond hulls | | | | | | | |
| Diflubenzuron | 0.1 | 0 | 77,93 | - | - | | |
| | | 32 | 76,86 | 80,75 | 78 | | |
| | | 103 | 77,90 | 60,57 | 59 | | |
| | | 180 | 97,91 | 77,76 | 77 | | |
| | | 321 | 89,92 | 75,75 | 75 | | |
| | | 379 | 95,98 | 75,79 | 77 | | |
| CPU | 0.1 | 0 | 65,62 | - | - | | |
| | | 34 | 87,85 | 59,70 | 65 | | |
| | | 130 | 74,70 | 79,30 | 55 | | |
| | | 208 | 48,57 | 55,62 | 58 | | |
| | | 278 | 36,50 | 30,28 | 29 | | |
| | | 378 | 47,39 | 19,42 | 30 | | |
| PCA | 0.1 | 0 | 104,106 | - | - | | |
| | | 34 | 105,105 | 92,86 | 89 | | |
| | | 110 | 100,101 | 63,61 | 62 | | |
| | | 182 | 101,101 | 62,63 | 63 | | |
| | | 273 | 108,109 | 69,68 | 69 | | |
| | | 384 | 104,104 | 55,67 | 61 | | |
| Peanut nutmeat | | | | | | | |
| Diflubenzuron | 0.5 | 295 | 75,69 | 65,68 | 67 | PR 07737 | |
| CPU | 0.5 | 421 | 79,84 | 54,53 | 54 | | |
| PCA | 0.1 | 298 | 70,71 | 41,39 | 40 | | |
| Peanut hay | | | | | | | |
| Diflubenzuron | 0.5 | 356 | 95,103 | 83,79 | 81 | | |
| CPU | 0.5 | 484 | 88,108 | 21,35 | 38 | | |
| PCA | 0.1 | 338 | 102,99 | 77,76 | 76 | | |
| Peanut meal | | | | | | | |

| Analyte | Fortification level (mg/kg) | Storage interval (days) | Procedural recovery (%) | % remaining | Mean %remaining | Reference | |
|--------------------|-----------------------------|-------------------------|-------------------------|-------------|-----------------|-----------|--|
| Diflubenzuron | 0.5 | 643 | 96,86 | 84, 65 | 75 | | |
| CPU | 0.5 | 645 | 110,106 | 90, 114 | 102 | | |
| PCA | 0.1 | 488 | 108,106 | 91, 88 | 90 | | |
| Peanut refined oil | | | | | | | |
| Diflubenzuron | 0.5 | 365 | 115,113 | 114, 117 | 115 | | |
| CPU | 0.5 | 294 | 91,81 | 82, 87 | 85 | | |
| PCA | 0.1 | 286 | 68,71 | 65, 65 | 65 | | |

USE PATTERN

Diflubenzuron is effective on a wide variety of insect pests, predominantly from the families Lepidoptera (caterpillars) and Diptera (flies). It is useful in controlling worms (e.g., fall webworm, armyworm), weevils (e.g., rice water weevil, cotton boll weevil), beetles (e.g., oat leaf beetle, Colorado potato beetle), Psylla species, moths (e.g., codling moth, winter moth), leaf-feeding larvae, leaf miners, rust mites, larvae of sciarid and phorid flies, maize stalk borers, tortrix, earwigs, cabbage white, leafrollers, grasshoppers, aphids, and fruit flies.

All supervised trials were conducted in the United States. Table 3 summarizes the registered uses of diflubenzuron.

Table 3 Registered use of diflubenzuron in the USA

| Crop | Formulation (g ai/L or g ai/kg) | Application | | | | PHI Days |
|-----------------------------|---------------------------------|-------------|-------------------|-------------|-----------------------|----------|
| | | kg ai/ha | Spray volume L/ha | kg ai/ha | Max. No. (per season) | |
| Tree nuts ^a | 2L (240 g ai/L) | 0.14–0.280 | 468–954 | 0.029–0.060 | 4 (3 for walnut) | 28 |
| Leafy brassica ^b | 2L (240 g ai/L) | 0.035–0.070 | 281 | 0.025 | 4 | 7 |
| Pepper | 25W (250 g ai/kg) | 0.070–0.140 | 281 | 0.025 | 5 | 7 |
| | 2L (240 g ai/L) | 0.070–0.140 | 281 | 0.025 | 5 | 7 |
| Stonefruit ^c | 2L (240 g ai/L) | 0.140–0.280 | 468–935 | 0.030–0.060 | 2 | 14 |
| Peanut | 2L (240 g ai/L) | 0.035–0.140 | 84–327 | 0.042–0.166 | 3 | 28 |
| Barley | 2L (240 g ai/L) | 0.035–0.070 | 47–140 | 0.050–0.150 | 1 | 50 |
| Wheat | 2L (240 g ai/L) | 0.035–0.070 | 47–140 | 0.050–0.150 | 1 | 50 |
| Oats | 2L (240 g ai/L) | 0.035–0.070 | 47–140 | 0.050–0.150 | 1 | 50 |
| Triticale | 2L (240 g ai/L) | 0.035–0.070 | 47–140 | 0.050–0.150 | 1 | 50 |

^a Tree nuts include almonds, pecans, walnuts, breach nuts, brazil nuts, butter nuts, chestnuts, chinquapin, bush nuts, hazelnuts, hickory nuts.

^b Leafy brassica includes mustard greens, broccoli raab, cabbage, collards, kales, mizunas, mustard spinach, rape greens.

^c Stonefruit includes peaches, plums, nectarines, apricots, prunes.

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

Supervised trial studies on peaches, plums, mustard greens, pepper, barley and wheat as well as almond and pecan were made available to the Meeting. The residue trials were conducted in the United States.

| Crop Group or Subgroup | Commodity | Table No. |
|------------------------|---------------|---------------|
| Stone fruit | Peach, plum | Table 4,5 |
| Leafy vegetables | Mustard green | Table 6 |
| Fruiting vegetables | Pepper | Table 7 |
| Cereal grains | Barley, wheat | Table 8,12,13 |
| Tree nut | Almond, pecan | Table 9,10,14 |
| Oilseed | Peanut | Table 11,15 |

Although all trials included control plots, no control data are recorded in the summary tables unless residues in control samples exceeded the LOQ. Results reported have not been corrected for concurrent method recoveries unless indicated.

In the trials, where multiple samples were taken from a single plot, the mean residue value which is underlined in the following table is selected for the estimation of the MRL and STMR. Where results from separate plots with distinguishing characteristics such as different formulations, varieties or treatment schedules were reported, results are listed for each plot.

Residues and application rates have generally been rounded to two significant figures. Residue values from the trials conducted according to the $\pm 25\%$ of maximum GAP has been used for the estimation of maximum residue levels.

Stone Fruit

Peach and Plum

A study was conducted in 2005 in the USA on peach and plum. The field portion of this study was conducted at eight field sites representative of peach and plum growing areas in the USA. Each trial site included one untreated (control) plot and one treated plot, to which diflubenzuron (2L) was applied twice by air-blast sprayer. The test substance was mixed with water and 0.25% v/v crop oil and applied to peaches or plums. The second application to each treated plot was made approximately 14 days after the first, and at the same rate. Tables 4 and 5 summarise the peach and plum residue results.

Table 4 Residues of Diflubenzuron, CPU, and PCA in peaches

| Location, year (variety) | Application | | | | | PHI days | Residues, mg/kg | | | Reference |
|---|-------------|--------------|----------------|-------------------|-----|-------------|--|----------------------------|-------------------------------|-----------|
| | Form | kg ai/ha | kg ai/hL | Interval, days | no. | | DFB | CPU | PCA | |
| Athens USA/GA 2005 Contender | 240 SC | 0.28 0.28 | 0.032 0.032 | 14 | 2 | 14 | 0.42 0.17 0.095 <u>Av 0.23</u> | < 0.01 < 0.01 < 0.01 | < 0.005 < 0.005 < 0.005 | 2005-005 |
| Orfield USA/PA 2005 Garnet Beauty | 240 SC | 0.29 0.28 | 0.046 0.046 | 14 | 2 | 14 | 0.13 0.11 0.27 <u>Av 0.17</u> | < 0.01 < 0.01 < 0.01 | < 0.005 < 0.005 < 0.005 | 2005-005 |
| Sultana USA/CA 2005 September Delight | 240 SC | 0.28 0.28 | 0.037 0.044 | 15 | 2 | 13 | 0.069 0.20 0.33 <u>Av 0.20</u> | < 0.01 < 0.01 < 0.01 | < 0.005 < 0.005 < 0.005 | 2005-005 |
| Hughson USA/CA 2005 Fairtime | 240 SC | 0.28 0.28 | 0.040 0.039 | 14 | 2 | 14 | 0.25 0.22 0.15 <u>Av 0.21</u> | < 0.01 < 0.01 < 0.01 | < 0.005 < 0.005 < 0.005 | 2005-005 |
| Waller USA/TX 2005 Texas Royal | 240 SC | 0.29 0.28 | 0.051 0.048 | 14 | 2 | 14 | 0.064 0.084 0.21 <u>Av 0.12</u> | < 0.01 < 0.01 < 0.01 | < 0.005 < 0.005 < 0.005 | 2005-005 |

Table 5 Residues of Diflubenzuron, CPU, and PCA in plums

| Location, year (variety) | Application | | | | | PHI days | Residues, mg/kg | | | Reference |
|--------------------------------------|-------------|--------------|----------------|-------------------|-----|-------------|--|------------------|-------------------------------|-----------|
| | Form | kg ai/ha | kg ai/hL | Interval, days | no. | | DFB | CPU | PCA | |
| Conklin USA/MI 2005 Stanley | 240 SC | 0.28 0.28 | 0.042 0.041 | 14 | 2 | 14 | 0.15 0.16 0.19 <u>Av 0.17</u> | < 0.01 < 0.01 | < 0.005 < 0.005 < 0.005 | 2005-005 |

| Location, year (variety) | Application | | | | | PHI days | Residues, mg/kg | | | Reference |
|-------------------------------------|-------------|--------------|----------------|-------------------|-----|-------------|--|------------------|-------------------------------|-----------|
| | Form | kg ai/ha | kg ai/hL | Interval, days | no. | | DFB | CPU | PCA | |
| Live USA/CA 2005 French | 240 SC | 0.28 0.28 | 0.040 0.040 | 14 | 2 | 14 | 0.076 0.10 0.070 <u>Av 0.08</u> | < 0.01 < 0.01 | < 0.005 < 0.005 < 0.005 | 2005-005 |
| Zillah USA/WA 2005 Italian | 240 SC | 0.27 0.28 | 0.033 0.033 | 15 | 2 | 14 | 0.12 0.20 0.18 <u>Av 0.17</u> | < 0.01 < 0.01 | < 0.005 0.020 0.021 | 2005-005 |

Mustard Green

Eight field trials on mustard greens were conducted in the USA during the 2001 growing season. At each location, diflubenzuron was applied four times (except for one trial site where only three treatments were made) as broadcast foliar applications using ground equipment. Treatments were made during the crop's vegetative growth stage at a retreatment interval of 8–15 days. Duplicate control and treated samples of mature mustard greens were harvested from each site at 6–8 days after the last treatment (DAT). Mustard green samples were stored frozen prior to analysis at the analytical laboratory.

Table 6 Residues of Diflubenzuron, CPU, and PCA in/on mustard green

| Location Year Variety | Application | | | | | PHI days | Residues, mg/kg | | | Reference |
|---------------------------------------|-------------|----------|---------------|----------|-----|-------------|---|------------------|--------------------|------------------------|
| | Form | kg ai/ha | Water L/ha | kg ai/hL | no. | | DFB | CPU | PCA | |
| Weslaco USA/TX 2001 TX27 | 240 SC | 0.073 | 281– 393 | 0.026 | 4 | 6 | 1.0 1.2 <u>Av 1.1</u> | < 0.01 < 0.01 | < 0.005 < 0.005 | PR08031/ 2005-059 |
| Tifton USA/GA 2001 GA20 | 240 SC | 0.072 | 187 | 0.038 | 3 | 7 | 0.82 2.2 <u>Av 1.5</u> | < 0.01 < 0.01 | < 0.005 < 0.005 | PR# 08031/ 2005-059 |
| Tifton USA/GA 2001 GA21 | 240 SC | 0.072 | 187 | 0.038 | 4 | 8 | 1.9 3.1 <u>Av 2.5</u> | < 0.01 < 0.01 | < 0.005 < 0.005 | PR# 08031/ 2005-059 |
| Salina USA/CA 2001 CA76 | 240 SC | 0.074 | 655– 842 | 0.011 | 4 | 6 | 1.1 0.91 <u>Av 1.0</u> | < 0.01 < 0.01 | < 0.005 < 0.005 | PR# 08031/ 2005-059 |
| Salina UAS/CA 2001 CA77 | 240 SC | 0.073 | 318– 692 | 0.023 | 4 | 6 | < 0.05 < 0.05 <u>Av < 0.05</u> | < 0.01 < 0.01 | < 0.005 < 0.005 | PR# 08031/ 2005-059 |
| Celeryville USA/OH 2001 OH19 | 240 SC | 0.073 | 468– 477 | 0.016 | 4 | 7 | 1.2 1.2 <u>Av 1.2</u> | < 0.01 < 0.01 | < 0.005 < 0.005 | PR# 08031/ 2005-059 |
| Crossville USA/TN 2001 TN11 | 240 SC | 0.072 | 215 – 224 | 0.033 | 4 | 7 | 1.3 2.9 <u>Av 2.1</u> | < 0.01 < 0.01 | < 0.005 < 0.005 | PR# 08031/ 2005-059 |
| Gainesville USA/FL 2001 FL37 | 240 SC | 0.072 | 281 | 0.026 | 4 | 7 | 7.1 6.6 <u>Av 6.8</u> | 0.018 0.019 | < 0.005 < 0.005 | PR# 08031/ 2005-059 |

Pepper

Nine residue trial were conducted on bell and non-bell (chilli) peppers in the USA in 1997. The number of trials and geographic representation is adequate for peppers. Five foliar applications of the diflubenzuron 25 WP (250 g ai/kg) formulation were made. The applications were made at 7-day intervals using a backpack, self-propelled, or tractor mounted equipment. At harvest, seven days after the last application, peppers were handpicked from each plot in a random pattern. Two samples were collected from each field plot. The samples were frozen and shipped to the analytical laboratory for analysis.

A further two trials were conducted in 2003 also in the USA (CA and FL) using diflubenzuron 2L (240 SC) formulation. In each site, two plots were established, with one subplot treated with the formulation only, while the other subplot treated with the formulation plus a crop oil (0.25% paraffinic oil). Three applications were made using boom sprayers. Three samples were collected 7 days after the last application from each subplot. The samples were frozen and shipped to the analytical laboratory for analysis.

Table 7 Residues of Diflubenzuron, CPU, and PCA in/on bell/non-bell peppers

| Location, year variety | Application | | | | | PHI days | Residues, mg/kg | | | Reference |
|---|--|----------|---------------|----------|-----|-------------|-----------------------------------|-------------------------------|-------------------------------|-----------|
| | Form | kg ai/ha | Water L/ha | kg ai/hL | no. | | DFB | CPU | PCA | |
| Sweet (Bell) Pepper | | | | | | | | | | |
| Lodi USA/CA 1997 Bomby | 250 WP | 5×0.14 | 5×281 | 5×0.050 | 5 | 7 | 0.23 0.25 <u>Av 0.24</u> | < 0.01 < 0.01 | < 0.005 < 0.005 | RP-97016 |
| Noblesville USA/IN 1997 California Wonder | 250 WP | 5×0.14 | 5×281 | 5×0.050 | 5 | 7 | 0.21 0.26 <u>Av 0.24</u> | 0.013 0.014 | < 0.005 < 0.005 | RP-97016 |
| Knightdale USA/NC 1997 Yolo Wonder | 250 WP | 5×0.14 | 5×281 | 5×0.050 | 5 | 7 | 0.050 0.093 <u>Av 0.07</u> | < 0.01 0.010 | < 0.005 < 0.005 | RP-97016 |
| San Marcos USA/CA 1997 Jupiter | 250 WP | 5×0.14 | 5×281 | 5×0.050 | 5 | 7 | < 0.05 0.098 <u>Av 0.07</u> | < 0.01 0.038 | < 0.005 < 0.005 | RP-97016 |
| Immokalee USA/FL 1997 California Wonder | 250 WP | 5×0.14 | 5×281 | 5×0.050 | 5 | 7 | < 0.05 0.012 <u>Av 0.08</u> | < 0.01 < 0.01 | < 0.005 < 0.005 | RP-97016 |
| Donna USA/TX 1997 Jupiter | 250 WP | 5×0.14 | 5×281 | 5×0.050 | 5 | 7 | 0.33 0.33 <u>Av 0.33</u> | 0.027 0.057 | < 0.005 < 0.005 | RP-97016 |
| Fresno USA/CA 2003 Orion | 240 SC | 3×0.14 | 3×281 | 3×0.050 | 3 | 7 | 0.088 0.094 0.092 | < 0.005 < 0.005 < 0.005 | < 0.005 < 0.005 < 0.005 | RP-03007 |
| | 240 SC, +0.25% paraffinic oil | 3×0.14 | 3×281 | 3×0.050 | 3 | 7 | 0.071 0.065 0.079 | < 0.005 < 0.005 < 0.005 | < 0.005 < 0.005 < 0.005 | |
| Oveido USA/ FL 2003 Crusader | 240 SC | 3×0.14 | 3×281 | 3×0.050 | 3 | 7 | 0.070 0.067 0.071 | < 0.005 < 0.005 < 0.005 | < 0.005 < 0.005 < 0.005 | |

| Location, year variety | Application | | | | | PHI days | Residues, mg/kg | | | Reference |
|--------------------------------|-------------------------------|----------|------------|----------|-----|----------|--------------------------------|-------------------------------|-------------------------------|-----------|
| | Form | kg ai/ha | Water L/ha | kg ai/hL | no. | | DFB | CPU | PCA | |
| | 240 SC, +0.25% paraffinic oil | 3×0.14 | 3×281 | 3×0.050 | 3 | 7 | 0.137 0.142 0.081 | < 0.005 < 0.005 < 0.005 | < 0.005 < 0.005 < 0.005 | RP-03007 |
| Chilli (non-bell) Pepper | | | | | | | | | | |
| Bernard USA/TX 1997 Anaheim | 250 WP | 5×0.14 | 5×281 | 5×0.050 | 5 | 7 | 0.90 0.94 <u>Av 0.92</u> | < 0.01 0.057 | < 0.005 < 0.005 | RP-97016 |
| Rincon USA/NM 1997 Anaheim | 250 WP | 5×0.14 | 5×281 | 5×0.050 | 5 | 7 | 0.92 0.95 <u>Av 0.94</u> | < 0.01 < 0.01 | < 0.005 < 0.005 | RP-97016 |
| San Marcos USA/CA 1997 Anaheim | 250 WP | 5×0.14 | 5×281 | 5×0.050 | 5 | 7 | 0.21 0.29 <u>Av 0.25</u> | < 0.01 < 0.01 | < 0.005 < 0.005 | RP-97016 |

Cereal grains

Seven field trials on barley (two winter and five spring varieties) and three trials on wheat (one winter and two spring varieties) were conducted in the USA between the 2002 and 2003 growing seasons. At each location, diflubenzuron was applied once to barley and wheat fields as a broadcast foliar application during crop development (pre-boot, pre-stem elongation, jointing, or Feekes 8 growth stage). A single control and duplicate treated samples of mature grain and straw were harvested from each site at 50–76 days after treatment (DAT). Hay was harvested from each site at 15–39 DAT, and wheat forage was harvested at 3–12 DAT.

Two samples of wheat grain, straw, and hay/forage was taken from each untreated and treated plot at 45 days PHI. Hay/forage and straw samples were randomly hand-picked while the grain was sampled using a commercial combine. Hay/forage was sampled at the milk to soft dough stage. The sampled crop could be used as forage or dried as hay. Immediately after sampling, the samples were placed in coolers containing an ice substitute and transported to an off-site facility where they were stored frozen until being shipped to the analytical laboratory, where the samples remained frozen until analysis.

Table 8 Residues of Diflubenzuron, CPU, and PCA in barley/wheat grain

| Location, year variety | Application | | | | | PHI days | Residues, mg/kg | | | Reference |
|-----------------------------------|-------------|----------|------------|----------|-----|----------|---|--------------------|--------------------|-----------|
| | Form | kg ai/ha | Water L/ha | kg ai/hL | no. | | DFB | CPU | PCA | |
| Barley Grain | | | | | | | | | | |
| Kimberly USA/ID 2002 Eight Twelve | 240 SC | 0.066 | 114 | 0.058 | 1 | 76 | < 0.05 < 0.05 <u>Av < 0.05</u> | < 0.005 < 0.005 | < 0.005 < 0.005 | PR 08024 |
| Aberdeen USA/ID 2002, (Gallatin) | 240 SC | 0.069 | 140 | 0.050 | 1 | 71 | < 0.05 < 0.05 <u>Av < 0.05</u> | < 0.005 < 0.005 | < 0.005 < 0.005 | PR 08024 |
| Minot USA/ND 2003 Robust | 240 SC | 0.070 | 95 | 0.074 | 1 | 55 | < 0.05 < 0.05 <u>Av < 0.05</u> | < 0.005 < 0.005 | < 0.005 < 0.005 | PR 08024* |
| Fargo USA/ND 2003 Robust | 240 SC | 0.070 | 112 | 0.062 | 1 | 54 | < 0.05 < 0.05 <u>Av < 0.05</u> | < 0.005 < 0.005 | < 0.005 < 0.005 | PR 08024 |

| Location, year variety | Application | | | | | PHI days | Residues, mg/kg | | | Reference |
|--|-------------|----------|------------|----------|-----|----------|-------------------------------|--------------------|--------------------|-----------|
| | Form | kg ai/ha | Water L/ha | kg ai/hL | no. | | DFB | CPU | PCA | |
| Minot USA/ND 2003 Robust | 240 SC | 0.069 | 93 | 0.074 | 1 | 54 | < 0.05 < 0.05 Av < 0.05 | < 0.005 < 0.005 | < 0.005 < 0.005 | PR 08024* |
| Fort Collins USA/CO 2003 (Moravian 37) | 240 SC | 0.069 | 111 | 0.062 | 1 | 75 | < 0.05 < 0.05 Av < 0.05 | < 0.005 < 0.005 | < 0.005 < 0.005 | PR 08024 |
| Velva USA/ND 2003 (Foster) | 240 SC | 0.070 | 185 | 0.038 | 1 | 50 | < 0.05 < 0.05 Av < 0.05 | < 0.005 < 0.005 | < 0.005 < 0.005 | PR 08024 |
| Wheat Grain | | | | | | | | | | |
| Kimberly USA/ID 2003 (Brundage) | 240 SC | 0.072 | 120 | 0.060 | 1 | 76 | < 0.05 < 0.05 Av < 0.05 | < 0.005 < 0.005 | < 0.005 < 0.005 | PR 08024 |
| Fargo USA/ND 2003 (Oxen) | 240 SC | 0.069 | 111 | 0.062 | 1 | 56 | < 0.05 < 0.05 Av < 0.05 | < 0.005 < 0.005 | < 0.005 < 0.005 | PR 08024 |
| Minto USA/ND 2003 (Mountrail) | 240 SC | 0.069 | 94 | 0.074 | 1 | 62 | < 0.05 < 0.05 Av < 0.05 | < 0.005 < 0.005 | < 0.005 < 0.005 | PR 08024 |

*: Trials were conducted in the same location with deferent application times.

Almonds and Pecans

Almond trials conducted in 1998 (RP-98003) were reviewed previously by the 2002 JMPR and are summarized in the table below.

In 2003, a further two almond trials (RP-03001) were conducted in the USA with Dimilin 240 SC (2L). Two subplots were set up for each trial, one with 0.25% crop oil, the other without. Four foliar applications were made, with a PHI of 28 days.

The 2002 JMPR also reviewed trials on pecans conducted in 1999 (RP-99002) in the USA which are also summarised below.

Table 9 Residues of Diflubenzuron, CPU, and PCA in/on almond nutmeat (kernels)

| Location, year (variety) | Application | | | | | PHI days | Residues, mg/kg | | | Reference |
|------------------------------|-------------|----------|----------|------------|-----|----------|--------------------------------|---------------------|---------------------|-----------|
| | Form | kg ai/ha | kg ai/hL | Water L/ha | no. | | DFB | CPU | PCA | |
| Madera USA/CA1998 Nonpareil | 250 WP | 0.56 | 0.06 | 935 | 4 | 28 | < 0.05, < 0.05 Av < 0.05 | < 0.005, < 0.005 | < 0.005, < 0.005 | RP-98003 |
| | | 0.28 | 0.06 | 467 | | | | | | |
| | | 0.28 | 0.06 | 467 | | | | | | |
| | | 0.56 | 0.12 | 467 | | | | | | |
| Kerman USA/CA 1998 Nonpareil | 250 WP | 0.57 | 0.06 | 935 | 4 | 28 | < 0.05, < 0.05 Av < 0.05 | < 0.005, < 0.005 | < 0.005, < 0.005 | RP-98003 |
| | | 0.27 | 0.059 | 467 | | | | | | |
| | | 0.29 | 0.061 | 467 | | | | | | |
| | | 0.56 | 0.12 | 467 | | | | | | |
| Reesley USA/CA 1998 Butte | 250 WP | 0.56 | 0.060 | 935 | 4 | 28 | < 0.05, < 0.05 Av < 0.05 | < 0.005, < 0.005 | < 0.005, < 0.005 | RP-98003 |
| | | 0.28 | 0.060 | 467 | | | | | | |
| | | 0.28 | 0.061 | 467 | | | | | | |
| | | 0.56 | 0.12 | 467 | | | | | | |
| | 800 WG | 0.57 | 0.060 | 935 | 4 | 28 | < 0.05 < 0.05 Av < 0.05 | < 0.005, < 0.005 | < 0.005, < 0.005 | |
| | | 0.28 | 0.059 | 467 | | | | | | |
| | | 0.28 | 0.060 | 467 | | | | | | |
| | | 0.56 | 0.12 | 467 | | | | | | |

| Location, year (variety) | Application | | | | | PHI days | Residues, mg/kg | | | Reference |
|---|-----------------------------|------------------------------|----------------------------------|--------------------------|-----|-------------|---|---------------------------------|---------------------------------|-----------|
| | Form | kg ai/ha | kg ai/hL | Water L/ha | no. | | DFB | CPU | PCA | |
| | 240 SC | 0.56 0.28 0.28 0.56 | 0.059 0.060 0.059 0.12 | 935 467 467 467 | 4 | 28 | < 0.05, < 0.05 Av < 0.05 | < 0.005, < 0.005 | < 0.005, < 0.005 | |
| Manteca USA/CA 1998 Nonpareil | 250 WP | 0.56 0.28 0.28 0.56 | 0.060 0.060 0.061 0.12 | 935 467 467 467 | 4 | 28 | < 0.05, < 0.05 Av < 0.05 | < 0.005, < 0.005 | < 0.005, < 0.005 | RP-98003 |
| Ripon USA/CA 1998 Nonpareil | 250 WP | 0.81 0.49 0.28 0.55 | 0.060 0.060 0.059 0.12 | 935 467 467 467 | 4 | 28 | < 0.05, < 0.05 Av < 0.05 | < 0.005, < 0.005 | < 0.005, < 0.005 | RP-98003 |
| Madera USA/CA 2003 Padre Butte | 240 SC | 0.28 0.28 0.28 0.28 | 0.029 0.060 0.060 0.060 | 954 477 468 477 | 4 | 28 | 0.029, 0.018, 0.089 Av 0.045 | < 0.005, < 0.005, < 0.005 | < 0.005, < 0.005, < 0.005 | RP-03001 |
| | 240 SC 0.25% crop oil | 0.28 0.28 0.28 0.28 | 0.029 0.060 0.060 0.060 | 944 468 468 477 | 4 | 28 | 0.033, 0.027, 0.084 Av 0.048 | < 0.005, < 0.005, < 0.005 | < 0.005, < 0.005, < 0.005 | |
| Winters USA/CA 2003 Butte | 240 SC | 0.28 0.28 0.28 0.28 | 0.032 0.062 0.062 0.062 | 888 449 449 458 | 4 | 28 | < 0.005, 0.019, 0.017 Av 0.013 | < 0.005, < 0.005, < 0.005 | < 0.005, < 0.005, < 0.005 | RP-03001 |
| | 240 SC 0.25% crop oil | 0.28 0.28 0.28 0.28 | 0.032 0.062 0.062 0.062 | 888 449 449 458 | 4 | 28 | 0.010, 0.084, < 0.005 Av 0.033 | < 0.005, < 0.005, < 0.005 | < 0.005, < 0.005, < 0.005 | |

Table 10 Residues of Diflubenzuron, CPU, and PCA in/on pecans (kernels)

| Location, year (variety) | Application | | | | | PHI days | Residues, mg/kg | | | Reference |
|--|-------------|------------------------------|---------------------------------|--------------------------|-----|-------------|-------------------------------|--------------------|--------------------|-----------|
| | Form | kg ai/ha | kg ai/hL | Water L/ha | no. | | DFB | CPU | PCA | |
| Finleyson USA/GA 1999 Stuarts | 240 SC | 0.56 0.28 0.28 0.56 | 0.060 0.058 0.062 0.12 | 935 467 467 467 | 4 | 28 | < 0.05 < 0.05 Av < 0.05 | < 0.005 < 0.005 | < 0.005 < 0.005 | RP-99002 |
| | 250 WP | 0.56 0.28 0.28 0.56 | 0.061 0.057 0.062 0.12 | 935 467 467 467 | 4 | 28 | < 0.05 < 0.05 Av < 0.05 | < 0.005 < 0.005 | < 0.005 < 0.005 | |
| | 800 WG | 0.56 0.28 0.28 0.56 | 0.061 0.057 0.062 0.12 | 935 467 467 467 | 4 | 28 | < 0.05 < 0.05 Av < 0.05 | < 0.005 < 0.005 | < 0.005 < 0.005 | |
| Eastman USA/GA 1999 Desirable | 240 SC | 0.57 0.28 0.28 0.55 | 0.061 0.059 0.061 0.12 | 935 467 467 467 | 4 | 28 | < 0.05 < 0.05 Av < 0.05 | < 0.005 < 0.005 | < 0.005 < 0.005 | RP-99002 |
| Opelousas USA/LA 1999 Melrose | 240 SC | 0.56 0.28 0.28 0.56 | 0.059 0.059 0.059 0.12 | 935 467 467 467 | 4 | 28 | < 0.05 < 0.05 Av < 0.05 | < 0.005 < 0.005 | < 0.005 < 0.005 | RP-99002 |
| | 250 WP | 0.56 0.28 0.28 0.56 | 0.059 0.059 0.059 0.12 | 935 467 467 467 | 4 | 28 | < 0.05 < 0.05 Av < 0.05 | < 0.005 < 0.005 | < 0.005 < 0.005 | |
| | 800 WG | 0.56 0.28 0.28 0.56 | 0.059 0.059 0.059 0.12 | 935 467 467 467 | 4 | 28 | < 0.05 < 0.05 Av < 0.05 | < 0.005 < 0.005 | < 0.005 < 0.005 | |

| Location, year (variety) | Application | | | | | PHI days | Residues, mg/kg | | | Reference |
|---|-------------|----------|----------|---------------|-----|-------------|---------------------|---------|---------|-----------|
| | Form | kg ai/ha | kg ai/hL | Water L/ha | no. | | DFB | CPU | PCA | |
| Ducan USA/OK 1999 Natives | 240 SC | 0.56 | 0.059 | 935 | 4 | 28 | < 0.05 | < 0.005 | < 0.005 | RP-99002 |
| | | 0.28 | 0.063 | 467 | | | < 0.05 | < 0.005 | < 0.005 | |
| | | 0.29 | 0.061 | 467 | | | <u>Av < 0.05</u> | | | |
| | | 0.56 | 0.12 | 467 | | | | | | |
| | 250 WP | 0.56 | 0.059 | 935 | 4 | 28 | < 0.05 | < 0.005 | < 0.005 | |
| | | 0.28 | 0.063 | 467 | | | < 0.05 | < 0.005 | < 0.005 | |
| | | 0.28 | 0.061 | 467 | | | Av < 0.05 | | | |
| | | 0.56 | 0.12 | 467 | | | | | | |
| 800 WG | 0.56 | 0.059 | 935 | 4 | 28 | < 0.05 | < 0.005 | < 0.005 | | |
| | 0.28 | 0.063 | 467 | | | < 0.05 | < 0.005 | < 0.005 | | |
| | 0.29 | 0.062 | 467 | | | Av < 0.05 | | | | |
| | 0.56 | 0.12 | 467 | | | | | | | |
| Ricon USA/NM 1999 Western Schley | 240 SC | 0.55 | 0.059 | 935 | 4 | 28 | < 0.05 | < 0.005 | < 0.005 | RP-99002 |
| | | 0.28 | 0.061 | 467 | | | < 0.05 | < 0.005 | < 0.005 | |
| | | 0.28 | 0.060 | 467 | | | <u>Av < 0.05</u> | | | |
| | | 0.54 | 0.12 | 467 | | | | | | |
| | | | | | | | | | | |

Peanut

Twelve peanut field trials were conducted in the USA during the 2001 growing season. At each trial location, diflubenzuron was applied three times as broadcast foliar applications using ground equipment. The first application was at first bloom, the second was 14 (\pm 1) days after the first, and the third was 28 (\pm 1) days before harvest at nine sites, 20 days at two sites and 26 days at one site. A single control and single or duplicate treated samples of peanuts and peanut hay were harvested from each site at 20–28 days after treatment (DAT). All samples were stored frozen for up to 481 days prior to residue extraction and analysis, an interval supported by available storage stability data.

Residues from the supervised field trials are summarised below, with residues according to the GAP underlined. Two sets of residue values are reported for each trial since replicate samples were taken from the same plot.

Table 11 Residues of Diflubenzuron, CPU, and PCA in/on peanut nutmeat

| Location, year (variety) | Application | | | | | PHI days | Residues, mg/kg | | | Reference |
|---|-------------|----------|---------------|------------------|-----|-------------|---|---|--------------------|-----------|
| | Form | kg ai/ha | Water L/ha | kg ai/ha | no. | | DFB | CPU | PCA | |
| Salisbury USA/MD 2001 VA C98R | 240 SC | 0.14 | 374 – 383 | 0.037– 0.037 | 3 | 28 | < 0.05 < 0.05 <u>Av < 0.05</u> | < 0.005 0.005 | < 0.005 < 0.005 | PR 07737 |
| Crossville USA/TN 2001 VA 98R | 240 SC | 0.14 | 215– 224 | 0.0675– 0.062 | 3 | 28 | < 0.05 < 0.05 <u>Av < 0.05</u> | c0.022**, c0.026**, 0.011, 0.019 | < 0.005 < 0.005 | PR 07737 |
| Crossville USA/TN 2001 VA-C92R | 240 SC | 0.14 | 215– 224 | 0.065– 0.062 | 3 | 28 | 0.059 0.060 <u>Av 0.06</u> | < 0.005 < 0.005 | < 0.005 < 0.005 | PR 07737 |
| Rocky USA/NC 2001 VA 98R | 240 SC | 0.14 | 168 | 0.083 | 3 | 28 | < 0.05 0.055 <u>Av 0.052</u> | < 0.005 < 0.005 | < 0.005 < 0.005 | PR 07737 |
| Weslaco USA/TX 2001 Florunner | 240 SC | 0.14 | 131– 159 | 0.107– 0.088 | 3 | 29 | < 0.05 < 0.05 Av < 0.05 | < 0.005 < 0.005 | < 0.005 < 0.005 | PR 07737* |
| Weslaco USA/TX 2001 Florunner | 240 SC | 0.14 | 131– 168 | 0.107– 0.083 | 3 | 28 | < 0.05 < 0.05 <u>Av < 0.05</u> | < 0.005 < 0.005 | < 0.005 < 0.005 | PR 07737* |

| Location, year (variety) | Application | | | | | PHI days | Residues, mg/kg | | | Reference |
|---|-------------|----------|---------------|-----------------|-----|-------------|---|--|--------------------|-----------|
| | Form | kg ai/ha | Water L/ha | kg ai/ha | no. | | DFB | CPU | PCA | |
| Tifton USA/GA 2001 NCV11 | 240 SC | 0.14 | 187 | 0.075 | 3 | 26 | < 0.05 < 0.05 <u>Av < 0.05</u> | c0.01**, c0.006**, < 0.005, < 0.005 | < 0.005 < 0.005 | PR 07737 |
| Tifton USA/GA 2001 C 99R | 240 SC | 0.14 | 187 | 0.075 | 3 | 20 | c0.060**, 0.052, 0.060 Av 0.056 | < 0.005 < 0.005 | < 0.005 < 0.005 | PR 07737 |
| Tifton USA/GA 2001 Georgia Green | 240 SC | 0.14 | 187 | 0.075 | 3 | 20 | c0.097**, c0.084**, 0.072, 0.097 Av 0.080 | < 0.005 < 0.005 | < 0.005 < 0.005 | PR 07737 |
| Colony USA/OK 2001 Tamspar | 240 SC | 0.14 | 94– 103 | 0.150– 0.136 | 3 | 27 | < 0.05 < 0.05 <u>Av < 0.05</u> | < 0.005 < 0.005 | < 0.005 < 0.005 | PR 07737 |
| Salisbury USA/MD 2001 VA-C98R | 240 SC | 0.14 | 383– 636 | 0.037– 0.022 | 3 | 28 | < 0.05 < 0.05 <u>Av < 0.05</u> | < 0.005 < 0.005 | < 0.005 < 0.005 | PR 07737 |
| Citra USA/FL 2001 Florunner | 240 SC | 0.14 | 140– 150 | 0.100– 0.094 | 3 | 28 | < 0.05 < 0.05 <u>Av < 0.05</u> | < 0.005 0.006 | < 0.005 < 0.005 | PR 07737 |

* Trials were conducted in the same location with different application times.

** Residues in untreated control samples.

Livestock feeds

Table 12 Residues of Diflubenzuron, CPU, and PCA in barley/wheat hay

| Location, year (variety) | Application | | | | | PHI days | Residues, mg/kg | | | Reference |
|--|-------------|----------|---------------|----------|-----|-------------|--------------------------------|----------------|--------------------|-----------|
| | Form | kg ai/ha | Water L/ha | kg ai/hL | no. | | DFB | CPU | PCA | |
| Barley hay | | | | | | | | | | |
| Kimberly USA/ID 2002 (Eight Twelve) | 240 SC | 0.066 | 114 | 0.058 | 1 | 27 | 0.73 0.75 <u>Av 0.74</u> | 0.006 0.005 | < 0.005 < 0.005 | PR 08024 |
| Aberdeen USA/ID 2002 (Gallatin) | 240 SC | 0.069 | 140 | 0.050 | 1 | 29 | 0.46 0.81 <u>Av 0.64</u> | 0.011 0.012 | < 0.005 < 0.005 | PR 08024 |
| Minot USA/ND 2003 (Robust) | 240 SC | 0.070 | 95 | 0.074 | 1 | 28 | 0.25 0.66 <u>Av 0.46</u> | 0.024 0.034 | < 0.005 < 0.005 | PR 08024* |
| Fargo USA/ND 2003 (Robust) | 240 SC | 0.070 | 112 | 0.062 | 1 | 31 | 0.52 0.64 <u>Av 0.58</u> | 0.016 0.015 | < 0.005 < 0.005 | PR 08024 |
| Minot USA/ND 2003 (Robust) | 240 SC | 0.069 | 93 | 0.074 | 1 | 25 | 0.52 0.70 <u>Av 0.61</u> | 0.038 0.025 | < 0.005 < 0.005 | PR 08024* |
| Fort USA/CO 2003 (Moravian 37) | 240 SC | 0.069 | 111 | 0.062 | 1 | 39 | 0.11 0.11 <u>Av 0.11</u> | 0.018 0.019 | < 0.005 < 0.005 | PR 08024 |

Diflubenzuron

| Location, year (variety) | Application | | | | | PHI days | Residues, mg/kg | | | Reference |
|--|-------------|----------|---------------|----------|-----|-------------|--------------------------------|--------------------|--------------------|-----------|
| | Form | kg ai/ha | Water L/ha | kg ai/hL | no. | | DFB | CPU | PCA | |
| Velva USA/ND 2003 (Foster) | 240 SC | 0.070 | 185 | 0.038 | 1 | 15 | 1.3 1.4 <u>Av 1.4</u> | 0.022 0.031 | < 0.005 < 0.005 | PR 08024 |
| Wheat hay | | | | | | | | | | |
| Kimberly USA/ID 2002 (Brundage) | 240 SC | 0.072 | 120 | 0.060 | 1 | 32 | 0.89 0.86 <u>Av 0.88</u> | < 0.005 < 0.005 | < 0.005 < 0.005 | PR 08024 |
| Fargo USA/ND 20023 (Oxen) | 240 SC | 0.069 | 111 | 0.062 | 1 | 31 | 1.2 1.3 <u>Av 1.2</u> | 0.012 < 0.005 | < 0.005 < 0.005 | PR 08024 |
| Minot USA/ND 2003 (Mountrail) | 240 SC | 0.069 | 94 | 0.074 | 1 | 28 | 0.25 0.10 <u>Av 0.18</u> | < 0.005 < 0.005 | < 0.005 < 0.005 | PR 08024 |

* Trials were conducted in the same location with deferent application times.

Table 13 Residues of Diflubenzuron, CPU, and PCA in barley/wheat straw

| Location, year (variety) | Application | | | | | PHI days | Residues, mg/kg | | | Reference |
|--|-------------|----------|---------------|----------|-----|-------------|---|--------------------|--------------------|-----------|
| | Form | kg ai/ha | Water L/ha | kg ai/hL | no. | | DFB | CPU | PCA | |
| Barley Straw | | | | | | | | | | |
| Kimberly USA/ID 2002 (Eight Twelve) | 240 SC | 0.066 | 114 | 0.058 | 1 | 76 | 0.22 0.14 <u>Av 0.18</u> | < 0.005 < 0.005 | < 0.005 < 0.005 | PR 08024 |
| Aberdeen USA/ID 2002 (Gallatin) | 240 SC | 0.069 | 140 | 0.050 | 1 | 71 | 0.096 0.13 <u>Av 0.12</u> | < 0.005 < 0.005 | < 0.005 < 0.005 | PR 08024 |
| Minot USA/ND 2003 (Robust) | 240 SC | 0.070 | 95 | 0.074 | 1 | 55 | 0.30 0.31 <u>Av 0.30</u> | < 0.005 < 0.005 | < 0.005 < 0.005 | PR 08024* |
| Fargo USA/ND 2003 (Robust) | 240 SC | 0.070 | 112 | 0.062 | 1 | 54 | 0.44 0.47 <u>Av 0.46</u> | < 0.005 < 0.005 | < 0.005 < 0.005 | PR 08024 |
| Minot USA/ND 2003 (Robust) | 240 SC | 0.069 | 93 | 0.074 | 1 | 54 | 0.57 0.55 <u>Av 0.56</u> | < 0.005 < 0.005 | < 0.005 < 0.005 | PR 08024* |
| Fort USA/CO 2003 (Moravian 37) | 240 SC | 0.069 | 111 | 0.062 | 1 | 75 | < 0.05 < 0.05 <u>Av < 0.05</u> | < 0.005 < 0.005 | < 0.005 < 0.005 | PR 08024 |
| Velva USA/ND 2003 (Foster) | 240 SC | 0.070 | 185 | 0.038 | 1 | 50 | 0.54 0.54 <u>Av 0.54</u> | 0.010 0.013 | < 0.005 < 0.005 | PR 08024 |
| Wheat Straw | | | | | | | | | | |
| Kimberly USA/ID 2002 (Brundage) | 240 SC | 0.072 | 120 | 0.060 | 1 | 76 | 0.23 0.34 <u>Av 0.28</u> | < 0.005 < 0.005 | < 0.005 < 0.005 | PR 08024 |

| Location, year (variety) | Application | | | | | PHI days | Residues, mg/kg | | | Reference |
|--|-------------|----------|---------------|----------|-----|-------------|-----------------------------------|--------------------|--------------------|-----------|
| | Form | kg ai/ha | Water L/ha | kg ai/hL | no. | | DFB | CPU | PCA | |
| Fargo USA/ND 20023 (Oxen) | 240 SC | 0.069 | 111 | 0.062 | 1 | 56 | 0.77 1.03 <u>Av 0.90</u> | 0.008 0.008 | < 0.005 < 0.005 | PR 08024 |
| Minot USA/ND 2003 (Mountrail) | 240 SC | 0.069 | 94 | 0.074 | 1 | 62 | < 0.05 0.066 <u>Av 0.06</u> | < 0.005 < 0.005 | < 0.005 < 0.005 | PR 08024 |

*: Trials were conducted in the same location with deference application time.

Table 14 Residues of Diflubenzuron, CPU, and PCA in/on almond hulls

| Location, year (variety) | Application | | | | | PHI days | Residues, mg/kg | | | Reference |
|---|-----------------------------|------------------------------|----------------------------------|--------------------------|-----|-------------|------------------------------------|----------------------------|---------------------------------|-----------|
| | Form | kg ai/ha | kg ai/hL | Water L/ha | no. | | DFB | CPU | PCA | |
| Madera USA/CA 1998 Nonpareil | 250 WP | 0.56 0.28 0.28 0.56 | 0.06 0.06 0.06 0.12 | 935 467 467 467 | 4 | 28 | 2.1 2.1 <u>Av 2.1</u> | < 0.01, < 0.01 | < 0.005, 0.0082 | RP-98003 |
| Kerman USA/CA 1998 Nonpareil | 250 WP | 0.57 0.27 0.29 0.56 | 0.06 0.059 0.061 0.12 | 935 467 467 467 | 4 | 28 | 2.3 2.3 <u>Av 2.3</u> | < 0.01, < 0.01 | < 0.005, < 0.005 | RP-98003 |
| Reedley USA/CA 1998 Butte | 250 WP | 0.56 0.28 0.28 0.56 | 0.060 0.060 0.061 0.12 | 935 467 467 467 | 4 | 28 | 2.9 3.6 Av 3.2 | < 0.01, < 0.01 | < 0.005, < 0.005 | RP-98003 |
| | 800 WG | 0.57 0.28 0.28 0.56 | 0.060 0.059 0.060 0.12 | 935 467 467 467 | 4 | 28 | 3.4 5.5 <u>Av 4.4</u> | < 0.01, < 0.01 | 0.0052, 0.0074 | |
| | 240 SC | 0.56 0.28 0.28 0.56 | 0.059 0.060 0.059 0.12 | 935 467 467 467 | 4 | 28 | 2.9 3.1 Av 3.0 | < 0.01, < 0.01 | < 0.005, 0.0067 | |
| Manteca USA/CA 1998 Nonpareil | 250 WP | 0.56 0.28 0.28 0.56 | 0.060 0.060 0.061 0.12 | 935 467 467 467 | 4 | 28 | 0.97 1.1 <u>Av 1.0</u> | < 0.01, < 0.01 | < 0.005, < 0.005 | RP-98003 |
| Ripon USA/CA 1998 Nonpareil | 250 WP | 0.81 0.49 0.28 0.55 | 0.060 0.060 0.059 0.12 | 935 467 467 467 | 4 | 28 | 1.5 1.6 <u>Av 1.6</u> | < 0.01, < 0.01 | < 0.005, < 0.005 | RP-98003 |
| Madera USA/CA 2003 Padre Butte | 240 SC | 0.28 0.28 0.28 0.28 | 0.029 0.060 0.060 0.060 | 954 477 468 477 | 4 | 28 | 4.2 3.6 4.1 <u>Av 4.0</u> | 0.020 0.0051 < 0.005 | < 0.005, < 0.005, < 0.005 | RP-03001 |
| | 240 SC 0.25% crop oil | 0.28 0.28 0.28 0.28 | 0.029 0.060 0.060 0.060 | 944 468 468 477 | 4 | 28 | 2.8 2.5 3.4 Av 2.9 | 0.012 0.019 < 0.005 | < 0.005, < 0.005, 0.0051 | |
| Winters USA/CA 2003 Butte | 240 SC | 0.28 0.28 0.28 0.28 | 0.032 0.062 0.062 0.062 | 888 449 449 458 | 4 | 28 | 1.9 2.0 2.3 <u>Av 2.1</u> | 0.011 0.011 0.0085 | < 0.005, < 0.005, < 0.005 | RP-03001 |
| | 240 SC 0.25% crop oil | 0.28 0.28 0.28 0.28 | 0.032 0.062 0.062 0.062 | 888 449 449 458 | 4 | 28 | 1.6 1.5 1.3 Av 1.5 | 0.075 0.050 0.064 | < 0.005, < 0.005, < 0.005 | |

Table 15 Residues of Diflubenzuron, CPU, and PCA in/on peanut hay

| Location, year (variety) | Application | | | | | PHI days | Residues, mg/kg | | | Reference |
|---|-------------|----------|---------------|------------------|-----|-------------|-----------------|--------|-------|-----------|
| | Form | kg ai/ha | Water L/ha | kg ai/hL | no. | | DFB | CPU | PCA | |
| Crossville USA/TN 2001 VA98R | 240 SC | 0.14 | 215– 224 | 0.0675– 0.062 | 3 | 28 | <u>18.4</u> | 0.052 | 0.016 | PR 07737 |
| Crossville USA/TN 2001 VA-C92R | 240 SC | 0.14 | 215– 224 | 0.065– 0.062 | 3 | 28 | 10.7 | 0.065 | 0.037 | PR 07737 |
| Rocky USA/NC 2001 VA98R | 240 SC | 0.14 | 168 | 0.083 | 3 | 28 | <u>17</u> | 0.018 | 0.018 | PR 07737 |
| Weslaco USA/TX 2001 Florunner | 240 SC | 0.14 | 131– 159 | 0.107– 0.088 | 3 | 29 | <u>8.4</u> | < 0.01 | 0.015 | PR 07737* |
| Weslaco USA/TX 2001 Florunner | 240 SC | 0.14 | 131– 168 | 0.107– 0.083 | 3 | 28 | <u>7.9</u> | 0.019 | 0.021 | PR 07737* |
| Tifton USA/GA 2001 NCV11 | 240 SC | 0.14 | 187 | 0.075 | 3 | 26 | <u>2.6</u> | 0.012 | 0.047 | PR 07737 |
| Tifton USA/GA 2001 C 99R | 240 SC | 0.14 | 187 | 0.075 | 3 | 20 | 1.1 | 0.013 | 0.054 | PR 07737 |
| Tifton USA/GA 2001 Georgia Green | 240 SC | 0.14 | 187 | 0.075 | 3 | 20 | 1.1 | 0.024 | 0.046 | PR 07737 |
| Colony USA/OK 2001 Tamspan | 240 SC | 0.14 | 94– 103 | 0.150– 0.136 | 3 | 27 | <u>1.6</u> | 0.019 | 0.013 | PR 07737 |
| Salisbury USA/MD 2001 VA-C98R | 240 SC | 0.14 | 383– 636 | 0.037– 0.022 | 3 | 28 | <u>7.1</u> | 0.046 | 0.017 | PR 07737 |
| Citra USA/FL 2001 Florunner | 240 SC | 0.14 | 140– 150 | 0.100– 0.094 | 3 | 28 | <u>1.9</u> | 0.025 | 0.014 | PR 07737 |

*: Trials were conducted in the same location with deferent application times.

APPRAISAL

Diflubenzuron [1-(4-chlorophenyl)-3-(2,6-difluorobenzoyl)urea] is an insect growth regulator used in agriculture. It was originally evaluated by the JMPR in 1981 and re-evaluated for residues several times up to 1988.

Under the periodic review program, toxicology data was re-evaluated by JMPR in 2001. The original ADI of 0–0.02 mg/kg bw/day was re-confirmed and an acute reference dose was unnecessary. The compound was re-evaluated for residues by the JMPR in 2002.

This Meeting received information on the residue analysis, storage stability, use patterns and supervised field residue trials for peaches, plums, peppers, mustard greens, barley, wheat, almond, pecan and peanut.

Analytical methods

The Meeting received details of several analytical methods used in supervised residue trials and in studies on storage stability, which are primarily based on the methods previously reviewed by JMPR in 2002, with some modifications to minimize matrix interference. All methods are single methods for determination of diflubenzuron.

For determination of diflubenzuron, HPLC analysis with UV detection was validated for almond, mustard greens, peppers, peanuts, barley and wheat. The limits of quantification were 0.005 or 0.05 mg/kg for almond hulls, 0.05 mg/kg for almond nutmeat, mustard green, peach, peanut nutmeat and oil, wheat and wheat processed commodities, 0.005 mg/kg for peppers, 0.5 mg/kg for peanut meal and peanut hay. GC-ECD analysis was validated for peppers with an LOQ of 0.05 mg/kg.

Stability of residues in stored analytical samples

The Meeting received data on the stability of residues in plant products (almond nutmeat and hulls, peach, plum, mustard greens, peanut nutmeat, peanut hay, peanut meal, peanut oil, wheat forage, wheat hay, wheat grain, wheat straw, wheat flour and wheat germ) in the corresponding supervised residue trials. The storage stability data covered the period of storage of field samples for residue analysis. The lowest freezer temperature was -24 °C. The average freezer temperature was -18 °C.

Diflubenzuron residues in fortified samples were stable over a period of 13 months frozen storage for peaches, 12 months for peppers and 14 months for mustard greens. Residues in fortified samples of wheat grain were stable for 296 days, barley straw for 301 days, wheat forage for 422 days and wheat hay for 337 days of frozen storage.

For wheat processed commodities, diflubenzuron residue is also stable in wheat flour for 6 months and wheat germ for 12 months of frozen storage.

In almond nutmeat, diflubenzuron residues were stable over a period of 12 months of frozen storage.

In peanuts, diflubenzuron was stable in nutmeat for 295 days, hay for 356 days, peanut meal for 643 days, and refined oil for 365 days.

Results of supervised trials on crops

The Meeting received supervised residue trials data following foliar application of diflubenzuron on peaches, plums, peppers, mustard greens, barley, wheat, almonds, pecans and peanuts.

Residues of diflubenzuron were reported in all studies. Supervised field trials conducted with different formulations (wetable powders, suspensions concentrates and wettable granules), but with identical crop varieties, locations and spray dates were not considered as independent. The highest result according to the corresponding GAP was selected in these cases. Where multiple samples were taken from a single plot and individual results are reported, the mean value is used for estimation of maximum residue level.

The OECD calculator was used as a tool in the estimation of the maximum residue level from the selected residue data set obtained from trials conducted according to GAP. As a first step, the Meeting reviewed all relevant factors related to each data set in arriving at a best estimate of the maximum residue level using expert judgment. Then, the OECD calculator was employed. If the statistical calculation spreadsheet suggested a different value from that recommended by the JMPR, a brief explanation of the deviation was supplied.

Peaches and Plums

Five field trials on peaches and three trials on plums conducted in 2005 in the USA matched the critical USA GAP for stone fruit, which is two applications at a rate of 0.28 kg ai/ha (interval 14 days) with a PHI of 14 days.

Residues of diflubenzuron in peaches from trials matching GAP, in ranked order, were (n = 5): 0.12, 0.17, 0.20, 0.21 and 0.23 mg/kg.

The ranked order of diflubenzuron residue data in plums matching GAP were (n = 3): 0.08, 0.17 and 0.17 mg/kg.

Individually there were insufficient trials from each crop to estimate commodity maximum residue levels. As the residue levels found were from similar populations, the Meeting agreed that the two data sets could be used for mutual support and decided to combine the data for evaluation. Residues found on peaches and plums, in ranked order, were (n = 8): 0.08, 0.12, 0.17, 0.17, 0.17, 0.20, 0.21 and 0.23 mg/kg.

The Meeting agreed to estimate an STMR of 0.17 mg/kg, and recommended a maximum residue level of 0.5 mg/kg for diflubenzuron in peaches and plums (including prunes). Further the Meeting agreed to extrapolate these recommendations to nectarines.

Mustard greens

Eight field trials on mustard greens were conducted in the USA during 2001 growing season matched the USA GAP, which is a maximum of four foliar applications at a rate of 0.07 kg ai/ha with a PHI of 7 days.

Residues of diflubenzuron found on mustard greens, in ranked order, were (n = 8): < 0.05, 1.0, 1.1, 1.2, 1.5, 2.1, 2.5 and 6.8 mg/kg.

The Meeting agreed to estimate an STMR of 1.35 mg/kg, and recommended a maximum residue level of 10 mg/kg for mustard greens.

Sweet peppers

Six field trials on sweet peppers and three field trials on chili peppers were conducted in the USA in 1997 according to the GAP of the USA, i.e., a maximum of five foliar applications at a rate of 0.14 kg ai/ha with a PHI of 7 days.

Residues of diflubenzuron found on sweet peppers, in ranked order, were (n = 6): 0.07, 0.07, 0.08, 0.24, 0.24, and 0.33 mg/kg.

The ranked order of diflubenzuron residue data in chili peppers were (n = 3): 0.25, 0.92 and 0.94 mg/kg.

It was considered that the datasets for sweet peppers and chili peppers were not from similar residue populations and as a consequence could not be combined. On the basis of the data for sweet peppers the Meeting agreed to estimate an STMR of 0.16 mg/kg, and recommended a maximum residue level of 0.7 mg/kg for sweet peppers.

As chili peppers are a minor crop, the Meeting agreed to estimate an STMR of 0.92 mg/kg, and recommended a maximum residue level of 3 mg/kg for chili peppers.

On the basis of the STMR and maximum residue level for chili peppers and the default dehydration factor of 7, the Meeting estimated an STMR of 6.44 mg/kg, and recommended a maximum residue level of 20 mg/kg for chili peppers, dry.

Cereal grains

Wheat and barley

Seven field trials on barley and three field trials on wheat were conducted in the USA between 2002 and 2003 growing seasons following the USA GAP for barley, wheat, oats and triticale, which is a maximum of one foliar application at a rate of 0.07 kg ai/ha up to boot stage (BBCH 41).

The diflubenzuron residue data in barley grain from trial according to GAP were (n = 7): < 0.05, < 0.05, < 0.05, < 0.05, < 0.05, < 0.05 and < 0.05 mg/kg.

The diflubenzuron residue data in wheat grain from trial according to GAP were (n = 3): < 0.05, < 0.05 and < 0.05 mg/kg.

As the applications from seven trials on barley and three trials on wheat before boot stage (BBCH 41) resulted in residue data below the LOQ of 0.05 mg/kg, the Meeting decided to combine the two datasets together for the evaluation.

The Meeting agreed to estimate an STMR of 0.05 mg/kg, and recommended a maximum residue level of 0.05* mg/kg for barley and wheat, and agreed to extrapolate these recommendation to oats and triticale.

Tree nuts

Almonds and pecans

For almonds, five field trials in 1998 and two field trials in 2003 were conducted in the USA, and for pecan five trials were conducted in 1999 in the USA.

In almond nutmeat, two trials followed the USA GAP for tree nuts, which is a maximum of four foliar applications at a rate of 0.28 kg ai/ha with PHI 28 days, the residue data were 0.033 and 0.048 mg/kg. The LOQ for these trials was 0.005 mg/kg.

Five trials were conducted with twice rate at PHI of 28 days and all residue data were below LOQ of 0.05 mg/kg.

In pecan kernels, all residue data in five trials with twice rate at PHI of 28 days were below LOQ of 0.05 mg/kg

The Meeting agreed to combine all residue data for the evaluation. The diflubenzuron residue data in almond nutmeat and pecan kernels from trial were (n = 12): 0.033, 0.048 and < 0.05 (10) mg/kg.

Considering the maximum residue value on individual replicate sample prior to averaging up to 0.089 mg/kg (mean trial value was 0.045 mg/kg), the Meeting agreed to estimate an STMR of 0.05 mg/kg, and recommended a maximum residue level of 0.2 mg/kg for tree nuts.

Peanuts

Field trials were conducted in the USA in the 2001 growing season following the USA GAP of a maximum of three foliar applications at a rate of 0.14 kg ai/ha with PHI of 28 days.

The ranked order of diflubenzuron residue data in peanut nutmeat were (n = 9): < 0.05, < 0.05, < 0.05, < 0.05, < 0.05, < 0.05, < 0.05, 0.05 and 0.06 mg/kg.

The Meeting agreed to estimate an STMR of 0.05 mg/kg, and recommended a maximum residue level of 0.15 mg/kg for peanut nutmeat.

Animal feed commodities

Hay or fodder (dry) of grasses

Seven field trials on barley and three field trials on wheat were conducted in the USA between 2002 and 2003 growing seasons following the USA GAP for barley, wheat, oats and triticale, which is a maximum of one foliar application at a rate of 0.07 kg ai/ha up to boot stage.

In barley hay, the diflubenzuron residue data from trial matching GAP were (n = 7): 0.11, 0.46, 0.58, 0.61, 0.64, 0.74 and 1.4 mg/kg.

Residues of diflubenzuron found in wheat hay following treatments complying with the US GAP were (n = 3): 0.18, 0.88 and 1.2 mg/kg.

The Meeting agreed that the residues found in wheat and barley were from the same population and could be combined for evaluation. The ranked order of diflubenzuron residues found

in barley and wheat hay were (n = 10): 0.11, 0.18, 0.46, 0.58, 0.61, 0.64, 0.74, 0.88, 1.2 and 1.4 mg/kg.

The Meeting agreed to estimate a median residue of 0.625 mg/kg, a highest residue of 1.4 mg/kg, and recommend a maximum residue level of 3 mg/kg for hay or fodder (dry) of grasses.

Straw and fodder (dry) of cereal grain

Seven field trials on barley and three field trials on wheat were conducted in the USA between 2002 and 2003 growing seasons matching the GAP of the USA in barley, wheat, oats and triticale, which is a maximum of one foliar application at a rate of 0.07 kg ai/ha up to boot stage.

In barley straw, the diflubenzuron residue data from trial matching GAP were (n = 7): < 0.05, 0.12, 0.18, 0.30, 0.46, 0.54 and 0.56 mg/kg.

In wheat straw, the diflubenzuron residue data from trial matching GAP were (n = 3): 0.06, 0.28 and 0.90 mg/kg.

The Meeting agreed that the residues found in wheat and barley were from the same population and could be combined for evaluation. The ranked order of diflubenzuron residues found in barley and wheat straw were (n = 10): < 0.05, 0.06, 0.12, 0.18, 0.28, 0.30, 0.46, 0.54, 0.56 and 0.90 mg/kg.

The Meeting agreed to estimate a median residue of 0.29 mg/kg, a highest residue of 0.90 mg/kg, and recommend a maximum residue level of 1.5 mg/kg for straw and fodder (dry) of cereal grain.

Almond hulls

Field trials in 1998 and four field trials in 2003 on almonds were conducted in the USA complying with the GAP of the USA, i.e., a maximum of four foliar applications at a rate of 0.28 kg ai/ha with a PHI of 28 days.

Residues in almond hulls from two trials, matching the US GAP, were 2.1 mg/kg and 4.0 mg/kg.

Five trials were conducted at a double rate (0.56 kg ai/ha) and a PHI of 28 days. Residue data from these trials were: 1.0, 1.6, 2.1, 2.3 and 4.4 mg/kg. The Meeting agreed that the results from these trials could be scaled to match the US GAP (0.28 kg ai/ha application rate) by dividing by 2 (0.56/0.28). The proportionally adjusted residues in almond hull were: 0.5, 0.8, 1.05, 1.15 and 2.2 mg/kg.

The Meeting agreed that the two dataset matching the USA GAP were not significantly different and could be combined for evaluation. The combined residue data were (n = 7): 0.5, 0.8, 1.05, 1.15, 2.1, 2.2 and 4.0 mg/kg.

The Meeting agreed to estimate a median residue of 1.15 mg/kg.

Peanut hay

Field trials were conducted in the USA in 2001 growing season following the USA GAP, i.e., 3 × 0.14 kg ai/ha with a PHI of 28 days.

The ranked order of diflubenzuron residue concentrations in peanut hay were (n = 8): 1.6, 1.9, 2.6, 7.1, 7.9, 8.4, 17.0 and 18.4 mg/kg.

The Meeting agreed to estimate a median residue of 7.5 mg/kg, a highest residue of 18.4 mg/kg and recommended a maximum residue level of 40 mg/kg for peanut hay.

Fate of residues during processing

The Meeting did not receive any information on the fate of incurred residue of diflubenzuron in processing of relevant commodities.

Residues of animal commodities

Farm animal dietary burden

In 2002, the JMPR estimated the dietary burden from residues in wet pomace of apples, grass forage, rice grain and rice straw of diflubenzuron residues in farm animals from the diets listed in Appendix IX of the FAO Manual (FAO, 2002).

The present Meeting estimated the dietary burden of diflubenzuron in farm animals on the basis of the diets listed in Appendix X of the FAO Manual (OECD Feedstuffs Derived from Field Crops, FAO, 2009). Calculation from the highest residues, the STMRs (some bulk commodities) and STMR-P values provides the levels in feed suitable for estimating maximum residue levels, while calculation from the STMRs and STMR-P values for feed is suitable for estimating STMR values for animal commodities. Dietary burden calculations for beef cattle, dairy cattle, broilers and laying poultry are provided in Annex 6 of the 2011 JMPR Report.

The present Meeting calculated the dietary burdens from residues in wet pomace of apples, grass forage, rice grain, rice straw, barley and wheat grain, hay, straw, almond hulls, and peanut hay. The results are summarized in the following table.

| | Livestock dietary burden, diflubenzuron, ppm of dry matter diet | | | | | | | |
|-----------------|---|------|-------------------|-------------------|--------------------|-------------------|-------|-------|
| | US/CAN | | EU | | Australia | | Japan | |
| | max | mean | max | mean | max | mean | max | mean |
| Beef cattle | 0.27 | 0.13 | 11.52 | 4.82 | 20.99 ^a | 8.11 ^b | 1.41 | 0.38 |
| Dairy cattle | 13.45 | 5.19 | 12.77 | 4.73 | 20.99 ^c | 8.02 ^d | 2.14 | 0.69 |
| Poultry-broiler | 0.04 | 0.04 | 0.04 | 0.04 | 0.04 | 0.04 | 0.006 | 0.006 |
| Poultry-layer | 0.04 | 0.04 | 2.05 ^e | 0.71 ^f | 0.04 | 0.04 | | |

^a Highest maximum beef or dairy cattle burden suitable for maximum residue level estimates for mammalian meat

^b Highest mean beef or dairy cattle dietary burden suitable for STMR estimates for mammalian meat.

^c Highest maximum dairy cattle dietary burden suitable for maximum residue level estimates for milk.

^d Highest mean dairy cattle dietary burden suitable for STMR estimates for milk.

^e Highest maximum poultry dietary burden suitable for maximum residue level estimates for poultry meat and eggs.

^f Highest mean poultry dietary burden suitable for STMR estimates for poultry meat and eggs.

The dietary burdens were recalculated using the OECD tables and the addition of barley and wheat grain, hay straw, almond hulls, and peanut hay to the animal feed diet did not significantly increase the dietary burden value. The Meeting decided that it is not necessary to re-evaluated animal commodities for maximum residue levels.

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 dietary intake assessment.

The definition of the residue for plant and animal commodities (for compliance with the MRL and for estimation of dietary intake): *diflubenzuron*.

The residue is fat soluble.

| Commodity | | Recommended MRL mg/kg | STMR or STMR-P | HR or HR-P |
|-----------|----------------|-----------------------|----------------|------------|
| CCN | Name | New | mg/kg | mg/kg |
| GC 0640 | Barley | 0.05* | 0.05 | |
| VL 0485 | Mustard greens | 10 | 1.35 | |
| FS 0245 | Nectarine | 0.5 | 0.17 | |
| GC 0647 | Oats | 0.05* | 0.05 | |
| FS 0247 | Peach | 0.5 | 0.17 | |
| SO 0697 | Peanut | 0.15 | 0.05 | 0.0 |

| Commodity | | Recommended MRL mg/kg | STMR or STMR-P | HR or HR-P |
|-----------|--|-----------------------|----------------|------------|
| CCN | Name | New | mg/kg | mg/kg |
| VO 4444 | Peppers, Chili | 3 | 0.92 | |
| HS 0444 | Peppers Chili, dried | 20 | 6.44 | |
| FS 0014 | Plums (including prunes) | 0.5 | 0.17 | |
| VO 0445 | Peppers, Sweet (including Pimento or pimiento) | 0.7 | 0.16 | |
| TN 0085 | Tree nuts | 0.2 | 0.05 | 0.0 |
| GC 0653 | Triticale | 0.05* | 0.05 | |
| GC 0654 | Wheat | 0.05* | 0.05 | |
| AM 0660 | Almond Hulls | – | 1.15 | – |
| AS 0162 | Hay or fodder (dry) of grasses | 3 | 0.625 | 1.4 |
| AL 0697 | Peanut fodder | 40 | 7.5 | 18.4 |
| AS 0081 | Straw and fodder (dry) of cereal grain | 1.5 | 0.29 | 0.90 |

*: at or about the limit of quantification

DIETARY RISK ASSESSMENT

Long-term intake

The acceptable daily intake (ADI) of 0–0.02 mg/kg bw/day based on the NOAEL for haematological effects of 2 mg/kg bw per day in a 2-year studies in rats and the 52-week study in dogs was re-confirmed by 2001 JMPR.

International Estimated Daily Intake (IEDI) was calculated for commodities of human consumption for which STMRs for diflubenzuron were estimated. Results are presented in Annex 3 of the 2011 JMPR Report. The IEDI for the 13 GEMS/Food cluster diets were only 2–10% of the maximum ADI. The intake of residues of diflubenzuron resulting from its proposed uses is unlikely to present a public health concern.

Short-term intake

The JMPR in 2001 concluded that it was unnecessary to establish an ARfD, and therefore the short-term intake of diflubenzuron residues is unlikely to present a public health concern.

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