

**DRAFT**

**Water and Sediment Quality Criteria Report for Fipronil**

Phase III: Application of the pesticide water and sediment quality criteria  
methodologies



Prepared for the Central Valley Regional Water Quality Control Board

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*October 2016*

## **Disclaimer**

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## *List of acronyms and abbreviations*

|                                  |   |
|----------------------------------|---|
| ACR                              | Acute-to-Chronic Ratio  |
| AF                               | Assessment Factor   |
| ASTM                             | American Society for Testing and Materials                    |
| BAF                              | Bioaccumulation Factor  |
| BCF                              | Bioconcentration Factor                                       |
| BMF                              | Biomagnification Factor                                       |
| BSQC                             | Bioavailable Sediment Quality Criteria                        |
| CAS                              | Chemical Abstract Service                                     |
| CDFG/CDFW                        | California Department of Fish and Wildlife                    |
| CDPR                             | California Department of Pesticide Regulation                 |
| CDWR                             | California Department of Water Resources                      |
| CVRWQCB                          | Central Valley Regional Water Quality Control Board           |
| DOC                              | Dissolved Organic Carbon                                      |
| DOM                              | Dissolved Organic Matter                                      |
| EC <sub>x</sub>                  | Concentration that affects x% of exposed organisms            |
| FDA                              | Food and Drug Administration                                  |
| FT                               | Flow-through test   |
| GMAV                             | Genus Mean Acute Value  |
| IA                               | Independent Action  |
| IC <sub>x</sub>                  | Inhibition concentration; concentration causing x% inhibition |
| ICE                              | Interspecies Correlation Estimation                           |
| IUPAC                            | International Union of Pure and Applied Chemistry             |
| K                                | Interaction Coefficient                                       |
| K <sub>H</sub>                   | Henry's law constant  |
| K <sub>ow</sub>                  | Octanol-Water partition coefficient                           |
| K <sub>oc</sub>                  | Organic Carbon sorption partition coefficient                 |
| K <sub>p</sub> or K <sub>d</sub> | Solid-Water partition coefficient                             |
| LC <sub>x</sub>                  | Concentration lethal to x% of exposed organisms               |
| LD <sub>x</sub>                  | Dose lethal to x% of exposed organisms                        |
| LL                               | Less relevant, Less reliable study                            |
| LOEC                             | Lowest-Observed Effect Concentration                          |
| LOEL                             | Lowest-Observed Effect Level                                  |
| LR                               | Less relevant, Reliable study                                 |
| MATC                             | Maximum Acceptable Toxicant Concentration                     |
| N                                | Not relevant or Not reliable study                            |
| n/a                              | Not applicable  |
| NEC                              | No-effect concentration                                       |
| NOAEL                            | No-Observed Adverse Effect Level                              |
| NOEC                             | No-Observed Effect Concentration                              |
| NR                               | Not reported  |
| OC                               | Organic Carbon  |
| PBO                              | Piperonyl butoxide  |
| pK <sub>a</sub>                  | Acid dissociation constant                                    |
| RL                               | Relevant, Less reliable study                                 |

|       |   |
|-------|---|
| RR    | Relevant and Reliable study   |
| S     | Static test   |
| SMAV  | Species Mean Acute Value  |
| SMCV  | Species Mean Chronic Value  |
| SPME  | Solid-phase Microextraction   |
| SR    | Static renewal test   |
| SSD   | Species Sensitivity Distribution  |
| TES   | Threatened and Endangered Species   |
| TIE   | Toxicity Identification Evaluation  |
| UCDM  | University of California Davis water quality criteria derivation methodology    |
| UCDSM | University of California Davis sediment quality criteria derivation methodology |
| US    | United States   |
| USEPA | United States Environmental Protection Agency                                   |

## 1 Introduction

Two new methodologies for deriving freshwater water quality criteria (TenBrook et al. 2009) and sediment quality criteria (Fojut et al. 2014) for the protection of aquatic life have been developed by the University of California, Davis. The need for these new methodologies was identified by the California Central Valley Regional Water Quality Control Board (CVRWQCB 2006, CRWQCB-CVR 2011) and findings from reviews of existing methodologies (TenBrook & Tjeerdema 2006, TenBrook et al. 2009, Fojut et al. 2011, 2013). These new methodologies are currently being used to derive aquatic life criteria for several pesticides of particular concern in the Sacramento River and San Joaquin River watersheds. The water quality criteria methodology report (TenBrook et al. 2009) and the sediment quality criteria report (Fojut et al. 2014) each contain an introduction; the rationale of the selection of specific methods; detailed procedures for criteria derivation; and a criteria report for a specific pesticide. This criteria report for fipronil and select degradates describes, section by section, the procedures used to derive both the water quality criteria and sediment quality criteria according to the UC-Davis Method (UCDM) and UC-Davis Sediment Method (UCDSM), respectively. Also included are references to specific sections of the methodology procedures detailed in these reports so that the reader can refer to the appropriate report for further details (TenBrook et al. 2009, Fojut et al. 2014).

Fipronil is sold as a racemic mixture of 50:50 (+) : (-) enantiomers. It has been shown that the enantiomers are uniquely toxic. As shown in the following report, the (+) enantiomer is more toxic than both the racemate and the (-) enantiomer to a variety of taxa.

In the environment, fipronil readily and abiotically degrades to several degradates that are more or less stable. Distinct degradates are formed through photolysis, hydrolysis, oxidation, and reduction in soil and/or water. This report includes all of the available degradates. Some sections do not mention a particular degradate due to a dearth of data for that particular chemical species. Environmental persistence of the degradates could not be established or discussed because  $K_{OW}$  were not available. The data tables are color coded to assist the reader in separating each of the degradates from the parent compound fipronil. The enantiomers are not color coded but are included within the fipronil tables.

## 2 Basic information

Chemical: Fipronil (Fig. 1)

CAS: 5-amino-1-[2,6-dichloro-4-(trifluoromethyl)phenyl]-4-[(trifluoromethyl)sulfinyl]-1*H*-pyrazole-3-carbonitrile

IUPAC: 5-amino-1-(2,6-dichloro- $\alpha,\alpha,\alpha$ -trifluoro-*p*-tolyl)-4-trifluoromethylsulfinylpyrazole-3-carbonitrile

Chemical Formula:  $C_{12}H_4Cl_2F_6N_4OS$

CAS Number: 120068-37-3

CA DPR Chem Code: 3995

Trade names: Regent, Goliath, Nexa, Adonis, Termidor, Ultrathor, Taurus, Frontline TopSpot, Fiproguard, Flevox, PetArmor and Agenda, Ascend, Blitz, Cosmos, Frontline Spot-on, Frontline Spray, Granedo MC, Grenade MC, Maxforce FC, Maxforce FC Select Roach Killer Bait Gel, TopChoice.

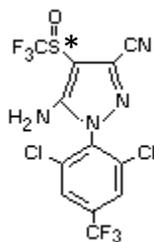


Figure 1 Structure of fipronil, a phenylpyrazole, with the chiral center noted.

(Hamernik 1997)

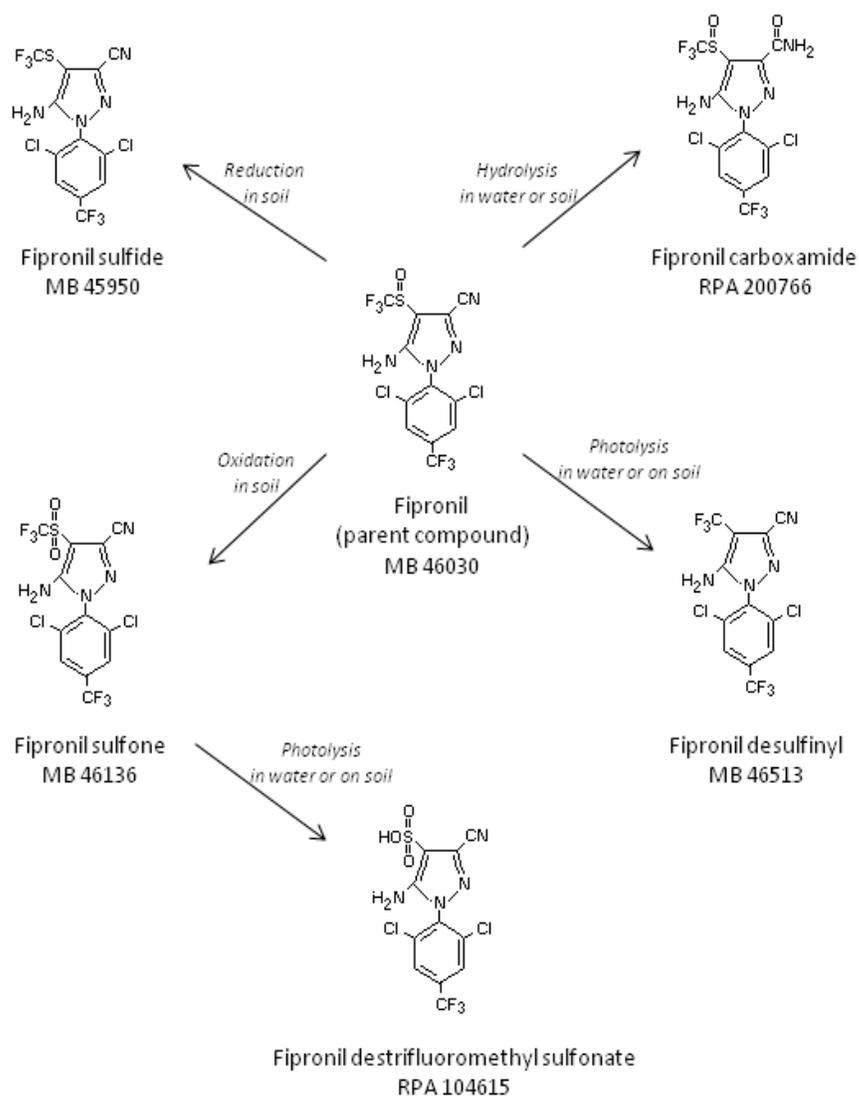


Figure 2 Environmental degradation pathway for fipronil.

(Adapted from Hamernik 1997)

### 3 Physicochemical data

Data is included for all degradates when available.

#### Molecular Weight (g/mol)

|                      |        |                     |
|----------------------|--------|---------------------|
| Fipronil             | 437.15 | USEPA 2015a         |
| Fipronil-sulfide     | 421.15 | Sigma-Aldrich 2016a |
| Fipronil-carboxamide | 455.16 | Sigma-Aldrich 2016b |
| Fipronil-sulfone     | 453.15 | Sigma-Aldrich 2016c |
| Fipronil-desulfinyl  | 389.08 | Sigma-Aldrich 2016d |

|   |           |              |
|---|-----------|--------------|
| <u>Density (g/mL)</u>   |           |              |
| Fipronil  | 1.71      | PPDB 2015    |
| <u>Water Solubility (mg/L)</u>  |           |              |
| Fipronil  | 2.4       | Stark 2005   |
| Fipronil  | 3.78      | PPDB 2015    |
| Fipronil  | 0.3743    | USEPA 2015a  |
| Fipronil  | 1.9       | Bobè 1997    |
| Fipronil  | 1.9       | USEPA 2015a  |
| <b>Geomean: 1.65</b>  |           |              |
| Fipronil-sulfone  | 0.16      | USEPA 2011   |
| Fipronil-desulfinyl   | 0.95      | USEPA 2011   |
| <u>Melting Point (°C)</u>   |           |              |
| Fipronil  | 200.5     | USEPA 2011   |
| Fipronil  | 203.92    | USEPA 2011   |
| <u>Vapor Pressure (Pa, 25°C)</u>  |           |              |
| Fipronil  | 1.51E-7   | USEPA 2011   |
| Fipronil  | 3.71E-7   | USEPA 2011   |
| Fipronil  | 2.0E-5    | PPDB 2015    |
| Fipronil  | 1.9E-6    | Goel 2007    |
| <b>Geomean: 1.04E-6</b>   |           |              |
| <u>Organic Carbon Sorption Partition Coefficients (<math>K_{oc}</math>, L/kg)</u> |           |              |
| Fipronil  | 5,923     | USEPA 2015a  |
| Fipronil  | 10,040    | USEPA 2015a  |
| Fipronil  | 37,154    | Brennan 2009 |
| Fipronil  | 32,359    | Brennan 2009 |
| Fipronil  | 802       | Lin 2009     |
| Fipronil  | 396       | Spomer 2010  |
| <b>Fipronil geomean: 5,321</b>  |           |              |
| Fipronil-sulfide  | 3,911     | Burr 1997    |
| Fipronil-sulfide  | 489,779   | Brennan 2009 |
| Fipronil-sulfide  | 398,107   | Brennan 2009 |
| Fipronil-sulfide  | 3,684     | Lin 2009     |
| <b>Fipronil-sulfide geomean: 40,904</b>   |           |              |
| Fipronil-sulfone  | 1,621,810 | Brennan 2009 |
| Fipronil-sulfone  | 630,957   | Brennan 2009 |
| Fipronil-sulfone  | 3,543     | Lin 2009     |
| <b>Fipronil-sulfone geomean: 153,623</b>  |           |              |

Fipronil-desulfinyl 1,150-1,498 Feung and Mislankar 1996  
 Fipronil-desulfinyl 1,296 Lin 2009  
**Fipronil-desulfinyl geomean: 1,310**

Henry's constant ( $K_H$ , atm m<sup>3</sup>/mole)

Fipronil 3.17E-18 USEPA 2015a  
 Fipronil 8.42E-10 USEPA 2015a  
**Geomean: 5.17E-14**

Log  $K_{ow}$

\*Values referenced from the BioByte Bio-Loom program (2015)

Fipronil 6.64 USEPA 2015a  
 Fipronil 4.00 USEPA 2015a  
 Fipronil 4.00 Tomlin 1997\*  
 Fipronil 3.68 Donovan and Pescatore 2002\*  
**Geomean: 4.45**

Environmental Fate

Table 1 Bioconcentration factors (BCF) for fipronil

NR: not reported.

| Species | BCF (L/kg) | Exposure | Reference   |
|---------|------------|----------|-------------|
| NR      | 202.4      | NR       | USEPA 2015a |
| NR      | 321        | NR       | PPDB 2015   |
| NR      | 207.6      | NR       | USEPA 2015a |

Table 2 fipronil hydrolysis, photolysis, and biodegradation.

Values are for fipronil unless otherwise specified. NR: not reported.

|                      | Half- life (d)       | Water                                      | Temp (°C)       | pH                | Reference             |
|----------------------|----------------------|--|-----------------|-------------------|-----------------------|
| Hydrolysis           | 770 <sup>a</sup>     | Aqueous methanol solution (2.5 % in water) | 22 <sup>a</sup> | 9.0 <sup>a</sup>  | Bobè 1998             |
|                      | 114 <sup>b</sup>     |  | 22 <sup>b</sup> | 10.0 <sup>b</sup> |                       |
|                      | 11 <sup>c</sup>      |  | 22 <sup>c</sup> | 11.0 <sup>c</sup> |                       |
|                      | 2.4 <sup>d</sup>     |  | 22 <sup>d</sup> | 12.0 <sup>d</sup> |                       |
|                      | 75 <sup>e</sup>      |  | 30 <sup>e</sup> | 10.0 <sup>e</sup> |                       |
|                      | 43 <sup>f</sup>      |  | 37 <sup>f</sup> | 10.0 <sup>f</sup> |                       |
|                      | 18 <sup>g</sup>      |  | 45 <sup>g</sup> | 10.0 <sup>g</sup> |                       |
|                      | 55190 <sup>a,1</sup> |  | Aqueous buffer  | 5 <sup>a</sup>    |                       |
|                      | 39794 <sup>b,1</sup> | 22 <sup>b</sup>                            |                 | 7.1 <sup>2</sup>  |                       |
|                      | 609 <sup>c,1</sup>   | 32 <sup>c</sup>                            |                 | 9.1 <sup>3</sup>  |                       |
|                      | 92 <sup>d,1</sup>    | 50 <sup>d</sup>                            |                 |                   |                       |
|                      | 63895 <sup>a,2</sup> |  |                 |                   |                       |
|                      | 33367 <sup>b,2</sup> |  |                 |                   |                       |
|                      | 374 <sup>c,2</sup>   |  |                 |                   |                       |
| 81 <sup>d,2</sup>    |                      |  |                 |                   |                       |
| 48816 <sup>a,3</sup> |                      |  |                 |                   |                       |
| 26489 <sup>b,3</sup> |                      |  |                 |                   |                       |
| 270 <sup>c,3</sup>   |                      |  |                 |                   |                       |
| 48 <sup>d,3</sup>    |                      |  |                 |                   |                       |
| Aqueous Photolysis   | 0.33                 | 0.90 mg/L acetonitrile in deionized water  | 25              | 5                 | Corgier and Plewa1992 |

|                          |   |                                |       |   |                      |
|--------------------------|---|--------------------------------|-------|---|----------------------|
|                          | 36.7 h  | Rice paddy water               | 17-19 | NR                                      | Thuyet 2011          |
| Biodegradation (aerobic) | Sandy loam: 128<br>Sand: 308  | Soils                          | 25    | Sandy loam: 7.8<br>Sand: 6.1            | Waring 1993          |
|                          | Sandy loam: 74.8<br>Sandy loam overlying water: 5.85<br>Sandy loam/water system: 31.68<br>Sandy clay loam: 47.54<br>Sandy clay loam overlying water: 13.41<br>Sandy clay loam/water system: 21.20 | Sediments and associated water | 20    | Sandy loam: 8.2<br>Sandy clay loam: 6.8 | Ayliffe 1998         |
|                          | Sediment: 14.5<br>(partitioned into sediment)   | Sandy loam from pond           | 25    | 5.80                                    | Feung and Yenne 1997 |
|                          | <i>Fipronil-desulfinyl</i><br>Loamy sand I: 630<br>Loamy sand II: 693   | Soils                          | 25    | Loamy sand I: 5.8<br>Loamy sand II: 6.2 | Mislankar 1997       |
|                          |   |                                |       |   |                      |

|                               |   |  |    |   |              |
|-------------------------------|---|--|----|---|--------------|
| Biodegradation<br>(anaerobic) | 15-21   | Sediment                                       | 23 | NR  | Brennan 2009 |
|                               | Clay loam:<br>6.3, 431<br><br>Clay: 5.0, 296<br><br>Faster, slower<br>reactions<br>regulated by<br>desorption<br>from soil                  | Rice paddy<br>sediment and<br>irrigation water | NR | NR  | Doran 2009   |
|                               | Sandy loam:<br>5.0<br><br>Sand: 4.6<br><br>Loamy sand:<br>18.5  | Soils  | 21 | Sandy<br>loam: 6.7<br><br>Sand: 8.0<br><br>Loamy<br>sand: 7.6 | Lin 2008     |
|                               | Loamy sand<br><br><i>Fipronil-<br/>sulfide</i> : 589<br><br><i>Fipronil-<br/>sulfone</i> : 712<br><br><i>Fipronil-<br/>desulfinyl</i> : 388 | Soil   | 21 | 7.6   | Lin 2009     |

#### 4 Human and wildlife dietary values

There are no FDA action levels for fipronil (USFDA 2000), but food tolerances are provided for human consumption of various produce and meat commodities, ranging from 0.005 to 1.50 mg/kg (USEPA 2015b). There are currently no food tolerances for the human consumption of fish products.

### Wildlife LC<sub>50</sub> values (dietary) for animals with significant food sources in water

The US EPA Ecological Risk Assessment for Fipronil Uses (USEPA 2007) includes limited data on fipronil toxicity to mallard duck. The reported acute oral LC<sub>50</sub> for fipronil for mallard exceeds 2,150 mg/kg (Pedersen 1993a, rated L by the UCDM) and the acute dietary LC<sub>50</sub> exceeds 5,000 mg/kg (Pedersen 1993b, rated R by the UCDM). No LC<sub>50</sub> data for fipronil was available for wildlife species with significant food sources in water during the present report preparation. If highly rated measured data for mallard duck become available in the future, they should be examined to determine the potential risk to wildlife.

Only one definitive value was reported for fipronil desulfinyl at 437 mg/kg for oral acute toxicity (Helsten and Solatycki 1994).

### Wildlife dietary NOEC values for animals with significant food sources in water

The Ecological Risk Assessment (USEPA 2007) reports a NOEC value of 1000 mg/kg for fipronil (Pedersen and Lesar 1993), the highest concentration tested. The Pedersen study (1993b) reported a NOEC of 1250 mg/kg for fipronil. No other NOEC data was available for wildlife species with significant food sources in water during the present report preparation. If highly rated measured data for mallard duck become available in the future, they should be examined to determine the potential risk to wildlife.

There were no NOEC values available for any fipronil degradates for wildlife species with significant food sources in water during the present report preparation. If highly rated measured data for mallard duck become available in the future, they should be examined to determine the potential risk to wildlife.

## **5 Ecotoxicity data**

Aquatic and sediment toxicity effects studies were identified in the peer-reviewed open literature and from unpublished studies submitted to the USEPA and CDPR for fipronil and all degradates. Each study was reviewed according to the UCDM or UCDSM paradigms to determine the usefulness of these studies for water or sediment quality criteria derivation, respectively. Studies were divided into three categories to be rated: (1) single-species effects, (2) ecosystem-level studies, and (3) terrestrial wildlife studies.

The UCDM and UCDSM provide detailed numeric rating schemes for single-species effects studies that assigns (1) a relevance score and (2) a reliability score, which are summarized in TenBrook et al. (2009) and Fojut et al. (2014). The possible relevance scores were relevant (R), less relevant (L), or not relevant (N). The studies rated N were deemed irrelevant for criteria derivation and only the relevant (R) and less relevant (L) studies were evaluated for reliability. For all studies, study details and scoring were summarized in data summary sheets (Appendices

A-D). The reliability evaluation assigned possible scores of reliable (R), less reliable (L), or not reliable (N) so that each single-species study is described by a two-letter code, corresponding to the relevance and reliability ratings. The only studies used directly in criteria calculations were those rated as relevant and reliable (RR), which are summarized in Tables 3-7 and Tables 9-12 for aqueous studies and Tables 15-21 for sediment studies. Studies that were rated as relevant and less reliable (RL), less relevant and reliable (LR), or less relevant and less reliable (LL) were used to evaluate the derived criteria against data for any particularly sensitive, threatened, or endangered species found in these data sets. Studies that were rated N for either relevance or reliability were not considered in any aspect of criteria derivation.

Multispecies studies conducted in mesocosms, microcosms, and other field and laboratory ecosystems were rated for reliability. The results of the studies that were rated reliable (R) or less reliable (L) were compared to the derived criteria to ensure that they are protective of ecosystems. Studies of the effects of fipronil on mallard ducks were rated for reliability using the terrestrial wildlife evaluation. Mallard studies rated as reliable (R) or less reliable (L) were used to consider bioaccumulation of fipronil.

## 6 Data Prioritization

Multiple toxicity values for fipronil for the same species were reduced to one species mean toxicity value according to the data prioritization procedures described in the UCDM or UCDSM methodology reports.

### Aqueous data

The aqueous toxicity data that were reduced and the reasons for their exclusion are shown in Table 8. Reasons for reduction of data include: definitive toxicity values were available, more sensitive endpoints were available, and more sensitive timepoints were available.

The final acute data set for water quality criteria calculation for fipronil, fipronil-sulfide, fipronil-sulfone, fipronil-desulfinyl, and fipronil-carboxamide contain 17, 6, 15, 2, and 1 SMAVs, respectively (**Error! Reference source not found.-7**). In addition, there are two SMAVs each for the (+) and (-) enantiomers of fipronil. The final chronic data set for water quality criteria calculation contains 2 SMCVs for fipronil and one each for fipronil-sulfide, fipronil-sulfone, fipronil-desulfinyl, and the (-) enantiomer of fipronil (Tables 9-12).

### Sediment data

There were no sediment toxicity data that were reduced or excluded from the final data set. The final acute data set for bioavailable sediment quality criteria calculation for fipronil, fipronil-sulfide, and fipronil-sulfone contained one SMAV each (Tables 15-17). The final

chronic bioavailable sediment data set contained one, three, two, and two SMCVs each for fipronil, fipronil-sulfide, fipronil-sulfone, and fipronil-desulfinyl, respectively (Tables 18-21).

## 7 Water quality criteria calculations

### 7.1 Acute water quality criteria

#### 7.1.1 Fipronil acute water quality criterion

At least five acceptable acute toxicity values were available and fulfilled the five taxa requirements of the species sensitivity distribution (SSD) procedure (section 3-3.1, TenBrook et al. 2009). The five taxa requirements are a warm water fish, a fish from the family Salmonidae, a planktonic crustacean, a benthic crustacean, and an insect. Acute values for fipronil were plotted in a histogram (Figure 3) and appear to be separated by phylum. However, the fit test results (below) verify that this bimodality does not affect the validity of the SSD.

The Burr Type III SSD procedure (section 3-3.2.1, TenBrook et al. 2009a) was used for the acute criterion calculation because more than eight acceptable acute toxicity values were available in the fipronil data set (Table 3). The Burr Type III SSD procedure was used to derive the median 5<sup>th</sup> percentile and the median 1<sup>st</sup> percentiles or the 95% confidence limits for these percentiles. The median 5<sup>th</sup> percentile is recommended for use in criteria derivation because it is the most robust of the distributional estimates (section 3-3.2, TenBrook et al. 2009a). The distribution fit parameters were not available from the current version of the software (BurrliOZ 2.0, CSIRO 2016).

The fit of the Burr III distribution from the BurrliOZ 2.0 software is shown in Figure 4. This distribution provided a satisfactory fit ( $\chi^2_{2n} = 0.1188$ ; Appendix A) according to the fit test based on cross validation and Fisher's combined test (section 3-3.2.4, TenBrook et al. 2009a), indicating that the data set is valid for criteria derivation. Because the toxicity data used to calculate the criterion only reported two significant figures, the criterion is rounded to two significant figures (section 3-3.2.6, TenBrook et al. 2009a).

5<sup>th</sup> percentile, 50% confidence limit: 0.027 µg/L

5<sup>th</sup> percentile, 95% confidence limit: 0.0013 µg/L

1<sup>st</sup> percentile, 50% confidence limit: 0.0085 µg/L

1<sup>st</sup> percentile, 95% confidence limit: 0.0038 µg/L

Recommended acute value = 0.027 µg/L (median 5th percentile)

$$\begin{aligned}
 \text{Acute criterion} &= \text{acute value} \div 2 \\
 &= 0.027 \mu\text{g/L} \div 2 \\
 &= 0.0135 \mu\text{g/L} \\
 &= 0.014 \mu\text{g/L}
 \end{aligned}$$

**Acute criterion for fipronil** = 0.014  $\mu\text{g/L}$  = 14  $\text{ng/L}$

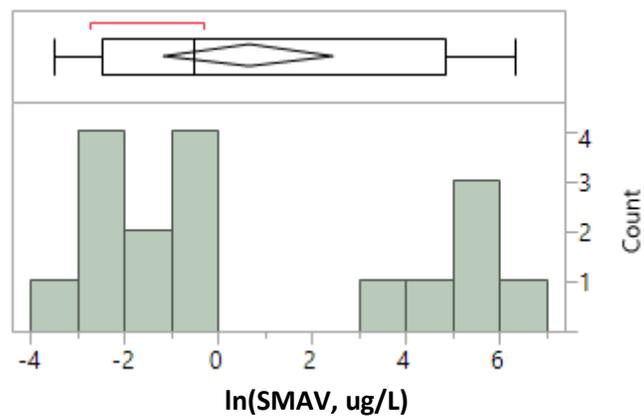


Figure 3 Histogram of acceptable acute aqueous fipronil data.

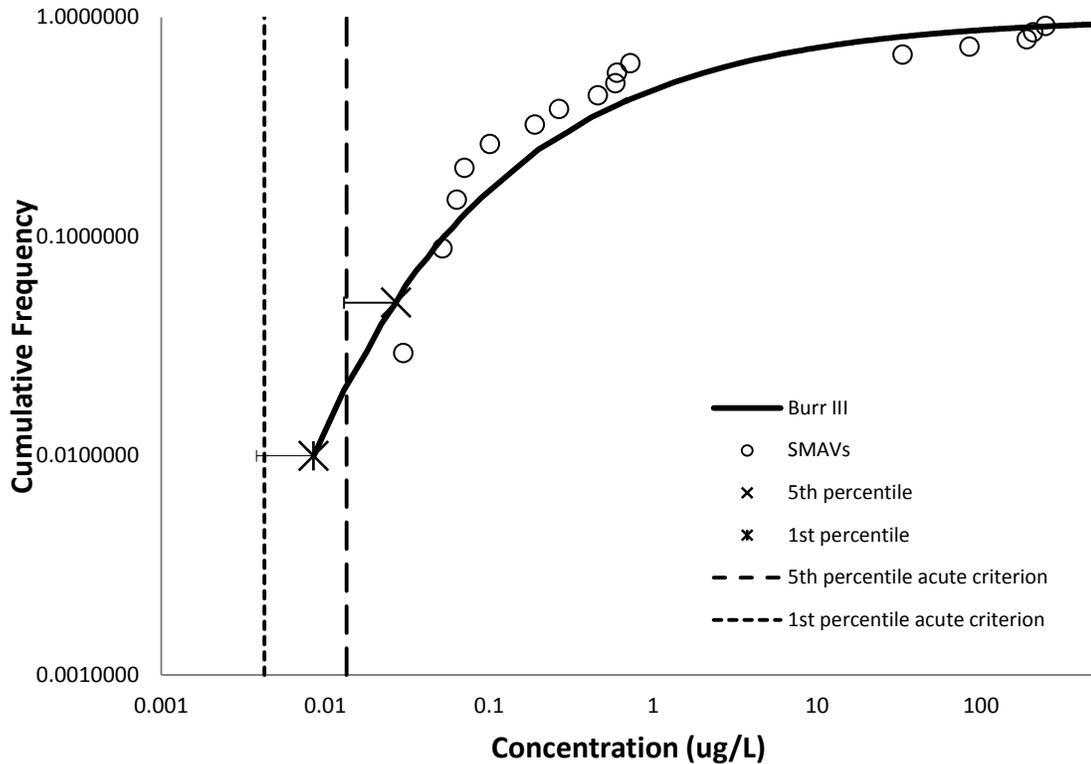


Figure 4 Fit of the Burr Type III distribution of fipronil to the acute aqueous data set.

The criterion and percentiles are shown. The median 5<sup>th</sup> percentile acute value and the median 1<sup>st</sup> percentile acute value are each displayed with their respective lower 95% confidence limit. The acute water quality criterion calculated with the median 5<sup>th</sup> percentile value is displayed as a vertical line.

### 7.1.2 Fipronil-sulfide acute water quality criterion

Acceptable acute toxicity values were not available from the five required taxa for a species sensitivity distribution. The Assessment Factor (AF) procedure can be used to calculate the acute criterion in such data sets, when at least one of the values is from the family Daphniidae (section 3-3.3, TenBrook et al. 2009a). The fipronil-sulfide data set contains a toxicity value for *Daphnia magna*, which meets this requirement. Fipronil is an organic pesticide, and the AFs given in the methodology (Table 3.13, TenBrook et al. 2009a) are the most specific AFs available for organic pesticides. The methodology points out that the AFs are limited in that they are based on organochlorine and organophosphate, and pyrethroid pesticides, which are neurotoxic insecticides, while fipronil is an organofluorine neurotoxic insecticide that blocks and overstimulates the nervous and muscular systems. However, fipronil and its degradates do exhibit toxicity to other animals and to plants with an unclear mechanism and are organic pesticides, thus, it is reasonable to use the AF procedure for fipronil and its degradates.

The AFs given in the methodology will be used for fipronil-sulfide with the understanding that AFs based on measured pesticide toxicity data are likely more accurate than choosing an arbitrary AF. The methodology points out that AFs are recognized as a conservative approach for dealing with uncertainty in assessing risks posed by chemicals (section 2-3.2, TenBrook et al. 2009a). Using an AF to calculate a criterion always involves a high degree of uncertainty and there is potential for under- or over-protection, which is strongly dependent on the representation of sensitive species in the available data set. The methodology instructs that the derived criterion should be compared to all available ecotoxicity data to ensure that it will be protective of all species (section 3-6.0, TenBrook et al. 2009a).

Only three of the five taxa requirements necessary to fit a SSD were available for fipronil-sulfide, thus an assessment factor was used to derive the acute criterion. The benthic crustacean requirement was fulfilled by the *H. azteca*, the planktonic crustacean requirement was fulfilled by *D. magna* and the remaining species were insects. The two missing taxa include a fish of the family Salmonidae and a warm water fish. The AF method calculates the criterion by dividing the lowest SMAV from the acceptable (RR) data set by an AF, which is determined by the number of taxa available in the data set (section 3-3.3, TenBrook et al. 2009a). The lowest SMAV was the 96-h *C. dilutus* EC<sub>50</sub> value of 0.0093 µg/L. This value was divided by an AF of 8 because there are acceptable data from three taxa (Table 17, Fojut et al. 2014). The acute value calculated using the AF represents an estimate of the median 5<sup>th</sup> percentile value of the SSD, which is the recommended acute value. The recommended acute value is divided by a factor of 2 to calculate the acute criterion (section 3-3.3, TenBrook et al. 2009a). Because the toxicity data used to calculate the criterion only reported two significant figures, the criterion is rounded to two significant figures (section 3-3.2.6, TenBrook et al. 2009a).

$$\begin{aligned}\text{Acute value} &= \text{lowest value in data set} \div \text{assessment factor} \\ &= 0.0093 \mu\text{g/L} \div 8 \\ &= 0.00116 \mu\text{g/L}\end{aligned}$$

$$\begin{aligned}\text{Acute criterion} &= \text{acute value} \div 2 \\ &= 0.00116 \mu\text{g/L} \div 2 \\ &= 0.00058 \mu\text{g/L} \\ &= 0.58 \text{ ng/L}\end{aligned}$$

**Acute criterion for fipronil-sulfide = 0.58 ng/L**

### 7.1.3 Fipronil-sulfone acute water quality criterion

At least five acceptable acute toxicity values for fipronil-sulfone were available and fulfilled the five taxa requirements of the species sensitivity distribution (SSD) procedure (section 3-3.1, TenBrook et al. 2009). The five taxa requirements are a warm water fish, a fish from the family Salmonidae, a planktonic crustacean, a benthic crustacean, and an insect. Acute values for fipronil were plotted in a histogram (Figure 3).

The Burr Type III SSD procedure (section 3-3.2.1, TenBrook et al. 2009a) was used for the acute criterion calculation because more than eight acceptable acute toxicity values were available in the fipronil-sulfone data set (Table 5). The Burr Type III SSD procedure was used to derive the median 5<sup>th</sup> percentile and the median 1<sup>st</sup> percentiles or the 95% confidence limits for these percentiles. The median 5th percentile is recommended for use in criteria derivation because it is the most robust of the distributional estimates (section 3-3.2, TenBrook et al. 2009a).

The BurrliOZ 2.0 software program (CSIRO 2016) was used to fit a Burr III distribution to the data set. This distribution did not provide a satisfactory fit according to the fit test described in section 3-3.2.4 of TenBrook et al. (2009). The  $\chi^2_{2n}$  statistic using the fit test based on cross validation and Fisher's combined test could not be calculated, indicating that the fit of the distribution to the data set is not valid for criteria derivation (Appendix D: Acute WQC Fit Test for fipronil-sulfone, Burr III SSD). All values were confirmed from the original, highly rated studies and do not appear to be erroneous. The distribution fit parameters were not available from the current version of the software.

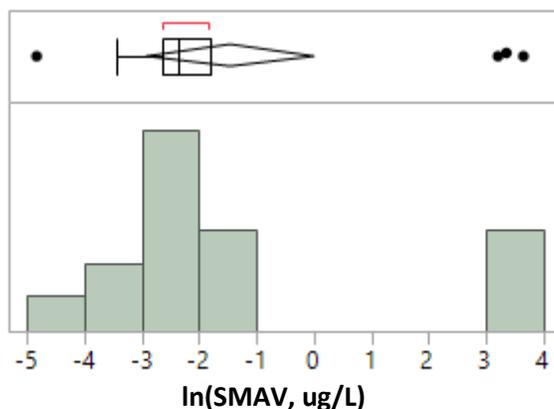


Figure 5 Histogram of acceptable acute aqueous fipronil-sulfone data.

Because the Burr Type III distribution did not provide a satisfactory fit to the data set, a log-logistic distribution was used instead because it contains fewer fitting parameters (section 3-3.2.5., TenBrook et al. 2009). The log-logistic SSD procedure (section 3-3.2.2, TenBrook et al. 2009) was used to derive 5<sup>th</sup> percentile values (median and lower 95% confidence limit), as well

as 1<sup>st</sup> percentile values (median and lower 95% confidence limit). The median 5<sup>th</sup> percentile value is recommended for use in criteria derivation by the methodology because it is the most robust of the distributional estimates (section 3-3.2, TenBrook et al. 2009). Comparing the median estimate to the lower 95% confidence limit of the 5<sup>th</sup> percentile values, it can be seen that the second significant figures of the two values are different (0.0002185 and 0.0026770 µg/L). Because there is uncertainty in the second significant digit, the final criterion will be reported with two significant digits (section 3- 3.2.6, TenBrook et al. 2009).

The ETX 1.3 Software program (Aldenberg 1993) was used to fit a log-logistic distribution to the data set, which is plotted with the acute values in Figure 6. This distribution provided a satisfactory fit according to the fit test described in section 3-3.2.4 of TenBrook et al. (2009). No significant lack of fit was found ( $\chi^2_{2n} = 0.3615$ ) using the fit test based on cross validation and Fisher's combined test (Appendix E – Acute WQC Fit Test for fipronil-sulfone, log logistic), indicating that the data set is valid for criteria derivation.

### **Log-logistic distribution**

HC5 Fitting Parameter Estimates:  $\alpha = -0.631$ ,  $\beta$  (median) = 0.6593,  $\beta$  (lower 95% CI) = 1.0394.

5<sup>th</sup> percentile, 50% confidence limit: 0.0026770 µg/L

5<sup>th</sup> percentile, lower 95% confidence limit: 0.0002035 µg/L

1<sup>st</sup> percentile, 50% confidence limit: 0.0002185 µg/L

1<sup>st</sup> percentile, lower 95% confidence limit: 0.0000039 µg/L

Recommended acute value = 0.0026770 µg/L (median 5th percentile value)

Acute WQC = Recommended acute value ÷ 2

$$= 0.0026770 \mu\text{g/L} \div 2$$

$$= 0.0013385 \mu\text{g/L}$$

**Acute WQC** = 0.0013 µg/L

$$= 1.3 \text{ ng/L}$$

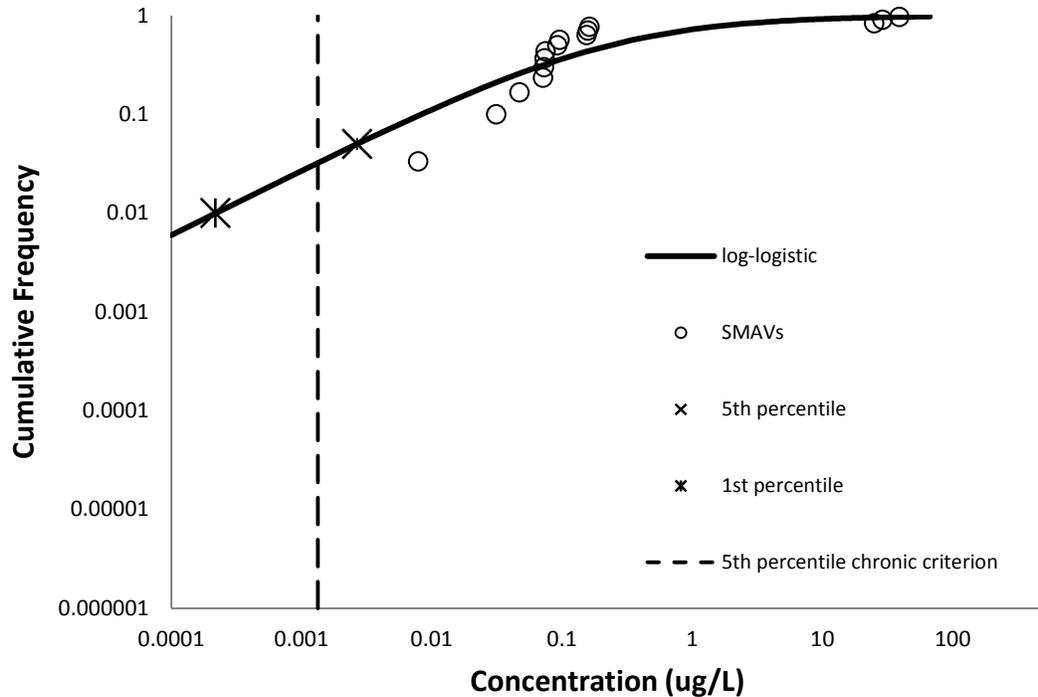


Figure 6 The fit of the log-logistic distribution to the acute aqueous data set.

The median 5<sup>th</sup> percentile acute value with the lower 95% confidence limit and the median 1<sup>st</sup> percentile acute value are each displayed. The acute water quality criteria calculated with the median 5<sup>th</sup> percentile value is displayed as a vertical line.

#### 7.1.4 Fipronil-desulfinyl acute water quality criterion

An acute criterion could not be calculated for fipronil-desulfinyl. Acceptable acute toxicity values were not available from the five taxa required to use a species sensitivity distribution. Only two acute toxicity values were available; however, they did not meet the requirements for criteria derivation using an Assessment Factor (AF). The methodology states that at least one of the acute toxicity values must be from the family Daphniidae or a criterion cannot be derived (section 3-3.3, TenBrook et al. 2009a). The available values were for fish species, which are not known to be particularly sensitive based on the data sets for fipronil, fipronil-sulfide, and fipronil-sulfone. Because neither a daphnid nor a known sensitive species is available for fipronil-desulfinyl, calculating an acute criterion with an assessment factor may not result in a criterion that is protective of aquatic organisms, and therefore an acute criterion is not calculated.

### 7.1.5 Fipronil-carboxamide acute water quality criterion

An acute criterion could not be calculated for fipronil-carboxamide. Only one acute toxicity value was available; however, it did not meet the requirements for criteria derivation using an Assessment Factor (AF). The methodology states that at least one of the acute data must be from one of three specific species in the family Daphniidae or a criterion cannot be derived (section 3-3.3, TenBrook et al. 2009a). The single acute value available was an LC<sub>50</sub> of 250 µg/L for a chironomid (*C. riparius*) from the Chironomidae family (Table 7), and is therefore not acceptable for the AF procedure.

## 7.2 Chronic water quality criteria

### 7.2.1 Fipronil chronic water quality criterion

Chronic toxicity values from fewer than five different families were available for fipronil, which does not meet the taxa requirements to fit a species sensitivity distribution. Instead, an acute-to-chronic ratio (ACR) was used to calculate a chronic water quality criterion for fipronil (section 3-4.2.2, TenBrook et al. 2009). The final acute and chronic data sets contained three paired species, however paired data for only one species met the requirements to calculate a species mean acute-to-chronic ratio (SMACR). The methodology states that paired data are acceptable to calculate an ACR if they were conducted as part of the same study or part of a different study in the same laboratory and dilution water. *Oncorhynchus mykiss* acute and chronic values were from different laboratories using different dilution water and therefore do not qualify for the ACR method. A single study with *Ceriodaphnia dubia* yielded both acute and chronic data from different generations (Wilson et al. 2008). The acute data was from a second generation that had been previously exposed to fipronil. Therefore, this data is not appropriate to calculate an ACR. McNamara tested the toxicity of fipronil to *Daphnia magna* and reported a LC<sub>50</sub> of 190 µg/L (1990a) and a MATC of 14 µg/L (1990d) in different studies that used the same dilution water. This allowed for calculation of a species mean acute-to-chronic ratio (SMACR) for *D. magna*.

$$\text{SMACR} = \text{acute toxicity value} \div \text{chronic toxicity value}$$

$$D. magna \text{ SMACR} = 190 \mu\text{g/L} \div 14 \mu\text{g/L} = 13.6 \mu\text{g/L}$$

The final multispecies ACR was then calculated as the geometric mean of the *D. magna* SMACR and two default ACRs (section 3-4.2.2, TenBrook et al. 2009), as follows:

$$\text{Final multispecies ACR} = \text{geomean}(13.6, 11.4, 11.4) = 12.1$$

The chronic criterion was calculated using the recommended acute value and the final multispecies ACR as follows:

$$\begin{aligned} \text{Chronic criterion} &= \text{Recommended acute value} \div \text{final multispecies ACR} \\ &= 0.027 \mu\text{g/L} \div 12.1 \\ &= 0.00223 \mu\text{g/L} \end{aligned}$$

$$\begin{aligned} \text{Chronic criterion} &= 0.0022 \mu\text{g/L} \\ &= 2.2 \text{ ng/L} \end{aligned}$$

### 7.2.2 Fipronil-sulfide chronic water quality criterion

Chronic toxicity values from fewer than five different families were available for fipronil-sulfide, which does not meet the taxa requirements to fit a species sensitivity distribution. Instead, an acute-to-chronic ratio (ACR) was used to calculate a chronic water quality criterion for fipronil-sulfide (section 3-4.2.2, TenBrook et al. 2009). The final acute and chronic data sets contained a single paired species that met the requirements to calculate a species mean acute-to-chronic ratio (SMACR). The methodology states that paired data are acceptable to calculate an ACR if they were conducted as part of the same study or part of a different study in the same laboratory and dilution water. McNamara tested the toxicity of fipronil-sulfide to *Daphnia magna* and reported a LC<sub>50</sub> of 100 μg/L (1990b) and a MATC of 17 μg/L (1990e) in different studies that used the same dilution water. This allowed for calculation of a species mean acute-to-chronic ratio (SMACR) for *D. magna*.

$$\text{SMACR} = \text{acute toxicity value} \div \text{chronic toxicity value}$$

$$D. magna \text{ SMACR} = 100 \mu\text{g/L} \div 17 \mu\text{g/L} = 5.88 \mu\text{g/L}$$

The final multispecies ACR was then calculated as the geometric mean of the *D. magna* SMACR and two default ACRs (section 3-4.2.2, TenBrook et al. 2009), as follows:

$$\text{Final multispecies ACR} = \text{geomean}(5.88, 11.4, 11.4) = 9.14$$

The chronic criterion was calculated using the recommended acute value and the final multispecies ACR as follows:

$$\begin{aligned} \text{Chronic criterion} &= \text{Recommended acute value} \div \text{final multispecies ACR} \\ &= 0.00116 \mu\text{g/L} \div 9.14 \\ &= 0.000127 \mu\text{g/L} \end{aligned}$$

$$\begin{aligned} \text{Chronic criterion} &= 0.00013 \mu\text{g/L} \\ &= 0.13 \text{ ng/L} \end{aligned}$$

### 7.2.3 Fipronil-sulfone chronic water quality criterion

Two chronic toxicity values for *Daphnia magna* were available for fipronil-sulfone. The final and acute data sets contained one paired species. One of the values in the chronic data set met the requirements to calculate a SMACR. The methodology states that paired data are acceptable to calculate an ACR if they were conducted as part of the same study or part of a different study in the same laboratory and dilution water. One set of *Daphnia magna* acute and chronic values were from the same laboratory using the same dilution water and therefore qualify for the ACR method. McNamara tested the toxicity of fipronil-sulfone to *D. magna* and determined a 48 hour EC<sub>50</sub> of 29 µg/L (1990c) and a MATC of 0.97 µg/L (1992). This allowed for calculation of a species mean acute-to-chronic ratio (SMACR) for *D. magna*.

$$\text{SMACR} = \text{acute toxicity value} \div \text{chronic toxicity value}$$

$$D. magna \text{ SMACR} = 29 \mu\text{g/L} \div 0.97 \mu\text{g/L} = 29.9 \mu\text{g/L}$$

The final multispecies ACR was then calculated as the geometric mean of the *D. magna* SMACR and two default ACRs (section 3-4.2.2, TenBrook et al. 2009), as follows:

$$\text{Final multispecies ACR} = \text{geomean}(29.9, 11.4, 11.4) = 15.7$$

The chronic criterion was calculated using the recommended acute value and the final multispecies ACR as follows:

$$\begin{aligned} \text{Chronic criterion} &= \text{Recommended acute value} \div \text{final multispecies ACR} \\ &= 0.0026770 \mu\text{g/L} \div 15.7 \\ &= 0.00017 \mu\text{g/L} \end{aligned}$$

$$\begin{aligned} \text{Chronic criterion} &= 0.00017 \mu\text{g/L} \\ &= 0.17 \text{ ng/L} \end{aligned}$$

### 7.2.4 Fipronil-desulfinyl chronic water quality criterion

A chronic criterion could not be calculated for fipronil-desulfinyl because there are insufficient chronic data to fit a SSD and there is no acute value available from which a chronic criterion can be extrapolated. Only one chronic value was available, an MATC of 1.67 µg/L for *Daphnia magna*.

### 7.2.5 Fipronil-carboxamide chronic water quality criterion

A chronic criterion could not be calculated for fipronil-carboxamide because there are no chronic water toxicity data available and there is also no acute value available from which a chronic criterion can be extrapolated.

## 8 Interim bioavailable sediment quality criteria calculations

The UC Davis Sediment Methodology (UCDSM) is considered only a framework and not a final method because large diverse data sets were not available to use in the development of the method. For this reason, the resulting bioavailable sediment quality criteria (BSQC) are termed interim values, and are not recommended for use as firm regulatory values. The term interim is used because there is a high degree of uncertainty in the values because they are based on so few data and species. In order to aid the method development process, sediment toxicity data were gathered for fipronil and interim BSQC were calculated. The interim BSQC calculations are described to provide information to environmental managers, but are not intended to be used as regulatory values.

### 8.1 Interim acute bioavailable sediment quality criteria

#### 8.1.1 Fipronil interim acute bioavailable sediment quality criterion

Two acute toxicity values are available for fipronil. In order to use the Assessment Factor (AF) procedure, the methodology requires that at least one of the acute data must be a benthic crustacean (section 3.5.2, Fojut et al. 2014). There is a toxicity value for *Hyalella azteca* that meets the benthic crustacean requirement. To calculate the interim BSQC with an assessment factor, the lowest acute value is divided by an AF. The lowest acute value in the data set is an EC<sub>50</sub> of 0.10 µg/g OC for *Chironomus dilutus* (Table 16). This value was divided by an AF of 12 because there are acceptable data from two taxa (Table 17, Fojut et al. 2014). The interim acute BSQC is rounded to two significant figures because the toxicity value used to calculate the criterion reports two significant figures.

$$\begin{aligned}\text{Acute value} &= \text{lowest value in data set} \div \text{assessment factor} \\ &= 0.10 \text{ } \mu\text{g/g OC} \div 12 \\ &= 0.008333 \text{ } \mu\text{g/g OC}\end{aligned}$$

$$\begin{aligned}\text{Interim acute BSQC} &= \text{acute value} \div 2 \\ &= 0.008333 \text{ } \mu\text{g/g OC} \div 2 \\ &= 0.004167 \text{ } \mu\text{g/g OC}\end{aligned}$$

$$= 4.2 \text{ ng/g OC}$$

### 8.1.2 Fipronil-sulfide interim acute bioavailable sediment quality criterion

Two acute toxicity values are available for fipronil-sulfide. In order to use the Assessment Factor (AF) procedure, the methodology requires that at least one of the acute data must be a benthic crustacean (section 3.5.2, Fojut et al. 2014). There is a toxicity value for *Hyalella azteca* that meets the benthic crustacean requirement. To calculate the interim BSQC with an assessment factor, the lowest acute value is divided by an AF. The lowest acute value in the data set is an EC<sub>50</sub> of 0.06 µg/g OC for *Chironomus dilutus* (Table 16). This value was divided by an AF of 12 because there are acceptable data from two taxa (Table 17, Fojut et al. 2014). The interim acute BSQC is rounded to one significant figure because the toxicity value used to calculate the criterion reports one significant figure.

$$\begin{aligned} \text{Acute value} &= \text{lowest value in data set} \div \text{assessment factor} \\ &= 0.06 \text{ } \mu\text{g/g OC} \div 12 \\ &= 0.005 \text{ } \mu\text{g/g OC} \end{aligned}$$

$$\begin{aligned} \text{Interim acute BSQC} &= \text{acute value} \div 2 \\ &= 0.005 \text{ } \mu\text{g/g OC} \div 2 \\ &= 0.0025 \text{ } \mu\text{g/g OC} \\ &= 3 \text{ ng/g OC} \end{aligned}$$

### 8.1.3 Fipronil-sulfone interim acute bioavailable sediment quality criterion

Two acute toxicity values are available for fipronil-sulfone. In order to use the Assessment Factor (AF) procedure, the methodology requires that at least one of the acute data must be a benthic crustacean (section 3.5.2, Fojut et al. 2014). There is a toxicity value for *Hyalella azteca* that meets the benthic crustacean requirement. To calculate the interim BSQC with an assessment factor, the lowest acute value is divided by an AF. The lowest acute value in the data set is an EC<sub>50</sub> of 0.04 µg/g OC for *Chironomus dilutus* (Table 17). This value was divided by an AF of 12 because there are acceptable data from two taxa (Table 17, Fojut et al. 2014). The interim acute BSQC is rounded to one significant figure because the toxicity value used to calculate the criterion reports one significant figure.

$$\text{Acute value} = \text{lowest value in data set} \div \text{assessment factor}$$

$$= 0.04 \mu\text{g/L} \div 12$$

$$= 0.00333 \mu\text{g/g OC}$$

$$\text{Interim acute BSQC} = \text{acute value} \div 2$$

$$= 0.00333 \mu\text{g/g OC} \div 2$$

$$= 0.00166 \mu\text{g/g OC}$$

$$= 2 \text{ ng/g OC}$$

#### 8.1.4 Fipronil-desulfinyl interim acute bioavailable sediment quality criterion

Two acute toxicity values are available for fipronil-desulfinyl. In order to use the Assessment Factor (AF) procedure, the methodology requires that at least one of the acute data must be a benthic crustacean (section 3.5.2, Fojut et al. 2014). There is a toxicity value for *Hyalella azteca* that meets the benthic crustacean requirement. To calculate the interim BSQC with an assessment factor, the lowest acute value is divided by an AF. The lowest acute value in the data set is an EC<sub>50</sub> of 28 μg/g OC for *Chironomus dilutus* (Table 17). This value was divided by an AF of 12 because there are acceptable data from two taxa (Table 17, Fojut et al. 2014). The interim acute BSQC is rounded to two significant figure because the toxicity value used to calculate the criterion reports two significant figure.

$$\text{Acute value} = \text{lowest value in data set} \div \text{assessment factor}$$

$$= 28 \mu\text{g/L} \div 12$$

$$= 2.33 \mu\text{g/g OC}$$

$$\text{Interim acute BSQC} = \text{acute value} \div 2$$

$$= 2.33 \mu\text{g/g OC} \div 2$$

$$= 1.166 \mu\text{g/g OC}$$

$$= 1.2 \text{ ng/g OC}$$

#### 8.1.5 Fipronil-carboxamide interim acute bioavailable sediment quality criterion

An acute criterion could not be calculated for fipronil-carboxamide because there are no acute sediment toxicity data available.

## 8.2 *Interim chronic bioavailable sediment quality criteria*

### 8.2.1 *Fipronil interim chronic bioavailable sediment quality criterion*

There were no chronic sediment toxicity data available for fipronil so a chronic BSQC cannot be calculated with a species sensitivity distribution or an empirical acute-to-chronic ratio. A chronic interim BSQC is calculated with a default ACR of 11.4 (Fojut et al. 2014) and the recommended acute value from the interim acute BSQC calculation.

$$\begin{aligned}\text{Interim chronic BSQC} &= \text{Recommended acute value} \div \text{ACR} \\ &= 0.008333 \mu\text{g/g OC} \div 11.4 \\ &= 0.00073 \mu\text{g/g OC} \\ &= 0.7 \text{ ng/g OC}\end{aligned}$$

### 8.2.2 *Fipronil-sulfide interim chronic bioavailable sediment quality criterion*

One chronic sediment toxicity value was available for fipronil-sulfide, a 28-d MATC of 0.16  $\mu\text{g/g OC}$  for *Chironomus riparius*. Due to insufficient chronic sediment data to fit a species sensitivity distribution or calculate empirical acute-to-chronic ratios, the default ACR is used to calculate the interim chronic BSQC (section 3.6.3 of the UCDSM). A chronic interim BSQC is calculated with a default ACR of 11.4 (Fojut et al. 2014) and the recommended acute value from the interim acute BSQC calculation.

$$\begin{aligned}\text{Interim chronic BSQC} &= \text{Recommended acute value} \div \text{ACR} \\ &= 0.005 \mu\text{g/g OC} \div 11.4 \\ &= 0.000439 \mu\text{g/g OC} \\ &= 0.4 \text{ ng/g OC}\end{aligned}$$

### 8.2.3 *Fipronil-sulfone interim chronic bioavailable sediment quality criterion*

There were no chronic sediment toxicity data available for fipronil-sulfone so a chronic BSQC cannot be calculated with a species sensitivity distribution or an empirical acute-to-chronic ratio. A chronic interim BSQC is calculated with a default ACR of 11.4 (Fojut et al. 2014) and the recommended acute value from the interim acute BSQC calculation.

$$\begin{aligned}\text{Interim chronic BSQC} &= \text{Recommended acute value} \div \text{ACR} \\ &= 0.00333 \mu\text{g/g OC} \div 11.4\end{aligned}$$

$$= 0.000292 \mu\text{g/g OC}$$

$$= 0.3 \text{ ng/g OC}$$

#### 8.2.4 *Fipronil-delsulfinyl interim chronic bioavailable sediment quality criterion*

There were no chronic sediment toxicity data available for fipronil-desulfinyl so a chronic BSQC cannot be calculated with a species sensitivity distribution or an empirical acute-to-chronic ratio. A chronic interim BSQC is calculated with a default ACR of 11.4 (Fojut et al. 2014) and the recommended acute value from the interim acute BSQC calculation.

$$\text{Interim chronic BSQC} = \text{Recommended acute value} \div \text{ACR}$$

$$= 2.33 \mu\text{g/g OC} \div 11.4$$

$$= 0.204 \mu\text{g/g OC}$$

$$= 0.20 \text{ ng/g OC}$$

#### 8.2.5 *Fipronil-carboxamide interim chronic bioavailable sediment quality criterion*

A chronic criterion could not be calculated for fipronil-carboxamide because there are no chronic sediment toxicity data available and there is also no acute value available from which a chronic criterion can be extrapolated.

## 9 Water Quality Effects

### 9.1 *Bioavailability*

No studies were found concerning the bioavailability of fipronil, its degradates, or its enantiomers in the water column that differentiates when these compounds are sorbed to solids, sorbed to dissolved solids, or freely dissolved. Until there is more information that discusses the bioavailability of these three phases, it is recommended that compliance is based on the total concentration of fipronil, its degradates, and its enantiomers in water (section 3-5.1, TenBrook et al. 2009a).

## 9.2 Mixtures

Fipronil can occur in the environment with other pesticides, of similar or different modes of action. Fipronil is a phenylpyrazole insecticide that causes hyperexcitation of insect nerve and muscle systems by blocking GABA-gated chloride channels and glutamate-gated chloride (GluCl) channels. The concentration addition model and the non-additive interaction model are the only predictive mixture models recommended by the methodology (section 3-5.2, TenBrook et al. 2009a), so other models found in the literature will not be considered for compliance.

Few studies were available that tested the toxicity of fipronil in mixtures to aquatic species. Key et al. (2007) studied three pesticides alone and in binary and ternary mixtures to the saltwater shrimp *Palaemonetes pugio*. Fipronil was tested alone and in combination with atrazine and imidacloprid. Atrazine is a photosystem II inhibitor in plants and imidacloprid is an insecticide that inhibits acetylcholine function in the nervous system. Binary mixtures of fipronil-atrazine were no more toxic than the pesticides tested alone whereas fipronil-imidacloprid mixtures were additive (1.8 times more toxic to larvae). Ternary mixtures resulted in greater than additive toxicity of fipronil to the shrimp (2.4 times more toxic to larvae).

Lizotte et al. (2009) tested the effects of pesticide mixtures in a mesocosm study to *Hyalella azteca*. Commercial formulations of fipronil, atrazine, and metolachlor were used. Mixture toxicity occurred due to fipronil and fipronil-sulfone that was produced naturally within the system but toxicity models were not fit to the data.

No studies on aquatic organisms were identified in the literature that could provide a quantitative means to consider mixtures of fipronil with other classes of pesticides.

## 9.3 Temperature, pH, and other water quality effects

Temperature, pH, and other water quality effects on the toxicity of fipronil were examined to determine if any effects are described well enough in the literature to incorporate into criteria compliance (section 3-5.3, TenBrook et al. 2009). Only one study was available that studied the effects of some compounds typically present in natural waters. Walse et al. (2004) showed that DOM reduced the photodegradation of fipronil into fipronil-desulfinyl through energetic quenching and by effectively blocking the light. Fipronil-desulfinyl production decreased by more than 27% between DOM loadings of 15 to 25 mg/L. However, under increased salinity at levels similar to marine systems, it was shown that fipronil degradation to fipronil-desulfinyl increased by 20% over salt-free systems. This study shows that water quality parameters have a dynamic effect on the fate of fipronil in aquatic systems, which can lead to the formation of toxic degradates.

## 10 Comparison of ecotoxicity data to derived criteria

### 10.1 Sensitive species

A data comparison was conducted to assess if the derived criteria for fipronil are protective of the most sensitive species. The derived WQC are compared to toxicity values for the most sensitive species in both the acceptable (RR) and supplemental (RL, LR, LL) data sets. Similarly, the interim BSQC are compared to toxicity values for the most sensitive species in both the acceptable (RR) and supplemental (RL, LR, LL) data sets.

#### 10.1.1 Fipronil

The lowest acute toxicity value in the fipronil aqueous data set is the LC<sub>50</sub> value of 0.43 ng/L for both *Aedes taeniorhynchus* and *Anopheles quadrimaculatus*, two mosquito species (Ali 1998). The acute WQC of 14 ng/L is two orders of magnitude greater than this LC<sub>50</sub> and would therefore not be protective of these species. Other *Aedes* species are similarly sensitive to fipronil (Ali 1998 and Chaton 2001). The next lowest value is an LC<sub>50</sub> of 0.91 g/L for the midge *Glyptotendipes paripes* (Ali 1998). The studies by Ali and Chaton rated RL due to low reliability scores and missing control responses. Missing control responses makes it difficult to interpret the toxicity results in a study. In addition, the UCDM guidance is that criteria should only be adjusted based on data for sensitive species if the toxicity value is based on measured concentrations, which is not the case for any of these toxicity data. Therefore, the acute WQC for fipronil will not be adjusted to be protective of these insect species.

The lowest chronic toxicity value for fipronil is a MATC of 11 ng/L for the saltwater mysid *Americamysis bahia* based on 28-d survival (Machado 1995). The chronic WQC of 2.4 ng/L is below this value and would be protective of this species.

The lowest reported acute sediment toxicity value for fipronil in all data sets is the 10-d EC<sub>50</sub> of 0.10 µg/g OC for the chironomid *Chironomus dilutus* (Maul 2008). The interim acute BSQC of 0.0042 µg/g OC was calculated based on this toxicity value, and is therefore protective of this species. There were no chronic sediment toxicity values available to compare to the interim chronic BSQC for fipronil.

#### 10.1.2 Fipronil-sulfide

The lowest acute toxicity value in the fipronil-sulfide aqueous data set is the LC<sub>50</sub> value of 9.3 ng/L for the chironomid *Chironomus dilutus* (Weston 2014). The acute WQC of 0.58 ng/L is one order of magnitude lower than this LC<sub>50</sub> and would therefore be protective of this species.

The lowest chronic aqueous toxicity value is a MATC of 0.47 µg/L for the mysid *Americamysis bahia* based on 96-h survival (Putt 2000a). The chronic WQC of 0.10 ng/L is well below this value and would therefore be protective of this species.

The lowest reported acute sediment toxicity value in all data sets is the 10-d EC<sub>50</sub> of 0.06 µg/g OC for the chironomid *Chironomus dilutus* (Maul 2008). The interim acute BSQC of 0.003 µg/g OC was calculated based on this toxicity value, and is therefore protective of this species. The lowest reported chronic sediment toxicity value is the 28-d MATC of 0.16 µg/g OC for *Chironomus riparius*; this was also the only chronic sediment toxicity value available for any of the fipronil compounds. The interim chronic BSQC of 0.0004 µg/g OC is well below this toxicity value and would be protective of this species.

### 10.1.3 Fipronil-sulfone

The lowest acute toxicity value in the fipronil-sulfone aqueous data set is the LC<sub>50</sub> value of 7.9 ng/L for the chironomid *Chironomus dilutus* (Weston 2014). The acute WQC of 1.3 ng/L is below this LC<sub>50</sub> and would therefore be protective of this species.

The lowest chronic aqueous toxicity value is a MATC of 0.65 µg/L for *Daphnia magna* based on 21-d survival (McNamara 1990e). The chronic WQC of 0.24 ng/L is well below this value and would therefore adequately protect this species.

The lowest reported acute sediment toxicity value in all data sets is the 10-d EC<sub>50</sub> of 0.04 µg/g OC for the chironomid *Chironomus dilutus* (Maul 2008). The interim acute BSQC of 0.002 µg/g OC was calculated based on this toxicity value, and is therefore protective of this species. There were no chronic sediment toxicity values available to compare to the interim chronic BSQC for fipronil-sulfone.

### 10.1.4 Fipronil-desulfinyl

The lowest acute toxicity value in the fipronil-desulfinyl aqueous data set is the LC<sub>50</sub> value of 20 µg/L for the bluegill sunfish *Leopmis macrochirus* (Collins 1993a). The lowest chronic aqueous toxicity value is a MATC of 0.1500 µg/L for the saltwater mysid *Americamysis bahia* based on 96-h survival (Putt 1992a). These values cannot be compared to criteria because there were insufficient data to calculate an acute or chronic water quality criterion for fipronil-desulfinyl.

The lowest acute sediment toxicity value for fipronil-desulfinyl in all data sets is the 10-d LC<sub>50</sub> of 8.3 µg/g OC for the amphipod *Leptocheirus plumulosus* (Picard 2015g). This study was supplemental because it is a saltwater species. The interim acute BSQC of 1.2 µg/g OC would be protective of this species. There were no chronic sediment toxicity values available to compare to the interim chronic BSQC for fipronil-desulfinyl.

### 10.1.5 Fipronil-carboxamide

The only toxicity value in all of the fipronil-carboxamide data sets is the acute aqueous LC<sub>50</sub> value of 250 µg/L for *Chironomus riparius* (Funk 2004). This value cannot be compared to an acute criterion because there were insufficient data to calculate an acute or chronic criterion for fipronil-carboxamide.

### 10.2 Ecosystem studies

The derived criteria are compared to acceptable laboratory, field, or semi-field multispecies studies (rated R or L) to determine if the criteria will be protective of ecosystems (section 3-6.2, TenBrook et al. 2009). Three studies describing effects of fipronil on mesocosm, microcosm and model ecosystems were identified and rated for reliability according to the UCDM (Table 3.9, TenBrook et al. 2009). One study were rated as reliable (R; Wirth et al. 2004), one study was rated as less reliable (L; Walse et al. 2004) and is used as supporting data. One study rated as not reliable (N) and is not discussed in this report (Aajoud et al. 2003).

Writh et al. (2004) studied three concentrations of fipronil in saltwater estuary mesocosms with an added macrofauna community of fish, clams, oysters, and shrimp. Only the grass shrimp (*Palaemonetes pugio*) experienced fipronil toxicity with a reported LC<sub>50</sub> of 0.357 µg/L. This value is more than 25 times the chronic WQC for fipronil. Chronic toxicity values for individual species or the community were not reported.

Walse et al. (2004) exposed artificial estuary mesocosms to a single concentration of fipronil to study the formation of degradates in the aqueous and sediment phases. Flora and fauna were not identified and toxicity values were not reported.

### 10.3 Threatened and endangered species

The derived criteria are compared to measured toxicity values for threatened and endangered species (TES), as well as to predicted toxicity values for TES, to ensure that they will be protective of these species. Current lists of state and federally listed threatened and endangered plant and animal species in California were obtained from the California Department of Fish and Wildlife (CDFW) website (<https://nrm.dfg.ca.gov/FileHandler.ashx?DocumentID=109405&inline>; CDFW 2016).

There are listed species that are represented in the acute toxicity data set by members of the same family or genus. *Oncorhynchus mykiss* can serve as a surrogate in estimates for other species in the same family using the USEPA interspecies correlation estimation website (Web-ICE v. 3.2.1; Raimondo et al. 2013). Table 23 summarizes the results of the ICE analyses for all fipronil compounds. One listed animal species is represented in the each of the WQC data sets

for fipronil, fipronil-sulfone, and fipronil-desulfinyl. Five Evolutionarily Significant Units of *Oncorhynchus mykiss* are listed as federally threatened or endangered throughout California.

### Fipronil

The acute WQC data set for fipronil includes a LC<sub>50</sub> for *O. mykiss* of 248 µg/L calculated from a study rated RR (Ward 1991b). The chronic WQC data set includes a 90-d MATC for *O. mykiss* of 20 µg/L from a study rated RR (Machado 1992a). The estimated acute toxicity values for fipronil range from 205 µg/L for Apache trout to 583 µg/L for Sockeye salmon. The estimated chronic toxicity values for fipronil range from 12 µg/L for Apache trout up to 31 µg/L for Chinook salmon. Based on the available data and estimated values for TES, there is no evidence that the calculated acute and chronic WQC will be underprotective of threatened and endangered species.

### Fipronil-sulfone

The acute WQC data set for fipronil-sulfone includes a LC<sub>50</sub> for *O. mykiss* of 39 µg/L reported in a RR rated study by Bettencourt 1992b. The supplemental acute WQC data set contains additional LC<sub>50</sub> values of different durations from the same study. The estimated acute toxicity values for fipronil-sulfone range from 26 µg/L for Apache trout up to 153 µg/L for Sockeye salmon. Based on the available data and estimated values for TES, there is no evidence that the calculated acute and chronic WQC will be underprotective of threatened and endangered species.

### Fipronil-desulfinyl

The acute WQC data set for fipronil-desulfinyl includes a LC<sub>50</sub> for *O. mykiss* of 31 µg/L calculated from a study rated RR (Collins 1993b). The supplemental acute WQC data set contains additional LC<sub>50</sub> values of different durations from the same study. The estimated acute toxicity values for fipronil-desulfinyl range from 20 µg/L for Apache trout up to 129 µg/L for Sockeye salmon. Based on the available data and estimated values for TES, there is no evidence that the calculated acute and chronic WQC will be underprotective of threatened and endangered species.

No listed threatened or endangered species are included in the acceptable and supplemental data sets used for fipronil compounds BSQC derivation (Tables 15-22). No data were found for effects of sediment-associated fipronil compounds on federally endangered crustaceans and insects, or acceptable surrogates (i.e., in the same family). Acute and chronic BSQC were not calculable for fipronil or its degradates from the available studies. If highly rated data becomes available in the future so that criteria can be calculated, the resulting BSQC should be converted to interstitial concentrations (µg/L), to compare to the aqueous toxicity values for TES.

## 11 Harmonization with other environmental media

### 11.1 Bioaccumulation

Bioaccumulation was assessed to ensure that the derived criteria will not lead to unacceptable levels of fipronil in food items (section 3-7.1, TenBrook et al. 2009). Fipronil has a log  $K_{ow}$  of 4.45 and a molecular weight of 437.15 (section 3), which indicates it has bioaccumulative potential (section 3-7.1, TenBrook et al. 2009). No biomagnification factor (BMF) values were found in the literature for fipronil, but bioconcentration of fipronil has been measured in some studies (Table 1).

To check that these criteria are protective of terrestrial wildlife that may consume aquatic organisms, a bioaccumulation factor (BAF) was used to estimate the water concentration that would roughly equate to a reported toxicity value for consumption of fish by terrestrial wildlife. These calculations are further explained in section 3-7.1 of the methodology (TenBrook et al. 2009). The BAF of a given chemical is the product of the bioconcentration factor (BCF) and a BMF, such that  $BAF = BCF * BMF$ . For a conservative estimate, the highest fish BCF of 321 L/kg for an unknown species (Table 1) and a default BMF of 1, chosen based on the log  $K_{ow}$  of fipronil (Table 3.15, TenBrook et al. 2009), were used to calculate a BAF.

A chronic dietary NOEC for an oral predator is preferred for this calculation because it is the most realistic value for extrapolation to bioaccumulation in the environment (section 3-7.1, TenBrook et al. 2009). The dietary NOEC for mallard duck to fipronil of 1250 mg/kg was used (Pedersen 1993b).

$$NOEC_{water} = \frac{NOEC_{oral\_predator}}{BCF_{food\_item} * BMF_{food\_item}}$$

Mallard:

$$NOEC_{water} = \frac{1250 \text{ mg/kg}}{321 \text{ L/kg} * 10} = 3.89 \text{ mg/L} = 3890 \text{ } \mu\text{g/L}$$

In this example, the chronic WQC of 2.2 ng/L is a approximately six orders of magnitude below the estimated  $NOEC_{water}$  for mallard, and is not likely to cause adverse effects to terrestrial wildlife. Bioaccumulation of fipronil is not likely because the  $NOEC_{water}$  exceeds the aqueous solubility of fipronil (1,650  $\mu\text{g/L}$ , see section 3). This analysis indicates that terrestrial wildlife will not likely be harmed by bioaccumulation of fipronil if the WQC is attained.

## 11.2 Air, Sediment, Water, etc.

This section addresses how the maximum allowable concentration of fipronil might impact life in other environmental compartments through partitioning (section 3-7.2, TenBrook et al. 2009). However, there are no federal or state sediment or air quality standards for fipronil (CARB 2005, CDWR 1995, USEPA 2015c, NOAA 1999) to enable this kind of extrapolation. For biota, the limited data on bioconcentration or biomagnification of fipronil were addressed in the bioaccumulation section (11.1).

Sediment toxicity should be assessed to determine the potential for desorption from sediment if equilibrium conditions were present. The interim chronic BSQC are converted from OC-normalized sediment concentrations to interstitial water concentrations and compared to the derived water quality criteria based on the  $K_{OC}$  of fipronil as the relevant partition coefficient as follows.

$$C_{\text{interstitial water}} = C_{\text{OC-normal sediment}} \div K_{OC}$$

$$C_{\text{interstitial water}} = \text{Chronic BSQC} \div K_{OC}$$

Fipronil: 
$$C_{\text{interstitial water}} = 0.0007 \mu\text{g/g OC} \div 5,321 \text{ L/kg OC} * 1000 \text{ g OC/kg OC}$$
$$= 0.00013 \mu\text{g/L}$$

Fipronil-sulfide: 
$$C_{\text{interstitial water}} = 0.0004 \mu\text{g/g OC} \div 40,904 \text{ L/kg OC} * 1000 \text{ g OC/kg OC}$$
$$= 0.0000098 \mu\text{g/L}$$

Fipronil-sulfone: 
$$C_{\text{interstitial water}} = 0.0003 \mu\text{g/g OC} \div 153,623 \text{ L/kg OC} * 1000 \text{ g OC/kg OC}$$
$$= 0.0000020 \mu\text{g/L}$$

Fipronil-desulfinyl: 
$$C_{\text{interstitial water}} = 0.00020 \mu\text{g/g OC} \div 1,310 \text{ L/kg OC} * 1000 \text{ g OC/kg OC}$$
$$= 0.00015 \mu\text{g/L}$$

The expected interstitial water concentrations for fipronil, fipronil-sulfide and fipronil-sulfone are all below the chronic water quality criteria calculated for these compounds (0.0022  $\mu\text{g/L}$ , 0.00013  $\mu\text{g/L}$ , and 0.00017  $\mu\text{g/L}$ , respectively). There is no chronic water quality criterion for fipronil-desulfinyl to compare to, but the expected interstitial water concentration is well-below the lowest toxicity value for this degradate of a MATC of 0.1500  $\mu\text{g/L}$  for the saltwater mysid *Americamysis bahia*.

## 12 Fipronil Criteria Summary

### 12.1 Assumptions, limitations, uncertainties

The assumptions, limitations and uncertainties involved in criteria derivation should be available to inform environmental managers of the accuracy and confidence in the derived criteria. This section summarizes any data limitations that affected the procedure used to determine the final fipronil criteria.

#### Aquatic data sets

Fipronil readily degrades in the environment into a host of stable degradates (see section 2). Toxicity to aquatic species has been shown by fipronil and many of its degradates. For most of the degradates, however, there is not enough highly rated data available to calculate final WQC. Although fipronil is an insecticide, there were no highly rated chronic aquatic insect studies available. There were also no plant studies available for fipronil, however plants are not expected to be particularly sensitive to fipronil and its degradates.

There were enough highly rated acute fipronil and fipronil-sulfone data to use a SSD to calculate each acute WQC (using Burr III and Log-logistic, respectively). One limitation in these data sets is that not all of the data are from flow-through tests to calculate the toxicity values. Flow-through tests are preferred in order to reduce inaccuracies due to sorption in the test vessels. The majority of the acute RR data for both fipronil and fipronil-sulfone are from static tests. Uncertainty of the acute WQC can be quantified by looking at the lower 95% confidence limits (sections 7.1 and 7.3). A second limitation is that the acute data set for fipronil-sulfone had a significant lack of fit for the Burr III SSD. This SSD is preferred for data sets containing >8 values. It is possible that additional toxicity values could improve the fit of the Burr III SSD.

The chronic data set for fipronil did not contain enough values to fit a SSD and only had appropriate paired data to calculate an empirical SMACR for one species, thus two default ACRs were included in the final ACR for fipronil. Additional highly rated chronic values for fipronil are needed in order to compute a chronic WQC using only empirical ACRs or to fit an SSD.

The limitation with fipronil-sulfide, fipronil-desulfinyl, fipronil-carboxamide, and fipronil-destrifluoromethyl-sulfonate was a lack of both acute and chronic water quality data. The acute data set for fipronil-sulfide contained only two of the five necessary taxa in order to fit a SSD, necessitating the use of an AF to calculate the acute WQC. Highly rated acute data from a fish of the family Salmonidae, a warm water fish, and a planktonic crustacean of a relevant genus (*Ceriodaphnia*, *Daphnia*, or *Simocephalus*) is needed for fipronil-sulfide to be able to fit a SSD. Similarly, the acute data set for fipronil-desulfinyl was lacking sufficient data to use a SSD or an AF to calculate an acute criterion. Insect, benthic crustacean, and planktonic crustacean data is needed for to fit a SSD to the fipronil-desulfinyl data set.

There was only one acute value available for fipronil-carboxamide and it did not meet the requirements for the AF procedure. No chronic values were available. The chronic water data set for fipronil-sulfone also contained only a single value. Additional highly rated values are needed in order to calculate WQC.

#### Sediment data sets

All fipronil compounds were lacking in both acute and chronic sediment data. For fipronil and the three degradates fipronil-sulfide, fipronil-sulfone, and fipronil-desulfinyl there were acute data available for two species. Only one chronic sediment toxicity value was located for any of the compounds, making chronic data the most significant limitation for calculating BSQC.

#### Bioavailability

There were no studies available that assessed the bioavailability of any fipronil compounds. Data is needed in this area in order to make a full assessment of the potential risk to the aquatic environment.

#### Mixtures

End users of agricultural and household pesticides could use multiple products on any given site. There is the potential use of insecticides in combination with herbicides or other targeted pesticides. Therefore it is important to have a range of studies available that study the mixture effects of fipronil with other compounds. Few mixture studies were available that focused on aquatic organisms and no studies were available that focused on benthic species.

#### Ecosystem studies

The few ecosystem studies that were available did not meet the requirements of the UCDM or UCDSM. The two acceptable studies (rated R or L) did not test the effects of fipronil degradates directly and were in saltwater. Neither study reported community-level toxicity values. Freshwater ecosystem studies are needed in order to determine adequate protection of the Central Valley watershed that the UCDM and UCDSM are designed to protect.

#### Wildlife data sets

Both acute and chronic wildlife data sets were lacking sufficient data for all fipronil degradates. The fipronil data set contained only approximated acute values. Only one study was available for a degradate, fipronil-desulfinyl, and it did not include a chronic value, thus preventing an estimation of its bioaccumulative potential. Given that all the degradates have been shown to form abiotically in the environment through photolysis, hydrolysis, oxidation, or reduction, it is important that aquatic wildlife toxicity studies are performed to assess the potential risk to species such as mallard duck.

## Enantiomers

High grade and commercial formulations of the insecticide fipronil are generally available as a 50:50 racemic mixture of the (+) and (-) enantiomers. As shown in the fipronil data set, the (+) and (-) enantiomers result in unique toxicities to the species tested. The (+) enantiomer is significantly more toxic both to crustaceans such as *D. magna* as well as to fish such as *P. promelas*. There was not enough data in the fipronil dataset to calculate enantiomer criteria, but it may become more important to do so in the future if enriched products become predominant in the market.

### *12.2 Comparison to EPA method and other criteria*

This section provides a comparison between UCDM WQC and the USEPA 1985 guidelines for WQC derivation (USEPA 1985). The fipronil data sets generated in this report was examined for use with the USEPA 1985 guidelines.

The USEPA acute method has three additional taxa requirements beyond the five required by the UCDM, they are:

1. A third family in the phylum Chordata (e.g., fish, amphibian);
2. A family in a phylum other than Arthropoda or Chordata (e.g., Rotifera, Annelida, Mollusca);
3. A family in any order of insect or any phylum not already represented.

## Fipronil

Two out of three of these additional requirements are met for fipronil as follows:

1. A third family in the phylum Chordata is met with data from channel catfish (*Ictalurus punctatus*).
2. This requirement is not met because all data are from organisms in the phylum Arthropoda or Chordata.
3. A family in any order of insect or any phylum not already represented is met with data from *Isoperla quinquepunctata* in the Perlodidae family.

The USEPA 1985 guidelines cannot be used to calculate an acute criterion for fipronil because one of the eight taxa requirements is not met. The California Department of Fish and Wildlife (formerly Fish and Game) have used data sets that met only seven of eight requirements in the USEPA methodology when the missing taxon was known to be insensitive. The missing taxa for fipronil are not known to be insensitive to fipronil, thus an acute WQC will not be calculated with the USEPA 1985 guidelines. The chronic data set is also deficient, only meeting two of the eight taxa requirements of the USEPA 1985 guidelines (*O. mykiss* and *C. dubia*).

To date, no USEPA sediment criteria or benchmarks are available for fipronil. The USEPA proposes an EqP-based approach, through which the chronic WQC is used to predict the corresponding sediment concentration using the  $K_{OC}$  (Di Toro et al. 2002). The lowest SMAV in the acceptable sediment data set was converted to an interstitial water concentration to compare it to existing WQC. The lowest SMAV in the RR data set of 0.10  $\mu\text{g/g OC}$  for *C. dilutus* was converted to an interstitial concentration of 0.13 ng/L using the geometric mean of  $K_{OC}$ s of 5,321. This value is compared to the chronic WQC for fipronil of 2.2 ng/L, which is approximately a factor of 17 lower than the lowest SMAV. Thus, the chronic WQC would likely be protective of short-term effects from sediment-associated fipronil. However, no chronic fipronil sediment effects data are available, so it is unclear as to whether the chronic WQC would also be protective of long-term sublethal effects in sediment.

$$C_{\text{interstitial water}} = C_{\text{OC-normal sediment}} \div K_{OC}$$

$$C_{\text{interstitial water}} = \text{Lowest sediment SMAV} \div K_{OC}$$

Fipronil:

$$C_{\text{interstitial water}} = 0.10 \mu\text{g/g OC} \div 5,321 \text{ L/kg OC} * 1000 \text{ g OC/kg OC}$$

$$= 0.00013 \mu\text{g/L}$$

$$= 0.13 \text{ ng/L}$$

### Fipronil-sulfide

One out of three of these additional requirements are met for fipronil-sulfide as follows:

1. This requirement is not met because there is no data from the phylum Chordata.
2. This requirement is not met because all data are from organisms in the phylum Arthropoda.
3. A family in any order of insect or any phylum not already represented is met with data from *C. dilutus* in the Chironomidae family.

The USEPA 1985 guidelines cannot be used to calculate an acute criterion for fipronil-sulfide because two of the eight taxa requirements are not met. The California Department of Fish and Wildlife (formerly Fish and Game) have used data sets that met only seven of eight requirements in the USEPA methodology when the missing taxon was known to be insensitive. The missing taxa for fipronil-sulfide are not known to be insensitive to fipronil-sulfide, thus an acute WQC will not be calculated with the USEPA 1985 guidelines. The chronic data set is also deficient, only meeting one of the eight taxa requirements of the USEPA 1985 guidelines (*D. magna*).

To date, no USEPA sediment criteria or benchmarks are available for fipronil-sulfide. The USEPA proposes an EqP-based approach, through which, the chronic WQC is used to predict the corresponding sediment concentration using the  $K_{OC}$  (Di Toro et al. 2002). The lowest

SMCV in the acceptable sediment data set was converted to an interstitial water concentration to compare it to existing WQC. The lowest SMCV in the RR data set of 0.16 µg/g OC for *C. riparius* was converted to an interstitial concentration of 0.0039 µg/L using the geometric mean of  $K_{OCs}$  of 40,904. This value is compared to the chronic WQC for fipronil-sulfide of 0.00013 µg/L, which is a factor of 30 lower than the lowest SMCV. Thus, the chronic WQC would likely be protective of long-term sublethal effects from sediment-associated fipronil-sulfide.

$$C_{\text{interstitial water}} = C_{\text{OC-normal sediment}} \div K_{OC}$$

$$C_{\text{interstitial water}} = \text{Lowest sediment SMCV} \div K_{OC}$$

Fipronil-sulfide:

$$C_{\text{interstitial water}} = 0.16 \mu\text{g/g OC} \div 40,904 \text{ L/kg OC} * 1000 \text{ g OC/kg OC}$$

$$= 0.0039 \mu\text{g/L}$$

$$= 3.9 \text{ ng/L}$$

### Fipronil-sulfone

Two out of three of these additional requirements are met for fipronil-sulfone as follows:

1. A third family in the phylum Chordata is met with data from channel rainbow trout (*O. mykiss*).
2. This requirement is not met because all data are from organisms in the phylum Arthropoda or Chordata.
3. A family in any order of insect or any phylum not already represented is met with data from *C. dilutus* in the Chironomidae family.

The USEPA 1985 guidelines cannot be used to calculate an acute criterion for fipronil-sulfone because one of the eight taxa requirements is not met. The California Department of Fish and Wildlife (formerly Fish and Game) have used data sets that met only seven of eight requirements in the USEPA methodology when the missing taxon was known to be insensitive. The missing taxa for fipronil-sulfone are not known to be insensitive to fipronil-sulfone, thus an acute WQC will not be calculated with the USEPA 1985 guidelines. The chronic data set is also deficient, only meeting one of the eight taxa requirements of the USEPA 1985 guidelines (*D. magna*).

To date, no USEPA sediment criteria or benchmarks are available for fipronil-sulfone. The USEPA proposes an EqP-based approach, through which, the chronic WQC is used to predict the corresponding sediment concentration using the  $K_{OC}$  (Di Toro et al. 2002). The lowest SMAV in the acceptable sediment data set was converted to an interstitial water concentration to compare it to existing WQC. The lowest SMAV in the RR data set of 0.04 µg/g OC for *C. dilutus* was converted to an interstitial concentration of x ng/L using the geometric mean of  $K_{OCs}$  of 153,623. This value is compared to the chronic WQC for fipronil of 0.17 ng/L, which is lower

than the lowest SMAV. Thus, the chronic WQC would likely be protective of short-term effects from sediment-associated fipronil-sulfone. However, no chronic fipronil-sulfone sediment effects data are available, so it is unclear as to whether the chronic WQC would also be protective of long-term sublethal effects in sediment.

$$C_{\text{interstitial water}} = C_{\text{OC-normal sediment}} \div K_{\text{OC}}$$

$$C_{\text{interstitial water}} = \text{Lowest sediment SMAV} \div K_{\text{OC}}$$

Fipronil-sulfone:

$$C_{\text{interstitial water}} = 0.04 \mu\text{g/g OC} \div 153,623 \text{ L/kg OC} * 1000 \text{ g OC/kg OC}$$

$$= 0.00026 \mu\text{g/L}$$

$$= 0.26 \text{ ng/L}$$

### Fipronil-desulfinyl

None out of three of these additional requirements are met for fipronil-desulfinyl. Therefore the USEPA 1985 guidelines cannot be used to calculate an acute criterion for fipronil-desulfinyl. The chronic data set is also deficient as it does not meet any of the eight taxa requirements of the USEPA 1985 guidelines.

To date, no USEPA sediment criteria or benchmarks are available for fipronil-desulfinyl. The USEPA proposes an EqP-based approach, through which, the chronic WQC is used to predict the corresponding sediment concentration using the  $K_{\text{OC}}$  (Di Toro et al. 2002). The lowest SMAV in the acceptable sediment data set was converted to an interstitial water concentration to compare it to existing WQC. The lowest SMAV in the RR data set of 28  $\mu\text{g/g OC}$  for *C. dilutus* was converted to an interstitial concentration of 21  $\mu\text{g/L}$  using the geometric mean of  $K_{\text{OCs}}$  of 1,310. This value would be compared to the chronic WQC for fipronil-desulfinyl; however, a chronic WQC could not be calculated. Thus, it is the protection of short-term effects from sediment-associated fipronil-desulfinyl is unclear. No chronic fipronil-desulfinyl sediment effects data are available, so it is unclear as to whether the chronic WQC would also be protective of long-term sublethal effects in sediment.

$$C_{\text{interstitial water}} = C_{\text{OC-normal sediment}} \div K_{\text{OC}}$$

$$C_{\text{interstitial water}} = \text{Lowest sediment SMAV} \div K_{\text{OC}}$$

Fipronil-desulfinyl:

$$C_{\text{interstitial water}} = 28 \mu\text{g/g OC} \div 1,310 \text{ L/kg OC} * 1000 \text{ g OC/kg OC}$$

$$= 21 \mu\text{g/L}$$

### *12.3 Final criteria statements*

Although the criteria were derived to be protective of aquatic life in the Sacramento and San Joaquin Rivers, these criteria would be appropriate for any freshwater ecosystem in North America, unless species more sensitive than are represented by the species examined in the development of the present criteria are likely to occur in the ecosystems of interest.

The final water quality criteria statements are:

#### Fipronil

Aquatic life should not be affected unacceptably if the four-day average concentration of fipronil does not exceed 0.0022 µg/L (2.2 ng/L) in the water column more than once every three years on average and if the one-hour average concentration does not exceed 0.014 µg/L (14 ng/L) more than once every three years on average.

The final acute WQC was derived using the Burr III SSD procedure (section 7.1.1) and the acute data used in criteria calculation are shown in Table 3. The chronic criterion was derived by use of an ACR calculated using a combination of empirical and default ACRs (section 7.2.1); chronic data rated RR are shown in Table 9.

The interim acute BSQC of 0.0042 µg/g OC (4.2 ng/g OC) was derived with an assessment factor (section 8.1.1) and acute data used in criteria calculation are shown in Table 15. The interim chronic BSQC of 0.0007 µg/g OC (0.7 ng/g OC) was derived with a default ACR (section 8.2.1).

#### Fipronil-sulfide

Aquatic life should not be affected unacceptably if the four-day average concentration of fipronil-sulfide does not exceed 0.00010 µg/L (0.10 ng/L) in the water column more than once every three years on average and if the one-hour average concentration does not exceed 0.00058 µg/L (0.58 ng/L) more than once every three years on average.

The final acute WQC was derived using the AF procedure (section 7.1.2) and the acute data used in criteria calculation are shown in Table 4. The chronic criterion was derived by use of an ACR calculated using a default ACR (section 7.2.2); chronic data rated RR are shown in Table 10.

The interim acute BSQC of 0.003 µg/g OC (3 ng/g OC) was derived with an assessment factor (section 8.1.2) and acute data used in criteria calculation are shown in Table 16. The interim chronic BSQC of 0.0004 µg/g OC (0.4 ng/g OC) was derived with a default ACR (section 8.2.2); chronic data rated RR are shown in Table 20.

### Fipronil-sulfone

Aquatic life should not be affected unacceptably if the four-day average concentration of fipronil-sulfone does not exceed 0.00024 µg/L (0.24 ng/L) in the water column more than once every three years on average and if the one-hour average concentration does not exceed 0.0013 µg/L (1.3 ng/L) more than once every three years on average.

The final acute WQC was derived using the Log-logistic procedure (section 7.1.3) and the acute data used in criteria calculation are shown in Table 5. The chronic criterion was derived by use of an ACR calculated using a default ACR (section 7.2.3); chronic data rated RR are shown in Table 11.

The interim acute BSQC of 0.002 µg/g OC (2 ng/g OC) was derived with an assessment factor (section 8.1.3) and acute data used in criteria calculation are shown in Table 17. The interim chronic BSQC of 0.0003 µg/g OC (0.3 ng/g OC) was derived with a default ACR (section 8.2.3).

### Fipronil-desulfinyl

Water quality criteria could not be calculated for fipronil-desulfinyl. The interim acute BSQC of 0.0012 µg/g OC (1.2 ng/g OC) was derived with an assessment factor (section 8.1.4) and acute data used in criteria calculation are shown in Table 18. The interim chronic BSQC of 0.00020 µg/g OC (0.20 ng/g OC) was derived with a default ACR (section 8.2.4).

Fipronil and degradates have been monitored in urban environments in California and a summary of this data is given to provide context for the use of water quality criteria for these compounds. Weston and Lydy (2013) report that of 24 samples in urban waterbodies during rain events, the detection frequencies of fipronil, fipronil-sulfide, fipronil-sulfone, and fipronil-desulfinyl were 88%, 42%, 88%, and 83%, respectively. A recent review of fipronil monitoring data from urban watersheds in California (Ruby 2013) includes data for fipronil and degradates in both water and sediment matrices. Fipronil was detected in 39% of 871 water samples. The degradates data was compiled, rather than separated by degradate, and at least one degradate was detected in 24% of 2,271 water samples. There were far fewer sediment samples available. Fipronil was detected in 19% of 16 sediment samples and at least one degradate was detected in 35% 48 sediment samples.

## References

- Ali A, Nayar JK and Gu WD. (1998) Toxicity of a phenyl pyrazole insecticide, fipronil, to mosquito and chironomid midge larvae in the laboratory. *Journal of the American Mosquito Control Association*, 14(2), 216-218.
- Aajoud A, Ravanel P and Tissut M. (2003) Fipronil metabolism and dissipation in a simplified aquatic ecosystem. *Journal of agricultural and food chemistry*, 51(5), 1347-1352.
- Ayliffe JM. (1998) [<sup>14</sup>C]-Fipronil degradation and retention in two water/sediment systems. Rhone-Poulenc Agriculture Limited, Essex, England. Laboratory project ID 13333. USEPA MRID 44661301.
- Baird S, Garrison A, Jones J, Avants J, Bringolf R and Black M. (2013) Enantioselective toxicity and bioaccumulation of fipronil in fathead minnows (*Pimephales promelas*) following water and sediment exposures. *Environmental Toxicology and Chemistry*, 32(1), 222-227.
- Beggel S, Werner I, Connon RE and Geist JP. (2010) Sublethal toxicity of commercial insecticide formulations and their active ingredients to larval fathead minnow (*Pimephales promelas*). *Science of the total environment*, 408(16), 3169-3175.
- Bettencourt MJ. (1992a) (M&B 46136)-Acute toxicity to bluegill sunfish (*Lepomis macrochirus*) under flow-through conditions. Springborn Laboratories, Inc., Wareham, Massachusetts. Laboratory study number 10566.0.391.6207.105. Submitted to Rhone-Poulenc Ag Company, Research Triangle, North Carolina. USEPA MRID 42918674. CA DPR 157302.
- Bettencourt MJ. (1992b) (M&B 46136)-Acute toxicity to rainbow trout (*Oncorhynchus mykiss*) under flow-through conditions. Springborn Laboratories, Inc., Wareham, Massachusetts. Laboratory study number 10566.0.391.6208.108. Submitted to Rhone-Poulenc Ag Company, Research Triangle, North Carolina. USEPA MRID 42918673. CA DPR 157303.
- Bobé A, Coste CM, and Cooper J. (1997) Factors influencing the adsorption of fipronil on soils. *Journal of Agricultural and Food Chemistry* 45.12, 4861-4865.
- Bobé A, Meallier P, Cooper JF and Coste CM. (1998) Kinetics and mechanisms of abiotic degradation of fipronil (hydrolysis and photolysis). *Journal of Agricultural and Food Chemistry*, 46(7), 2834-2839.
- Brennan AA, Harwood AD, You J, Landrum PF and Lydy MJ. (2009) Degradation of fipronil in anaerobic sediments and the effect on porewater concentrations. *Chemosphere*, 77(1), 22-28.

- Bringolf RB, Cope WG, Eads CB, Lazaro PR, Barnhart MC and Shea D. (2007) Acute and chronic toxicity of technical-grade pesticides to glochidia and juveniles of freshwater mussels (unionidae). *Environmental Toxicology and Chemistry*, 26(10), 2086-2093.
- Burr CM. (1997) [C<sup>14</sup>]-M&B 45950: Adsorption/desorption to and from four soils and one sediment. Rhone-Poulenc Agricultural Limited, Essex, England. Laboratory project ID 13510. Submitted to Rhone-Poulenc Agriculture. USEPA MRID 44537902.
- Cafarella MA. (2005) Fipronil: Life-cycle toxicity test with mysids (*Americamysis bahia*) under static conditions in a water-sediment system. Springborn Smithers Laboratories, Wareham, Massachusetts. Laboratory project ID 986.6163. Submitted to BASF Corporation, Research Triangle Park, North Carolina. US EPA MRID 46619103.
- CARB. 2005. California Ambient Air Quality Standards. [www.arb.ca.gov/research/aaqs/caaqs/caaqs.htm](http://www.arb.ca.gov/research/aaqs/caaqs/caaqs.htm). California Air Resources Board, Sacramento, CA.
- Cary TL, Chandler GT, Volz DC, Walse SS and Ferry JL. (2004) Phenylpyrazole insecticide fipronil induces male infertility in the estuarine meiobenthic crustacean *Amphiascus tenuiremis*. *Environmental science & technology*, 38(2), 522-528.
- CDFW (2013) State and federally listed endangered and threatened animals of California. California Natural Diversity Database. California Department of Fish and Wildlife, Sacramento, CA. Available from: <http://www.dfg.ca.gov/biogeodata/cnddb/pdfs/TEAnimals.pdf>
- CDWR (1995) Compilation of Sediment and Soil Standards, Criteria, and Guidelines. California Department of Water Resources, State of California, The Resources Agency, Sacramento, CA. URL <[http://www.water.ca.gov/pubs/waterquality/municipal\\_wq\\_investigations/mwqi\\_technical\\_documents/compilation\\_of\\_soil\\_and\\_sediment\\_standards\\_criteria\\_and\\_guidelines/compilation\\_of\\_soil\\_and\\_sediment\\_standards\\_criteria\\_and\\_guidelines.\\_february\\_1995.pdf](http://www.water.ca.gov/pubs/waterquality/municipal_wq_investigations/mwqi_technical_documents/compilation_of_soil_and_sediment_standards_criteria_and_guidelines/compilation_of_soil_and_sediment_standards_criteria_and_guidelines._february_1995.pdf)>
- Chandler GT, Cary TL, Volz DC, Walse SS, Ferry JL and Klosterhaus SL. (2004) Fipronil effects on estuarine copepod (*Amphiascus tenuiremis*) development, fertility, and reproduction: A rapid life-cycle assay in 96-well microplate format. *Environmental Toxicology and Chemistry*, 23(1), 117-124.
- Chaton PF, Ravanel P, Meyran JC and Tissut M. (2001) The toxicological effects and bioaccumulation of fipronil in larvae of the mosquito *Aedes aegypti* in aqueous medium. *Pesticide Biochemistry and Physiology*, 69(3), 183-188.

- Chaton PF, Ravanel P, Tissut M and Meyran JC. (2002) Toxicity and bioaccumulation of fipronil in the nontarget arthropodan fauna associated with subalpine mosquito breeding sites. *Ecotoxicology and Environmental Safety*, 52(1), 8-12.
- Collins MK. (1993a) MB46513-Acute toxicity to bluegill sunfish (*Lepomis macrochirus*) under static renewal conditions. Springborn Laboratories, Inc., Wareham, Massachusetts. Laboratory study number 10566.0492.6242.100. Submitted to Rhone-Poulenc Ag Company, Research Triangle, North Carolina. USEPA MRID 43279702.
- Collins MK. (1993b) MB46513-Acute toxicity to rainbow trout (*Oncorhynchus mykiss*) under static renewal conditions. Springborn Laboratories, Inc., Wareham, Massachusetts. Laboratory study number 10566.0492.6241.103. Submitted to Rhone-Poulenc Ag Company, Research Triangle, North Carolina. USEPA MRID 43279703. CA DPR 157299.
- Collins MK. (1993b) RPA 104615-Acute toxicity to rainbow trout (*Oncorhynchus mykiss*) under static renewal conditions. Springborn Laboratories, Inc., Wareham, Massachusetts. Laboratory study number 10566.0792.6246.103. Submitted to Rhone-Poulenc Ag Company, Research Triangle, North Carolina. USEPA MRID 43291718.
- Collins MK. (1993) RPA 104615-Acute toxicity to daphnids (*Daphnia magna*) under static. Springborn Laboratories, Inc., Wareham, Massachusetts. Laboratory study number 10566.0792.6245.110. Submitted to Rhone-Poulenc Ag Company, Research Triangle, North Carolina. USEPA MRID 43291719.
- Corgier MMC and Plewa AP. (1992) <sup>14</sup>C-MB 46030 Aqueous Photolysis. Rhone-Poulenc Secteur Agro, Lyon, France. Laboratory study number 91-55. USEPA MRID 42918661.
- CRWQCB-CVR (2011) The Water Quality Control Plan (Basin Plan) for the California Regional Water Quality Control Board Central Valley Region, fourth edition, the Sacramento River Basin and the San Joaquin River Basin. [Accessed September 21, 2012]. Available from: [http://www.waterboards.ca.gov/rwqcb5/water\\_issues/basin\\_plans/sacsjr.pdf](http://www.waterboards.ca.gov/rwqcb5/water_issues/basin_plans/sacsjr.pdf)
- CVRWQCB (2006) Sacramento and San Joaquin River Watersheds Pesticide Basin Plan Amendment Fact Sheet. Central Valley Regional Water Quality Control Board, Rancho Cordova, CA. [http://www.swrcb.ca.gov/rwqcb5/water\\_issues/tmdl/central\\_valley\\_projects/central\\_valley\\_pesticides/att2\\_fact](http://www.swrcb.ca.gov/rwqcb5/water_issues/tmdl/central_valley_projects/central_valley_pesticides/att2_fact).
- Dionne E. (1993) MB 46030-Acute toxicity to the eastern oyster (*Crassostrea virginica*) under flow-through conditions. Springborn Laboratories, Inc., Wareham, Massachusetts. Laboratory study number 10566.0393.6269.504. Submitted to Rhone-Poulenc Ag Company, Research Triangle, North Carolina. USEPA MRID 43291701. CA DPR 157285.

- Dionne E. (1997) Fipronil technical-acute toxicity to channel catfish (*Ictalurus punctatus*) under flow-through conditions. Springborn Laboratories, Inc., Wareham, Massachusetts. Laboratory study number 10566.1096.6408.107. Submitted to Rhone-Poulenc Ag Company, Research Triangle, North Carolina. USEPA MRID 44299401. CA DPR 157281.
- Dionne E. (2000) Fipronil technical-Chronic toxicity to the sheepshead minnow (*Cyprinodon variegatus*) during a full life-cycle exposure. Springborn Laboratories, Inc., Wareham, Massachusetts. Laboratory study number 10566.6580. Submitted to Aventis CropScience, Research Triangle, North Carolina. USEPA MRID 45265101.
- Di Toro DM, Hansen DJ, DeRosa LD, Berry WJ, Bell HE, Reiley MC, Zarba CS. (2002) Technical basis for the derivation of equilibrium partitioning sediment quality guidelines (ESGs) for the protection of benthic organisms: Nonionic organics. Draft report. 822-R-02-041. USEPA. Office of Science and Technology and Office of Research and Development, Washington, DC
- Donovan S, Pescatore M J. (2002) Method for measuring the logarithm of the octanol–water partition coefficient by using short octadecyl–poly(vinyl alcohol) high-performance liquid chromatography columns. *Journal of Chromatography A*, 952, 47-61.
- Doran G, Eberbach P and Helliwell S. (2009) Sorption and degradation of fipronil in flooded anaerobic rice soils. *Journal of agricultural and food chemistry*, 57(21), 10296-10301.
- Feung CS and Mislankar SG. (1996) Fipronil metabolite MB 46513: Soil adsorption/desorption. Rhone-Poulenc Ag Company, Research Triangle Park, North Carolina. Laboratory study number EC-96-333. USEPA MRID 44262831.
- Feung CS and Yenne SP. (1997) Fipronil: Aerobic aquatic metabolism. Rhone-Poulenc Ag Company, Research Triangle Park, North Carolina. Laboratory study number EC-95-315. USEPA MRID 44261909.
- Fojut TL, Vasquez ME, Tjeerdema RS (2011) Methodology for derivation of pesticide sediment quality criteria for the protection of aquatic life. Phase I: Review of existing methodologies. Report prepared by the University of California Davis for the Central Valley Regional Water Quality Control Board. Available at: [http://www.waterboards.ca.gov/rwqcb5/water\\_issues/tmdl/central\\_valley\\_projects/central\\_valley\\_pesticides/sediment\\_quality\\_criteria\\_method\\_development/ucd\\_sed\\_phase1final.pdf](http://www.waterboards.ca.gov/rwqcb5/water_issues/tmdl/central_valley_projects/central_valley_pesticides/sediment_quality_criteria_method_development/ucd_sed_phase1final.pdf)
- Fojut TL, Vasquez ME, Poulsen AH, Tjeerdema RS (2013) Methods for deriving pesticide aquatic life criteria for sediments. *Rev Environ Contamin Toxicol* 224:97-175

- Fojut TL, Vasquez M, Trunnelle KJ, Tjeerdema RS (2014) Methodology for Derivation of Pesticide Sediment Quality Criteria for the Protection of Aquatic Life, Report prepared by the University of California Davis for the Central Valley Regional Water Quality Control Board. Available at:  
[http://www.swrcb.ca.gov/rwqcb5/water\\_issues/tmdl/central\\_valley\\_projects/central\\_valley\\_pesticides/sediment\\_quality\\_criteria\\_method\\_development/index.shtml](http://www.swrcb.ca.gov/rwqcb5/water_issues/tmdl/central_valley_projects/central_valley_pesticides/sediment_quality_criteria_method_development/index.shtml)
- Funk M, Grote C. (2004) Effect of reg. no. 5300605 (metabolite of BAS 350 I, RPA 200766) on the mortality of *Chironomus riparius* in a 48 hours static, acute toxicity test. BASF Agricultural Center Limburgerhof. Limburgerhof, Germany. Study code 198235. Submitted to BASF Aktiengesellschaft, Limburgerhof, Germany. US EPA MRID 46376701.
- Goel A, McConnell LL and Torrents A. (2007) Determination of vapor pressure-temperature relationships of current-use pesticides and transformation products. *Journal of Environmental Science and Health Part B*, 42(4), 343-349.
- Hamernik KL. (1997) Fipronil. Toxicological and Environmental Evaluations. Monographs of Toxicological Evaluations. 932, Part II. FAO/WHO Joint Meeting of Pesticide Residues. Office of Pesticide Programs, US Environmental Protection Agency, Washington, DC, USA.
- Helsten BR and Solatycki AM. (1994) 14-day acute oral LD50 study with M & B 46513 in mallard duck. Bio-Life Associates, Limited, Neillsville, Wisconsin. Laboratory project ID 108-027-04. Submitted to Rhone-Poulenc Ag Company, Research Park Triangle, North Carolina. USEPA MRID 43776602.
- Hoberg JR (1993) MB46030-Toxicity to the freshwater green alga, *Selenastrum capricornutum*. Springborn Laboratories, Inc., Wareham, Massachusetts. Laboratory study number 10566.0393.6271.430. Submitted to Rhone-Poulenc Ag Company, Research Triangle, North Carolina. CA DPR 157291.
- Hoberg JR (1993a) MB46513-Toxicity to the freshwater green alga, *Selenastrum capricornutum*. Springborn Laboratories, Inc., Wareham, Massachusetts. Laboratory study number 10566.0492.6243.430. Submitted to Rhone-Poulenc Ag Company, Research Triangle, North Carolina. USEPA MRID 43279705.
- Hoberg JR (1993) MB46030-Toxicity to the freshwater diatom, *Navicula pelliculosa*. Springborn Laboratories, Inc., Wareham, Massachusetts. Laboratory study number 10566.0393.6272.440. Submitted to Rhone-Poulenc Ag Company, Research Triangle, North Carolina. CA DPR 157294.

- Hoberg JR. (1993) MB 46030-Toxicity to duckweed *Lemna gibba*. Springborn Laboratories, Inc., Wareham, Massachusetts. Laboratory study number 10566.0.393.6274.410. Submitted to Rhone-Poulenc Ag Company, Research Triangle, North Carolina. USEPA MRID 42918656. CA DPR 157293.
- Iwafune T, Yokoyama A, Nagai T and Horio T. (2011) Evaluation of the risk of mixtures of paddy insecticides and their transformation products to aquatic organisms in the Sakura River, Japan. *Environmental Toxicology and Chemistry*, 30(8), 1834-1842.
- Janson, GM. (2014) Chronic toxicity of the BAS 350 I metabolite MB46136 (Reg. No. 4673253) to *Daphnia magna* Straus in a 21 day semi-static test. BASF SE, Limburgerhof, Germany. Study code 367103. Submitted to BASF Corporation, Research Triangle Park, North Carolina. CA DPR277084.
- Key PB, Chung KW, Opatkiewicz AD, Wirth EF and Fulton MH. (2003) Toxicity of the insecticides fipronil and endosulfan to selected life stages of the grass shrimp (*Palaemonetes pugio*). *Bulletin of environmental contamination and toxicology*, 70(3), 0533-0540.
- Key P, Chung K, Siewicki T and Fulton M. (2007) Toxicity of three pesticides individually and in mixture to larval grass shrimp (*Palaemonetes pugio*). *Ecotoxicology and Environmental Safety*, 68(2), 272-277.
- Kolk, J. (2002) Chronic toxicity test with midge larvae (*Chironomus riparius*) in a water/sediment system. Springborn Laboratories (Europe), Horn, Switzerland. Laboratory ID 1067.006.173. Submitted to Bayer CropScience, Research Triangle Park, North Carolina. US EPA MRID 45851001.
- Konwick BJ, Fisk AT, Garrison AW, Avants JK and Black MC. (2005) Acute enantioselective toxicity of fipronil and its desulfinyl photoproduct to *Ceriodaphnia dubia*. *Environmental Toxicology and Chemistry*, 24(9), 2350-2355.
- Lima W. (2000) [<sup>14</sup>C]MB 46136-Life-cycle toxicity test with mysids (*Mysidopsis bahia*). Springborn Laboratories, Inc., Wareham, Massachusetts. Laboratory study number 13726.6116. Submitted to Aventis CropScience, Research Triangle, North Carolina. USEPA MRID 45259203.
- Lin K, Haver D, Oki L and Gan J. (2008) Transformation and sorption of fipronil in urban stream sediments. *Journal of agricultural and food chemistry*, 56(18), 8594-8600.
- Lin K, Haver D, Oki L and Gan J. (2009) Persistence and sorption of fipronil degradates in urban stream sediments. *Environmental Toxicology and Chemistry*, 28(7), 1462-1468.

- Lizotte Jr RE, Knight SS, Shields Jr FD and Bryant CT. (2009) Effects of an atrazine, metolachlor and fipronil mixture on *Hyalella azteca* (Saussure) in a modified backwater wetland. *Bulletin of environmental contamination and toxicology*, 83(6), 836-840.
- Machado MW. (1992a) The toxicity to rainbow trout (*Oncorhynchus mykiss*) during an early life-stage exposure. Springborn Laboratories, Inc., Wareham, Massachusetts. Laboratory study number 10566.0.391.6209.121. Submitted to Rhone-Poulenc Ag Company, Research Triangle, North Carolina. USEPA MRID 42918627. CA DPR 157287.
- Machado MW. (1993) MB 46030-Acute toxicity to sheepshead minnow (*Cyprinodon variegatus*) under flow-through conditions. Springborn Laboratories, Inc., Wareham, Massachusetts. Laboratory study number 10566.0393.6267.505. Submitted to Rhone-Poulenc Ag Company, Research Triangle, North Carolina. USEPA MRID 43291702. CA DPR 157284.
- Machado MW. (1994) Fipronil-Chronic toxicity to mysids (*Mysidopsis bahia*) under flow-through conditions. Springborn Laboratories, Inc., Wareham, Massachusetts. Laboratory study number 10566.1294.6353.530. Submitted to Rhone-Poulenc Ag Company, Research Triangle, North Carolina. USEPA MRID 43681201.
- Machado MW. (1994) MB 46030-Acute toxicity to mysids (*Mysidopsis bahia*) under static conditions. Springborn Laboratories, Inc., Wareham, Massachusetts. Laboratory study number 10566.0394.6340.510. Submitted to Rhone-Poulenc Ag Company, Research Triangle, North Carolina. USEPA MRID 43279701. CA DPR 157286.
- Maul JD, Brennan AA, Harwood AD and Lydy MJ. (2008) Effect of sediment-associated pyrethroids, fipronil, and metabolites on *Chironomus tentans* growth rate, body mass, condition index, immobilization, and survival. *Environmental Toxicology and Chemistry*, 27(12), 2582-2590.
- McNamara PC. (1992) (M&B 46136)-Chronic toxicity to daphnids (*Daphnia magna*) under flow-through conditions. Springborn Laboratories, Inc., Wareham, Massachusetts. Laboratory study number 10566.1090.6175.130. Submitted to Rhone-Poulenc Ag Company, Research Triangle, North Carolina. CA DPR 157305.
- McNamara PC. (1990a) Acute toxicity to daphnids (*Daphnia magna*) during a 48-hour flow-through exposure. Springborn Laboratories, Inc., Wareham, Massachusetts. Laboratory study number 10566.1089.6146.115. Submitted to Rhone-Poulenc Ag Company, Research Triangle, North Carolina. USEPA MRID 42918625. CA DPR 157282 (1990) and 157283 (1996 duplicate).
- McNamara PC. (1990b) (M & B 45950)-Acute toxicity to daphnids (*Daphnia magna*) during a 48-hour flow-through exposure. Springborn Laboratories, Inc., Wareham, Massachusetts.

- Laboratory study number 10566.1089.6147.115. Submitted to Rhone-Poulenc Ag Company, Research Triangle, North Carolina. USEPA MRID 42918669. CA DPR 157307.
- McNamara PC. (1990c) (M&B 46136)-Acute toxicity to daphnids (*Daphnia magna*) during a 48-hour flow-through exposure. Springborn Laboratories, Inc., Wareham, Massachusetts. Laboratory study number 10566.1089.6148.115. Submitted to Rhone-Poulenc Ag Company, Research Triangle, North Carolina. USEPA MRID 42918671. CA DPR 157304.
- McNamara PC. (1990d) The chronic toxicity of M&B 46030 to *Daphnia magna* under flow-through conditions. Springborn Laboratories, Inc., Wareham, Massachusetts. Laboratory study number 10566.1089.6146.130. Submitted to Rhone-Poulenc Ag Company, Research Triangle, North Carolina. USEPA MRID 42918626. CA DPR 157288.
- McNamara PC. (1990e) (M&B 45950)-Chronic toxicity to daphnids (*Daphnia magna*) under flow-through conditions. Springborn Laboratories, Inc., Wareham, Massachusetts. Laboratory study number 10566.1089.6147.130. Submitted to Rhone-Poulenc Ag Company, Research Triangle, North Carolina. USEPA MRID 42918670. CA DPR 157308.
- Mislankar SG. (1997) MB 46513: Aerobic soil metabolism. Rhone-Poulenc Ag Company, Research Triangle Park, North Carolina. Laboratory study number EC-95-318. USEPA MRID 44262830.
- NOAA. (1999) Sediment Quality Guidelines Developed for the National Status and Trends Program. National Oceanographic and Atmospheric Agency Office of Response and Restoration, Department of Commerce. URL<  
[http://archive.orr.noaa.gov/book\\_shelf/121\\_sedi\\_qual\\_guide.pdf](http://archive.orr.noaa.gov/book_shelf/121_sedi_qual_guide.pdf)>
- Overmyer JP, Mason BN and Armbrust KL. (2005) Acute toxicity of imidacloprid and fipronil to a nontarget aquatic insect, *Simulium vittatum* Zetterstedt cytospecies IS-7. *Bulletin of environmental contamination and toxicology*, 74(5), 872-879.
- Overmyer JP, Rouse DR, Avants JK, Garrison AW, DeLorenzo ME, Chung KW, Key PB, Wilson WA and Black MC. (2007) Toxicity of fipronil and its enantiomers to marine and freshwater non-targets. *Journal of Environmental Science and Health Part B*, 42(5), 471-480.
- Pedersen CA. (1993a) M & B 46030 technical: 21-day acute oral LD<sub>50</sub> study in mallard ducks. Bio-Life Associates, Limited, Neillsville, Wisconsin. Laboratory project ID 89 DD 70. Submitted to Rhone-Poulenc Ag Company, Research Park Triangle, North Carolina. USEPA MRID 42918616.

- Pedersen CA. (1993b) M & B 46030 technical: 22-day acute dietary LD<sub>50</sub> study in mallard duck. Bio-Life Associates, Limited, Neillsville, Wisconsin. Laboratory project ID 89 DC 132. Submitted to Rhone-Poulenc Ag Company, Research Park Triangle, North Carolina. USEPA MRID 42918621.
- Pedersen CA and Lesar CL. (1993) M&B 46030 technical: toxicity and reproduction study in mallard ducks. Bio-Life Associates, Limited, Neillsville, Wisconsin. Laboratory project ID 108-013-08. Submitted to Rhone-Poulenc Ag Company, Research Park Triangle, North Carolina. USEPA MRID 4291862. CADPR ID 157278.
- Picard CR (2015a) 10-day toxicity test exposing freshwater amphipods (*Hyalella azteca*) to fipronil sulfide (MB45950) applied to sediment under static-renewal conditions following OPPTS Draft Guideline 850.1735. Springborn Viscent, Wareham, Massachusetts. Laboratory project ID13798.6353. Submitted to Bayer CropScience, Research Triangle Park, North Carolina. CA DPR study ID: 283832.
- Picard CR (2015b) 10-day toxicity test exposing freshwater amphipods (*Hyalella azteca*) to fipronil sulfone (MB43136) applied to sediment under static-renewal conditions following OPPTS Draft Guideline 850.1735. Springborn Viscent, Wareham, Massachusetts. Laboratory project ID13798.6356. Submitted to Bayer CropScience, Research Triangle Park, North Carolina. CA DPR study ID: 283835.
- Picard CR (2015c) 10-day toxicity test exposing freshwater amphipods (*Hyalella azteca*) to fipronil-desulfinyl (MB46513) applied to sediment under static-renewal conditions following OPPTS Draft Guideline 850.1735. Springborn Viscent, Wareham, Massachusetts. Laboratory project ID13798.6360. Submitted to Bayer CropScience, Research Triangle Park, North Carolina. CA DPR study ID: 283837.
- Picard CR (2015d) 10-day toxicity test exposing estuarine amphipods (*Leptocheirus plumulosus*) to fipronil applied to sediment under static renewal conditions following OPPTS Draft Guideline 850.1740. Performed by Springborn Viscent, Wareham, MA. Laboratory project ID13798.6351. Submitted to Bayer CropScience, Research Triangle Park, North Carolina. CA DPR study ID: 283830.
- Picard CR (2015e) 10-day toxicity test exposing estuarine amphipods (*Leptocheirus plumulosus*) to fipronil sulfide (MB45950) applied to sediment under static conditions following OPPTS Draft Guideline 850.1740. Performed by Springborn Viscent, Wareham, MA. Laboratory project ID13798.6354. Submitted to Bayer CropScience, Research Triangle Park, North Carolina. CA DPR study ID: 283834.
- Picard CR (2015f) 10-day toxicity test exposing estuarine amphipods (*Leptocheirus plumulosus*) to fipronil sulfone (MB46136) applied to sediment under static conditions following OPPTS Draft Guideline 850.1740. Performed by Springborn Viscent, Wareham, MA.

- Laboratory project ID13798.6357. Submitted to Bayer CropScience, Research Triangle Park, North Carolina. CA DPR study ID: 283836.
- Picard CR (2015g) 10-day toxicity test exposing estuarine amphipods (*Leptocheirus plumulosus*) to fipronil desulfinyl (MB46513) applied to sediment under static conditions following OPPTS Draft Guideline 850.1740. Performed by Springborn Viscent, Wareham, MA. Laboratory project ID13798.6361. Submitted to Bayer CropScience, Research Triangle Park, North Carolina. CA DPR study ID: 283838.
- Picard CR (2015h) 10-day toxicity test exposing freshwater amphipods (*Hyalella azteca*) to fipronil applied to sediment under static-renewal conditions following OCSPP Draft Guideline 850.1735. Smithers Viscent, Wareham, Massachusetts. Laboratory project ID13798.6350. Submitted to Bayer CropScience, Research Triangle Park, North Carolina. CA DPR study ID: 283829.
- Putt AE. (1992a) MB46513-Chronic toxicity to daphnids (*Daphnia magna*) under static renewal conditions. Springborn Laboratories, Inc., Wareham, Massachusetts. Laboratory study number 10566.1090.6176.130. Submitted to Rhone-Poulenc Ag Company, Research Triangle, North Carolina. USEPA MRID 43279704. CA DPR 157300.
- Putt AE. (2000a) [<sup>14</sup>C]MB 45950-Acute toxicity to mysids (*Mysidopsis bahia*) under static acute conditions. Springborn Laboratories, Inc., Wareham, Massachusetts. Laboratory study number 10566.6547. Submitted to Rhone-Poulenc Ag Company, Research Triangle, North Carolina. USEPA MRID 45156302.
- Putt AE. (2000b) [<sup>14</sup>C]MB 46136-Acute toxicity to mysids (*Mysidopsis bahia*) under static acute conditions. Springborn Laboratories, Inc., Wareham, Massachusetts. Laboratory study number 10566.6545. Submitted to Rhone-Poulenc Ag Company, Research Triangle, North Carolina. USEPA MRID 45156301.
- Putt AE. (2000c) [<sup>14</sup>C]MB 46513-Acute toxicity to mysids (*Mysidopsis bahia*) under static acute conditions. Springborn Laboratories, Inc., Wareham, Massachusetts. Laboratory study number 10566.6549. Submitted to Rhone-Poulenc Ag Company, Research Triangle, North Carolina. USEPA MRID 45120001.
- Putt, AE. (2000d) [<sup>14</sup>C]MB 45950 – Toxicity to midge (*Chironomus tentans*) during a 10-day sediment exposure. Springbord Laboratories, Inc., Wareham, Massachusetts. Laboratory project ID 10566.6536. Submitted to Aventis CropScience, Research Triangle Park, North Carolina. US EPA MRID 45084801.
- Putt, AE. (2000e) [<sup>14</sup>C]MB 43163 – Toxicity to midge (*Chironomus tentans*) during a 10-day sediment exposure. Springbord Laboratories, Inc., Wareham, Massachusetts. Laboratory

- project ID 10566.6537. Submitted to Aventis CropScience, Research Triangle Park, North Carolina. US EPA MRID 45175901.
- Putt, AE. (2001) [<sup>14</sup>C]MB 46513 – Toxicity to midge (*Chironomus tentans*) during a 10-day sediment exposure. Springbord Laboratories, Inc., Wareham, Massachusetts. Laboratory project ID 10566.6538. Submitted to Aventis CropScience, Research Triangle Park, North Carolina. US EPA MRID 45375901.
- Putt AE. (2003a) Fipronil-Acute toxicity to mayfly nymphs (*Hexagenia* sp.) under static-renewal conditions. Springborn Smithers Laboratories, Wareham, Massachusetts. Laboratory study number 986.6160. Submitted to BSF, Research Triangle, North Carolina. USEPA MRID
- Putt AE. (2003b) Fipronil-Acute toxicity to clams (*Corbicula fluminea*) under static-renewal conditions. Springborn Smithers Laboratories, Wareham, Massachusetts. Laboratory study number 986.6161. Submitted to BASF Corporation, Research Triangle Park, North Carolina. USEPA MRID 46329904.
- Putt AE. (2003c) Fipronil-Acute toxicity to oligochaetes (*Lumbriculus variegatus*) under static-renewal conditions. Springborn Smithers Laboratories, Wareham, Massachusetts. Laboratory study number 986.6162. Submitted to BASF Corporation, Research Triangle Park, North Carolina. USEPA MRID 46329903.
- Putt, AE. (2003d) Fipronil-Toxicity to midge (*Chironomus tentans*) during a 10-day sediment exposure. Springborn Laboratories, Inc., Wareham, Massachusetts. Laboratory project ID 13798.6106. Submitted to Bayer CropScience, Research Triangle Park, North Carolina. US EPA MRID 45878001.
- Qu H, Ma RX, Liu DH, Wang P, Huang LD, Qiu XX and Zhou ZQ. (2014) Enantioselective toxicity and degradation of the chiral insecticide fipronil in *Scenedesmus obliquus* suspension system. *Environmental toxicology and chemistry*, 33(11), 2516-2521.
- Raimondo S, Jackson CR, Barron MG (2013) Web-based interspecies correlation estimation (Web-ICE) for acute toxicity: User manual. Version 3.2. EPA/600/R-12/603. US Environmental Protection Agency, Office of Research and Development, Gulf Ecology Division, Gulf Breeze, FL. Available at:  
[https://www3.epa.gov/webice/webice/WebICE\\_User\\_manual.pdf](https://www3.epa.gov/webice/webice/WebICE_User_manual.pdf)
- Ruby A. (2013) Review of pyrethroid, fipronil and toxicity monitoring data from California urban watersheds. Report prepared for the California Stormwater Quality Association (CASQA). Prepared by Armand Ruby Consulting.
- Schlenk D, Huggett DB, Allgood J, Bennett E, Rimoldi J, Beeler AB, Block D, Holder AW, Hovinga R and Bedient P. (2001) Toxicity of fipronil and its degradation products to

- Procambarus sp.: Field and laboratory studies. *Archives of Environmental Contamination and Toxicology*, 41(3), 325-332.
- Sigma-Aldrich. (2016a) Fipronil sulfide. Safety Data Sheet, version 5.0. Product number 34520. Sigma-Aldrich, Saint Louis, Missouri, February 6, 2015. URL <<http://www.sigmaaldrich.com/safety-center.html>> (accessed July 16, 2016).
- Sigma-Aldrich. (2016b) Fipronil carboxamide. Safety Data Sheet, version 5.0. Product number 34519. Sigma-Aldrich, Saint Louis, Missouri, February 6, 2015. URL <<http://www.sigmaaldrich.com/safety-center.html>> (accessed July 16, 2016).
- Sigma-Aldrich. (2016c) Fipronil sulfone. Safety Data Sheet, version 5.3. Product number 32333. Sigma-Aldrich, Saint Louis, Missouri, February 6, 2015. URL <<http://www.sigmaaldrich.com/safety-center.html>> (accessed July 16, 2016).
- Sigma-Aldrich. (2016d) Fipronil desulfinyl. Safety Data Sheet, version 5.4. Product number 41865. Sigma-Aldrich, Saint Louis, Missouri, February 6, 2015. URL <<http://www.sigmaaldrich.com/safety-center.html>> (accessed July 16, 2016).
- Sousa JV. (1998a) Fipronil technical-Early life-stage toxicity test with sheepshead minnow (*Cyprinodon variegatus*). Springborn Laboratories, Inc., Wareham, Massachusetts. Laboratory study number 10566.0796.6402.520. Submitted to Rhone-Poulenc Ag Company, Research Triangle, North Carolina. USEPA MRID 44605501. CA DPR 169427.
- Sousa JV. (1998b) Fipronil technical-Early life-stage toxicity test with sheepshead minnow (*Cyprinodon variegatus*). Springborn Laboratories, Inc., Wareham, Massachusetts. Laboratory study number 10566.0797.6438.520. Submitted to Rhone-Poulenc Ag Company, Research Triangle, North Carolina. USEPA MRID 44605502. CA DPR 169428.
- Spomer NA and Kamble ST. (2010) Sorption and desorption of fipronil in Midwestern soils. *Bulletin of environmental contamination and toxicology*, 84(2), 264-268.
- Stark JD, and Vargas RI. (2005) Toxicity and hazard assessment of fipronil to *Daphnia pulex*. *Ecotoxicology and environmental safety* 62.1, 11-16.
- Stevens MM, Burdett AS, Mudford EM, Helliwell S and Doran G. (2011) The acute toxicity of fipronil to two non-target invertebrates associated with mosquito breeding sites in Australia. *Acta tropica*, 117(2), 125-130.
- Stratman KN, Wilson PC, Overholt WA, Cuda JP and Netherland MD. (2013) Toxicity of fipronil to the midge, *Cricotopus lebetis* Sublette. *Journal of Toxicology and Environmental Health, Part A*, 76(12), 716-722.

- TenBrook PL, Tjeerdema RS (2006) Methodology for derivation of pesticide water quality criteria for the protection of aquatic life in the Sacramento and San Joaquin River Basins. Phase I: Review of existing methodologies. Report prepared for the Central Valley Regional Water Quality Control Board, Rancho Cordova, CA.
- TenBrook PL, Palumbo AJ, Fojut TL, Tjeerdema RS, Hann P, Karkoski J (2009) Methodology for derivation of pesticide water quality criteria for the protection of aquatic life in the Sacramento and San Joaquin River Basins. Phase II: methodology development and derivation of chlorpyrifos criteria. Report prepared for the Central Valley Regional Water Quality Control Board, Rancho Cordova, CA.
- TenBrook PL, Palumbo AJ, Fojut TL, Hann P, Karkoski J, Tjeerdema RS (2010) The University of California-Davis methodology for deriving aquatic life pesticide water quality criteria. *Rev Environ Contamin Toxicol* 209:1-155.
- Thuyet DQ, Watanabe H, Yamazaki K and Takagi K. (2011) Photodegradation of imidacloprid and fipronil in rice-paddy water. *Bulletin of environmental contamination and toxicology*, 86(5), 548-553.
- Tomlin C (1997) *The Pesticide Manual. (A World Compendium.) 10th Edition*. The British Crop Protection Council and The Royal Society of Chemistry, Surrey, England and Cambridge, England.
- USEPA (1985) Guidelines for deriving numerical national water quality criteria for the protection of aquatic organisms and their uses, PB-85-227049, section III-B-1. US Environmental Protection Agency, National Technical Information Service, Springfield, VA. URL<  
[http://www.waterboards.ca.gov/waterrights/water\\_issues/programs/bay\\_delta/deltaflow/docs/exhibits/sac\\_rcsd/srcsd\\_exh1w.pdf](http://www.waterboards.ca.gov/waterrights/water_issues/programs/bay_delta/deltaflow/docs/exhibits/sac_rcsd/srcsd_exh1w.pdf)>
- USEPA (2000) Methods for measuring the toxicity and bioaccumulation of sediment-associated contaminants with freshwater invertebrates. Second edition. U.S. Environmental Protection Agency, Washington, DC. EPA 600/R-99/064.
- USEPA. (2011) Registration Review Preliminary Problem Formulation for Ecological Risk and Environmental Fate, Endangered Species, and Drinking Water Assessments for Fipronil. United States Environmental Protection Agency, Washington, DC, USA.
- USEPA (2015a) Estimation Programs Interface Suite™ for Microsoft® Windows, v 4.11. United States Environmental Protection Agency, Washington, DC, USA.
- USEPA (2015b) Fipronil; Tolerances for residues, 40 CFR 180.517. US Environmental Protection Agency, National Technical Information Service, Springfield, VA. Available

at: <https://www.gpo.gov/fdsys/pkg/CFR-2015-title40-vol24/pdf/CFR-2015-title40-vol24-sec180-517.pdf>

- USEPA. (2015c) National Ambient Air Quality Standards website. United States Environmental Protection Agency, Washington, DC. URL < <https://www.epa.gov/criteria-air-pollutants/naaqs-table> >
- USFDA (2000) Guidance for Industry: Action Levels for Poisonous or Deleterious Substances in Human Food and Animal Feed. United States Food and Drug Administration, Washington, DC. Available at: <http://www.fda.gov/Food/GuidanceRegulation/GuidanceDocumentsRegulatoryInformation/ChemicalContaminantsMetalsNaturalToxinsPesticides/ucm077969.htm>
- Walse SS, Morgan SL, Kong L and Ferry JL. (2004) Role of dissolved organic matter, nitrate, and bicarbonate in the photolysis of aqueous fipronil. *Environmental science & technology*, 38(14), 3908-3915.
- Walse SS, Pennington PL, Scott GI and Ferry JL. (2004) The fate of fipronil in modular estuarine mesocosms. *Journal of Environmental Monitoring*, 6(1), 58-64.
- Ward GS. (1991a) M&B 46030: Acute toxicity to bluegill, *Lepomis macrochirus*, under flow-through test conditions. Toxikon Environmental Services, Jupiter, Florida. Laboratory project ID J9005012b. Submitted to Rhone-Poulenc Ag Company, Research Triangle Park, North Carolina. US EPA MRID 42918624. CA DPR 157279.
- Ward GS. (1991b) M&B 46030: Acute toxicity to rainbow trout, *Oncorhynchus mykiss*, under flow-through test conditions. Toxikon Environmental Services, Jupiter, Florida. Laboratory project ID J9005012a. Submitted to Rhone-Poulenc Ag Company, Research Triangle Park, North Carolina. US EPA MRID 42977902. CA DPR 157280.
- Waring AR. (1993) (<sup>14</sup>C)-MB 46,030: Aerobic soil metabolism. Hazelton UK, North Yorkshire, England. Laboratory project number 68/109-1015. Submitted to Rhone-Poulenc Agriculture, Essex, England. USEPA MRID 42918663.
- Weston DP and Lydy MJ. (2014) Toxicity of the insecticide fipronil and its degradates to benthic macroinvertebrates of urban streams. *Environmental science & technology*, 48(2), 1290-1297.
- Wilson WA, Konwick BJ, Garrison AW, Avants JK and Black MC. (2008) Enantioselective chronic toxicity of fipronil to *Ceriodaphnia dubia*. *Archives of environmental contamination and toxicology*, 54(1), 36-43.

Wirth EF, Pennington PL, Lawton JC, DeLorenzo ME, Bearden D, Shaddrix B, Sivertsen S and Fulton MH. (2004) The effects of the contemporary-use insecticide (fipronil) in an estuarine mesocosm. *Environmental Pollution*, 131(3), 365-371.

Yokoyama A, Ohtsu K, Iwafune T, Nagai T, Ishihara S, Kobara Y, Horio T and Endo S. (2009) A useful new insecticide bioassay using first-instar larvae of a net-spinning caddisfly, *Cheumatopsyche brevilineata* (Trichoptera: Hydropsychidae). *Journal of Pesticide Science*, 34(1), 13-20.

# Data Tables

To make the data tables easier to follow for fipronil and its degradates, data for each compound is color-coded in each table.

|            |          |                  |                  |                     |                      |                                       |
|------------|----------|------------------|------------------|---------------------|----------------------|---------------------------------------|
| Color key: | Fipronil | Fipronil-sulfide | Fipronil-sulfone | Fipronil-desulfinyl | Fipronil-carboxamide | Fipronil-destrifluoromethyl-sulfonate |
|------------|----------|------------------|------------------|---------------------|----------------------|---------------------------------------|

Table 3 Final acute toxicity data used to calculate fipronil WQC.

All studies were rated relevant and reliable (RR).

| Species                         | Common name     | Family          | Test type | Duration (d) | Temp (°C) | Endpoint          | Age/ size             | Nom/ Meas | LC/EC <sub>50</sub> (95% CI) (µg/L)                                | Reference          |
|---------------------------------|-----------------|-----------------|-----------|--------------|-----------|-------------------|-----------------------|-----------|--|--------------------|
| <i>Baetis tricaudatus</i>       | Mayfly          | Baetidae        | S         | 48 h         | 17        | Survival          | NR                    | Meas      | <b>0.0519</b> (0.0373-0.0720)                                      | Weston 2014        |
| <i>Chironomus dilutus</i>       | Chironomids     | Chironomidae    | S         | 96 h         | 23        | Survival          | NR                    | Meas      | <b>0.0300</b> (0.0233-0.0360)                                      | Weston 2014        |
| <i>Ceriodaphnia dubia</i>       | Daphnid         | Daphniidae      | S         | 24 h         | 25        | Impaired movement | <24 h                 | Nom       | <b>Racemate: 33.3</b> (3.1)<br>(+): 18.1 (4.7)<br>(-): 65.2 (15.8) | Wilson et al. 2008 |
| <i>Dipheter hageni</i>          | Mayfly          | Baetidae        | S         | 24 h         | 18        | Survival          | NR                    | Meas      | 0.163 (0.107-0.208)  | Weston 2014        |
| <i>Daphnia magna</i>            | Daphnid         | Daphniidae      | FT        | 48 h         | 20        | Immobilization    | <24 h                 | Meas      | <b>190</b> (110-280)   | McNamara 1990a     |
| <i>Fallceon quilleri</i>        | Mayfly          | Baetidae        | S         | 48 h         | 23        | Survival          | NR                    | Meas      | <b>0.0707</b> (0.0.365-0.0935)                                     | Weston 2014        |
| <i>Helicopsyche</i> sp.         | Caddisfly       | Helicopsychidae | S         | 96 h         | 13        | Survival          | NR                    | Meas      | <b>0.267</b> (0.210-0.338)   | Weston 2014        |
| <i>Hexagenia</i> sp.            | Mayfly          | Ephemeridae     | SR        | 96 h         | 22        | Survival          | 60 d, 6.7 mm          | Meas      | 0.44 (0.39-0.49)   | Putt 2003a         |
| <i>Hexagenia</i> sp.            | Mayfly          | Ephemeridae     | S         | 96 h         | 18        | Survival          | NR                    | Meas      | 0.480 (0.348-0.603)  | Weston 2014        |
| <i>Hyalella azteca</i>          | Amphipod        | Hyaellidae      | S         | 96 h         | 23        | Immobilization    | NR                    | Meas      | <b>0.727</b> (0.648-0.816)   | Weston 2014        |
| <i>Hydropsyche</i> sp.          | Caddisfly       | Hydropsychidae  | S         | 96 h         | 12        | Survival          | NR                    | Meas      | <b>0.602</b> (0.417-0.788)   | Weston 2014        |
| <i>Ictalurus punctatus</i>      | Channel catfish | Ictaluridae     | FT        | 96 h         | 22        | Survival          | 1.7 g, 59 mm          | Meas      | <b>560</b> (320-1200)  | Dionne 1997        |
| <i>Isoperla quinquepunctata</i> | Stonefly        | Perlodidae      | S         | 96 h         | 13        | Survival          | NR                    | Meas      | <b>0.101</b> (0.0846-0.119)  | Weston 2014        |
| <i>Lepomis macrochirus</i>      | Bluegill        | Centrarchidae   | FT        | 96 h         | 21        | Survival          | 17-23 mm, 0.12-0.44 g | Meas      | <b>85.2</b> (74.2-99.0)  | Ward 1991a         |
| <i>Nectopsyche</i> sp.          | Caddisfly       | Leptoceridae    | S         | 48 h         | 23        | Survival          | NR                    | Meas      | <b>0.634</b> (0.531-0.756)   | Weston 2014        |

| Species                     | Common name    | Family     | Test type | Duration (d) | Temp (°C) | Endpoint | Age/ size         | Nom/ Meas | LC/EC <sub>50</sub> (95% CI) (µg/L)  | Reference            |
|-----------------------------|----------------|------------|-----------|--------------|-----------|----------|-------------------|-----------|--|----------------------|
| <i>Oncorhynchus mykiss</i>  | Rainbow trout  | Salmonidae | FT        | 96 h         | 12        | Survival | 36 mm, 0.98 g     | Meas      | <b>248</b> (160-∞)   | Ward 1991b           |
| <i>Pimephales promelas</i>  | Fathead minnow | Cyprinidae | SR        | 7 d          | 25        | Survival | Larvae            | Nom       | <b>Racemate: 208</b> (191-224)<br>(+): 227 (201-243)<br>(-): 365 (333-397) | Baird 2013           |
| <i>Serratella micheneri</i> | Mayfly         | Baetidae   | S         | 48 h         | 23        | Survival | NR                | Meas      | <b>0.589</b> (0.478-0.742)   | Weston 2014          |
| <i>Simulium vittatum</i>    | Black fly      | Simuliidae | S         | 48 h         | 20        | Survival | 5th instar larvae | Meas      | <b>0.19</b> (0.16-0.21)  | Overmyer et al. 2005 |

Table 4 Final acute toxicity data used to calculate fipronil-sulfide WQC.

All studies were rated relevant and reliable (RR).

| Species                         | Common name | Family          | Test type | Duration (d) | Temp (°C) | Endpoint       | Age/size | Nom/Meas | LC/EC <sub>50</sub> (95% CI) (µg/L) | Reference                     |
|---------------------------------|-------------|-----------------|-----------|--------------|-----------|----------------|----------|----------|-------------------------------------|-------------------------------|
| <i>Baetis tricaudatus</i>       | Mayfly      | Baetidae        | S         | 48 h         | 12        | Immobilization | NR       | Meas     | <b>0.0803</b> (0.0531-0.108)        | Weston 2014                   |
| <i>Chironomus dilutus</i>       | Chironomids | Chironomidae    | S         | 96 h         | 23        | Immobilization | NR       | Meas     | <b>0.0093</b> (0.0076-0.0114)       | Weston 2014<br>McNamara 1990b |
| <i>Daphnia magna</i>            | Daphnid     | Daphniidae      | FT        | 48 h         | 20        | Immobilization | <24 h    | Meas     | <b>100</b> (81-130)                 | Weston 2014                   |
| <i>Helicopsyche sp.</i>         | Caddisfly   | Helicopsychidae | S         | 96 h         | 13        | Immobilization | NR       | Meas     | <b>0.177</b> (0.146-0.216)          | Weston 2014                   |
| <i>Hyalella azteca</i>          | Amphipod    | Hyalellidae     | S         | 96 h         | 23        | Immobilization | NR       | Meas     | <b>0.375</b> (0.325-0.433)          | Weston 2014                   |
| <i>Isoperla quinquepunctata</i> | Stonefly    | Perlodidae      | S         | 96 h         | 13        | Immobilization | NR       | Meas     | <b>0.0422</b> (0.0371-0.0474)       | Weston 2014                   |
| <i>Nectopsyche sp.</i>          | Caddisfly   | Leptoceridae    | S         | 48 h         | 23        | Immobilization | NR       | Meas     | <b>0.0285</b> (0.0187-0.0365)       | Weston 2014                   |

Table 5 Final acute toxicity data used to calculate fipronil-sulfone WQC.

All studies were rated relevant and reliable (RR).

| Species                         | Common name   | Family           | Test type | Duration (d) | Temp (°C) | Endpoint       | Age/size      | Nom/Meas | LC/EC <sub>50</sub> (95% CI) (µg/L) | Reference         |
|---------------------------------|---------------|------------------|-----------|--------------|-----------|----------------|---------------|----------|-------------------------------------|-------------------|
| <i>Baetis tricaudatus</i>       | Mayfly        | Baetidae         | S         | 48 h         | 12        | Immobilization | NR            | Meas     | <b>0.075</b> (0.0449-0.109)         | Weston 2014       |
| <i>Chironomus dilutus</i>       | Chironomids   | Chironomidae     | S         | 96 h         | 23        | Immobilization | NR            | Meas     | <b>0.0079</b> (0.0050-0.0103)       | Weston 2014       |
| <i>Daphnia magna</i>            | Daphnid       | Daphniidae       | FT        | 48 h         | 20        | Immobilization | <24 h         | Meas     | <b>29</b> (20-38)                   | McNamara 1990c    |
| <i>Dipheter hageni</i>          | Mayfly        | Baetidae         | S         | 48 h         | 18        | Immobilization | NR            | Meas     | <b>0.0926</b> (0.0565-0.128)        | Weston 2014       |
| <i>Fallceon quilleri</i>        | Mayfly        | Baetidae         | S         | 48 h         | 23        | Immobilization | NR            | Meas     | <b>0.0717</b> (0.0523-0.0906)       | Weston 2014       |
| <i>Helicopsyche sp.</i>         | Caddisfly     | Helicopsychidae  | S         | 96 h         | 13        | Immobilization | NR            | Meas     | <b>0.0738</b> (0.0386-0.140)        | Weston 2014       |
| <i>Hexagenia sp.</i>            | Mayfly        | Ephemeraeidae    | S         | 96 h         | 18        | Immobilization | NR            | Meas     | <b>0.163</b> (0.051-0.223)          | Weston 2014       |
| <i>Hyaella azteca</i>           | Amphipod      | Hyaellidae       | S         | 96 h         | 23        | Immobilization | NR            | Meas     | <b>0.155</b> (0.122-0.179)          | Weston 2014       |
| <i>Hydropsyche sp.</i>          | Caddisfly     | Hydropsychidae   | S         | 96 h         | 12        | Immobilization | NR            | Meas     | <b>0.0729</b> (0.0565-0.0940)       | Weston 2014       |
| <i>Isoperla quinquepunctata</i> | Stonefly      | Perlodidae       | S         | 96 h         | 13        | Immobilization | NR            | Meas     | <b>0.0474</b> (0.0402-0.0559)       | Weston 2014       |
| <i>Lepomis macrochirus</i>      | Bluegill      | Centrarchidae    | FT        | 96 h         | 22        | Survival       | 1.5 g, 45 mm  | Meas     | <b>25</b> (21-30)                   | Bettencourt 1992a |
| <i>Nectopsyche sp.</i>          | Caddisfly     | Leptoceridae     | S         | 48 h         | 23        | Immobilization | NR            | Meas     | <b>0.0313</b> (0.0230-0.0401)       | Weston 2014       |
| <i>Oncorhynchus mykiss</i>      | Rainbow trout | Salmonidae       | FT        | 96 h         | 12        | Survival       | 0.55 g, 39 mm | Meas     | <b>39</b> (35-43)                   | Bettencourt 1992b |
| <i>Serratella micheneri</i>     | Mayfly        | Baetidae         | S         | 48 h         | 23        | Immobilization | NR            | Meas     | <b>0.159</b> (0.106-0.214)          | Weston 2014       |
| <i>Taenionema sp.</i>           | Stonefly      | Taeniopterygidae | S         | 96 h         | 8         | Survival       | NR            | Meas     | <b>0.0959</b> (0.0621-0.126)        | Weston 2014       |

Table 6 Final acute toxicity data used to calculate fipronil-desulfinyl WQC.

All studies were rated relevant and reliable (RR).

| Species                    | Common name   | Family        | Test type | Duration (d) | Temp (°C) | Endpoint | Age/ size     | Nom/ Meas | LC/EC <sub>50</sub> (95% CI) (µg/L) | Reference     |
|----------------------------|---------------|---------------|-----------|--------------|-----------|----------|---------------|-----------|-------------------------------------|---------------|
| <i>Lepomis macrochirus</i> | Bluegill      | Centrarchidae | SR        | 96 h         | 22        | Survival | 0.51 g, 32 mm | Meas      | 20 (17-25)                          | Collins 1993a |
| <i>Oncorhynchus mykiss</i> | Rainbow trout | Salmonidae    | SR        | 96 h         | 12        | Survival | 0.85 g, 45 mm | Meas      | 31 (17-42)                          | Collins 1993b |

Table 7 Final aquatic acute toxicity data for fipronil-carboxamide

All studies were rated relevant and reliable (RR).

| Species                    | Common name | Family       | Test type | Duration (d) | Temp (°C) | Endpoint | Age/size   | Nom/Meas | LC/EC <sub>50</sub> (95% CI) (µg/L) | Reference |
|----------------------------|-------------|--------------|-----------|--------------|-----------|----------|------------|----------|-------------------------------------|-----------|
| <i>Chironomus riparius</i> | Chironomids | Chironomidae | S         | 48 h         | 21        | Survival | 1st instar | Meas     | <b>250</b> (100-630)                | Funk 2004 |

Table 8 Aqueous acute data for fipronil and degradates reduced from final data set.

All studies were rated relevant and reliable (RR).

Color key:

|          |                  |                  |                     |                                       |
|----------|------------------|------------------|---------------------|---------------------------------------|
| Fipronil | Fipronil-sulfide | Fipronil-sulfone | Fipronil-desulfinyl | Fipronil-destrifluoromethyl-sulfonate |
|----------|------------------|------------------|---------------------|---------------------------------------|

| Species                         | Common name   | Family          | Test type | Duration (d) | Temp (°C) | Endpoint       | Age/ size     | Nom/ Meas | LC/EC <sub>50</sub> (95% CI)(µg/L) | Reference      | Reason for reduction |
|---------------------------------|---------------|-----------------|-----------|--------------|-----------|----------------|---------------|-----------|------------------------------------|----------------|----------------------|
| <i>Baetis tricaudatus</i>       | Mayfly        | Baetidae        | S         | 48 h         | 17        | Immobilization | NR            | Meas      | 0.105 (0.076-0.146)                | Weston 2014    | 3                    |
| <i>Chironomus dilutus</i>       | Chironomids   | Chironomidae    | S         | 96 h         | 23        | Immobilization | NR            | Meas      | >0.0815                            | Weston 2014    | 1                    |
| <i>Daphnia magna</i>            | Daphnid       | Daphniidae      | FT        | 24 h         | 20        | Immobilization | <24 h         | Meas      | <280                               | McNamara 1990a | 1                    |
| <i>Diphetero hageni</i>         | Mayfly        | Baetidae        | S         | 24 h         | 18        | Immobilization | NR            | Meas      | 0.347 (0.196-0.568)                | Weston 2014    | 3                    |
| <i>Fallceon quilleri</i>        | Mayfly        | Baetidae        | S         | 48 h         | 23        | Immobilization | NR            | Meas      | >0.187                             | Weston 2014    | 1                    |
| <i>Helicopsyche sp.</i>         | Caddisfly     | Helicopsychidae | S         | 96 h         | 13        | Immobilization | NR            | Meas      | >842                               | Weston 2014    | 1                    |
| <i>Hexagenia sp.</i>            | Mayfly        | Ephemeroidea    | S         | 96 h         | 18        | Immobilization | NR            | Meas      | 1.231 (0.769-01.667)               | Weston 2014    | 3                    |
| <i>Hyaella azteca</i>           | Amphipod      | Hyalellidae     | S         | 96 h         | 23        | Survival       | NR            | Meas      | 1.593 (1.343-1.889)                | Weston 2014    | 3                    |
| <i>Hydropsyche sp.</i>          | Caddisfly     | Hydropsychidae  | S         | 96 h         | 12        | Immobilization | NR            | Meas      | 2.107 (1.218-2.668)                | Weston 2014    | 3                    |
| <i>Isoperla quinquepunctata</i> | Stonefly      | Perlodidae      | S         | 96 h         | 13        | Immobilization | NR            | Meas      | 0.113 (0.0942-0.135)               | Weston 2014    | 3                    |
| <i>Oncorhynchus mykiss</i>      | Rainbow trout | Salmonidae      | FT        | 72 h         | 12        | Survival       | 36 mm, 0.98 g | Meas      | 248 (160-∞)                        | Ward 1991b     | 2                    |
| <i>Nectopsyche sp.</i>          | Caddisfly     | Leptoceridae    | S         | 48 h         | 23        | Survival       | NR            | Meas      | >2.947                             | Weston 2014    | 1                    |
| <i>Serratella micheneri</i>     | Mayfly        | Baetidae        | S         | 48 h         | 23        | Immobilization | NR            | Meas      | >722                               | Weston 2014    | 1                    |

| Species                         | Common name | Family          | Test type | Duration (d) | Temp (°C) | Endpoint       | Age/ size | Nom/ Meas | LC/EC <sub>50</sub> (95% CI)(µg/L) | Reference      | Reason for reduction |
|---------------------------------|-------------|-----------------|-----------|--------------|-----------|----------------|-----------|-----------|------------------------------------|----------------|----------------------|
| <i>Baetis tricaudatus</i>       | Mayfly      | Baetidae        | S         | 48 h         | 12        | Survival       | NR        | Meas      | >0.717                             | Weston 2014    | 1                    |
| <i>Chironomus dilutus</i>       | Chironomids | Chironomidae    | S         | 96 h         | 23        | Survival       | NR        | Meas      | >0.0624                            | Weston 2014    | 1                    |
| <i>Chironomus dilutus</i>       | Chironomids | Chironomidae    | FT        | 24 h         | 20        | Immobilization | <24 h     | Meas      | 320 (210-950)                      | McNamara 1990b | 2                    |
| <i>Chironomus dilutus</i>       | Chironomids | Chironomidae    | FT        | 48 h         | 20        | Immobilization | <24 h     | Meas      | 100 (81-130)                       | McNamara 1990b | 2                    |
| <i>Daphnia magna</i>            | Daphnid     | Daphniidae      | FT        | 24 h         | 20        | Immobilization | <24 h     | Meas      | 320 (210-950)                      | McNamara 1990b | 2                    |
| <i>Helicopsyche sp.</i>         | Caddisfly   | Helicopsychidae | S         | 96 h         | 13        | Survival       | NR        | Meas      | >0.551                             | Weston 2014    | 1                    |
| <i>Hyalella azteca</i>          | Amphipod    | Hyalellidae     | S         | 96 h         | 23        | Survival       | NR        | Meas      | 1.356 (1.092-1.635)                | Weston 2014    | 3                    |
| <i>Isoperla quinquepunctata</i> | Stonefly    | Perlodidae      | S         | 96 h         | 13        | Survival       | NR        | Meas      | 0.0945 (0.0668-0.175)              | Weston 2014    | 3                    |
| <i>Nectopsyche sp.</i>          | Caddisfly   | Leptoceridae    | S         | 48 h         | 23        | Survival       | NR        | Meas      | 0.122 (0.0602-0.177)               | Weston 2014    | 3                    |
| <i>Baetis tricaudatus</i>       | Mayfly      | Baetidae        | S         | 48 h         | 12        | Survival       | NR        | Meas      | 0.535 (0.382-0.750)                | Weston 2014    | 3                    |
| <i>Chironomus dilutus</i>       | Chironomids | Chironomidae    | S         | 96 h         | 23        | Survival       | NR        | Meas      | >0.102                             | Weston 2014    | 1                    |
| <i>Daphnia magna</i>            | Daphnid     | Daphniidae      | FT        | 24 h         | 20        | Immobilization | <24 h     | Meas      | 110 (82-170)                       | McNamara 1990c | 2                    |
| <i>Dipheter hageni</i>          | Mayfly      | Baetidae        | S         | 48 h         | 18        | Survival       | NR        | Meas      | 0.330 (0.188-0.536)                | Weston 2014    | 3                    |
| <i>Fallceon quilleri</i>        | Mayfly      | Baetidae        | S         | 48 h         | 23        | Survival       | NR        | Meas      | >0.196                             | Weston 2014    | 1                    |
| <i>Helicopsyche sp.</i>         | Caddisfly   | Helicopsychidae | S         | 96 h         | 13        | Survival       | NR        | Meas      | >0.626                             | Weston 2014    | 1                    |
| <i>Hexagenia sp.</i>            | Mayfly      | Ephemeraidae    | S         | 96 h         | 18        | Survival       | NR        | Meas      | 0.257 (0.109-0.362)                | Weston 2014    | 3                    |
| <i>Hydropsyche sp.</i>          | Caddisfly   | Hydropsychidae  | S         | 96 h         | 12        | Survival       | NR        | Meas      | >824                               | Weston 2014    | 1                    |

| Species                         | Common name   | Family           | Test type | Duration (d) | Temp (°C) | Endpoint | Age/ size     | Nom/ Meas | LC/EC <sub>50</sub> (95% CI)(µg/L) | Reference         | Reason for reduction |
|---------------------------------|---------------|------------------|-----------|--------------|-----------|----------|---------------|-----------|------------------------------------|-------------------|----------------------|
| <i>Hyalella azteca</i>          | Amphipod      | Hyaellidae       | S         | 96 h         | 23        | Survival | NR            | Meas      | 0.426 (0.346-0.497)                | Weston 2014       | 3                    |
| <i>Isoperla quinquepunctata</i> | Stonefly      | Perlodidae       | S         | 96 h         | 13        | Survival | NR            | Meas      | 0.0500 (0.0431-0.0581)             | Weston 2014       | 3                    |
| <i>Lepomis macrochirus</i>      | Bluegill      | Centrarchidae    | FT        | 48 h         | 22        | Survival | 1.5 g, 45 mm  | Meas      | 38 (33-46)                         | Bettencourt 1992a | 2                    |
| <i>Lepomis macrochirus</i>      | Bluegill      | Centrarchidae    | FT        | 72 h         | 22        | Survival | 1.5 g, 45 mm  | Meas      | 30 (25-35)                         | Bettencourt 1992a | 2                    |
| <i>Lepomis macrochirus</i>      | Bluegill      | Centrarchidae    | FT        | 24 h         | 22        | Survival | 1.5 g, 45 mm  | Meas      | > 51                               | Bettencourt 1992a | 1                    |
| <i>Oncorhynchus mykiss</i>      | Rainbow trout | Salmonidae       | FT        | 24 h         | 12        | Survival | 0.55 g, 39 mm | Meas      | 59 (53-66)                         | Bettencourt 1992b | 2                    |
| <i>Oncorhynchus mykiss</i>      | Rainbow trout | Salmonidae       | FT        | 48 h         | 12        | Survival | 0.55 g, 39 mm | Meas      | 44 (39-50)                         | Bettencourt 1992b | 2                    |
| <i>Oncorhynchus mykiss</i>      | Rainbow trout | Salmonidae       | FT        | 72 h         | 12        | Survival | 0.55 g, 39 mm | Meas      | 40 (36-44)                         | Bettencourt 1992b | 2                    |
| <i>Nectopsyche</i> sp.          | Caddisfly     | Leptoceridae     | S         | 48 h         | 23        | Survival | NR            | Meas      | 0.0515 (0.0370-0.0691)             | Weston 2014       | 3                    |
| <i>Serratella micheneri</i>     | Mayfly        | Baetidae         | S         | 48 h         | 23        | Survival | NR            | Meas      | 0.331 (0.257-0.426)                | Weston 2014       | 3                    |
| <i>Taenionema</i> sp.           | Stonefly      | Taeniopterygidae | S         | 96 h         | 8         | Survival | NR            | Meas      | >0.261                             | Weston 2014       | 1                    |
| <i>Lepomis macrochirus</i>      | Bluegill      | Centrarchidae    | SR        | 24 h         | 22        | Survival | 0.51 g, 32 mm | Meas      | 32 (26-43)                         | Collins 1993a     | 2                    |
| <i>Lepomis macrochirus</i>      | Bluegill      | Centrarchidae    | SR        | 48 h         | 22        | Survival | 0.51 g, 32 mm | Meas      | 28 (16-43)                         | Collins 1993a     | 2                    |
| <i>Lepomis macrochirus</i>      | Bluegill      | Centrarchidae    | SR        | 72 h         | 22        | Survival | 0.51 g, 32 mm | Meas      | 22 (18-27)                         | Collins 1993a     | 2                    |

| Species                    | Common name   | Family     | Test type | Duration (d) | Temp (°C) | Endpoint       | Age/ size     | Nom/ Meas | LC/EC <sub>50</sub> (95% CI)(µg/L) | Reference     | Reason for reduction |
|----------------------------|---------------|------------|-----------|--------------|-----------|----------------|---------------|-----------|------------------------------------|---------------|----------------------|
| <i>Oncorhynchus mykiss</i> | Rainbow trout | Salmonidae | SR        | 24 h         | 12        | Survival       | 0.85 g, 45 mm | Meas      | 36 (28-42)                         | Collins 1993b | 2                    |
| <i>Oncorhynchus mykiss</i> | Rainbow trout | Salmonidae | SR        | 48 h         | 12        | Survival       | 0.85 g, 45 mm | Meas      | 34 (28-42)                         | Collins 1993b | 2                    |
| <i>Oncorhynchus mykiss</i> | Rainbow trout | Salmonidae | SR        | 72 h         | 12        | Survival       | 0.85 g, 45 mm | Meas      | 32 (17-42)                         | Collins 1993b | 2                    |
| <i>Daphnia magna</i>       | Daphnid       | Daphniidae | S         | 48 h         | 22        | Immobilization | <24 h         | Nom       | >100,000                           | Collins 1993  | 1                    |

1. Approximated toxicity value
2. More sensitive timepoint available
3. More sensitive endpoint available

Table 9 Final chronic toxicity data used to calculate fipronil WQC.

All studies were rated relevant and reliable (RR).

| Species                    | Common name   | Family     | Test type | Duration (d) | Temp (°C) | Endpoint     | Age/size | Nom/Meas | MATC (µg/L)                                | Reference          |
|----------------------------|---------------|------------|-----------|--------------|-----------|--------------|----------|----------|--|--------------------|
| <i>Ceriodaphnia dubia</i>  | Daphnid       | Daphniidae | S         | 8d           | 25        | Reproduction | <24 h    | Nom/Meas | <b>Racemate: 85</b><br>(+): 16<br>(-): 156 | Wilson et al. 2008 |
| <i>Daphnia magna</i>       | Daphnid       | Daphniidae | FT        | 21 d         | 20        | Growth       | <24 h    | Meas     | <b>14</b>                                  | McNamara 1990d     |
| <i>Oncorhynchus mykiss</i> | Rainbow trout | Salmonidae | FT        | 90 d         | 12        | Survival     | Eggs     | Meas     | <b>20</b>                                  | Machado 1992a      |

Table 10 Final chronic toxicity data used to calculate fipronil-sulfide WQC.

All studies were rated relevant and reliable (RR).

| Species              | Common name | Family     | Test type | Duration (d) | Temp (°C) | Endpoint                            | Age/size | Nom/Meas | MATC (µg/L) | Reference      |
|----------------------|-------------|------------|-----------|--------------|-----------|-------------------------------------|----------|----------|-------------|----------------|
| <i>Daphnia magna</i> | Daphnid     | Daphniidae | FT        | 21 d         | 20        | Growth, cumulative offspring/female | <24 h    | Meas     | 17          | McNamara 1990e |

Table 11 Final chronic toxicity data used to calculate fipronil-sulfone WQC.

All studies were rated relevant and reliable (RR).

| Species              | Common name | Family     | Test type | Duration (d) | Temp (°C) | Endpoint | Age/size | Nom/Meas | MATC (µg/L) | Reference     |
|----------------------|-------------|------------|-----------|--------------|-----------|----------|----------|----------|-------------|---------------|
| <i>Daphnia magna</i> | Daphnid     | Daphniidae | FT        | 21 d         | 21        | Length   | 14 d     | Meas     | 0.65        | Janson 2014   |
| <i>Daphnia magna</i> | Daphnid     | Daphniidae | FT        | 21 d         | 20        | Growth   | <24 h    | Meas     | 0.97        | McNamara 1992 |
| GEOMEAN              |             |            |           |              |           |          |          |          | <b>0.79</b> |               |

Table 12 Final chronic toxicity data used to calculate fipronil desulfinyl WQC.

All studies were rated relevant and reliable (RR).

| Species              | Common name | Family     | Test type | Duration (d) | Temp (°C) | Endpoint | Age/ size | Nom/ Meas | MATC (µg/L) | Reference  |
|----------------------|-------------|------------|-----------|--------------|-----------|----------|-----------|-----------|-------------|------------|
| <i>Daphnia magna</i> | Daphnid     | Daphniidae | SR        | 21 d         | 20        | Growth   | <24 h     | Meas      | <b>64</b>   | Putt 1992a |

Table 13 Aqueous chronic data for fipronil degradates reduced from final data set.

Color key: 

|          |                  |                     |
|----------|------------------|---------------------|
| Fipronil | Fipronil-sulfone | Fipronil-desulfinyl |
|----------|------------------|---------------------|

| Species              | Common name | Family     | Test type | Duration (d) | Temp (°C) | Endpoint             | Age/size | Nom / Meas | MATC (µg/L)      | Reference      | Reason for reduction |
|----------------------|-------------|------------|-----------|--------------|-----------|----------------------|----------|------------|------------------|----------------|----------------------|
| <i>Daphnia magna</i> | Daphnid     | Daphniidae | FT        | 1 d          | 20        | Growth               | <24 h    | Meas       | EC50: >79        | McNamara 1990d | 2                    |
| <i>Daphnia magna</i> | Daphnid     | Daphniidae | FT        | 2 d          | 20        | Growth               | <24 h    | Meas       | EC50: >79        | McNamara 1990d | 2                    |
| <i>Daphnia magna</i> | Daphnid     | Daphniidae | FT        | 4 d          | 20        | Growth               | <24 h    | Meas       | EC50: 61 (34-79) | McNamara 1990d | 2                    |
| <i>Daphnia magna</i> | Daphnid     | Daphniidae | FT        | 7 d          | 20        | Growth               | <24 h    | Meas       | EC50: 53 (34-79) | McNamara 1990d | 2                    |
| <i>Daphnia magna</i> | Daphnid     | Daphniidae | FT        | 14 d         | 20        | Growth               | <24 h    | Meas       | EC50: 41 (34-79) | McNamara 1990d | 2                    |
| <i>Daphnia magna</i> | Daphnid     | Daphniidae | FT        | 21 d         | 20        | Growth               | <24 h    | Meas       | EC50: 39 (34-79) | McNamara 1990d | 2                    |
| <i>Daphnia magna</i> | Daphnid     | Daphniidae | FT        | 21 d         | 21        | Survival             | 14 d     | Meas       | 2.63             | Janson 2014    | 2                    |
| <i>Daphnia magna</i> | Daphnid     | Daphniidae | FT        | 21 d         | 21        | Offspring per female | 14 d     | Meas       | 1.31             | Janson 2014    | 2                    |
| <i>Daphnia magna</i> | Daphnid     | Daphniidae | FT        | 21 d         | 21        | Age at first brood   | 14 d     | Meas       | 2.63             | Janson 2014    | 2                    |
| <i>Daphnia magna</i> | Daphnid     | Daphniidae | FT        | 21 d         | 21        | Weight               | 14 d     | Meas       | 2.63             | Janson 2014    | 2                    |
| <i>Daphnia magna</i> | Daphnid     | Daphniidae | FT        | 21 d         | 21        | Growth rate          | 14 d     | Meas       | 1.31             | Janson 2014    | 2                    |
| <i>Daphnia magna</i> | Daphnid     | Daphniidae | SR        | 1 d          | 20        | Growth               | <24 h    | Meas       | EC50: >260       | Putt 1992a     | 2                    |
| <i>Daphnia magna</i> | Daphnid     | Daphniidae | SR        | 2 d          | 20        | Growth               | <24 h    | Meas       | EC50: >260       | Putt 1992a     | 2                    |
| <i>Daphnia magna</i> | Daphnid     | Daphniidae | SR        | 4 d          | 20        | Growth               | <24 h    | Meas       | EC50: >260       | Putt 1992a     | 2                    |
| <i>Daphnia magna</i> | Daphnid     | Daphniidae | SR        | 7 d          | 20        | Growth               | <24 h    | Meas       | EC50: >260       | Putt 1992a     | 2                    |
| <i>Daphnia magna</i> | Daphnid     | Daphniidae | SR        | 14 d         | 20        | Growth               | <24 h    | Meas       | EC50: >260       | Putt 1992a     | 2                    |

| Species                         | Common name | Family         | Test type | Duration (d) | Temp (°C) | Endpoint     | Age/size    | Nom / | MATC                | Reference    | Reason for reduction |
|---------------------------------|-------------|----------------|-----------|--------------|-----------|--------------|-------------|-------|---------------------|--------------|----------------------|
| <i>Daphnia magna</i>            | Daphnid     | Daphniidae     | SR        | 21 d         | 20        | Growth       | <24 h       | Meas  | EC50: 230 (100-260) | Putt 1992a   | 2                    |
| <i>Raphidocelis subcapitata</i> | Alga        | Selenastraceae | S         | 5 d          | 24        | Cell density | Algal cells | Meas  | LOEC 12             | Hoberg 1993a | 1                    |

1. MATC not calculable
2. More sensitive endpoint available

Table 14 Supplemental studies for fipronil and degradates WQC derivation.

Color key:

|          |                  |                  |                     |
|----------|------------------|------------------|---------------------|
| Fipronil | Fipronil-sulfide | Fipronil-sulfone | Fipronil-desulfinyl |
|----------|------------------|------------------|---------------------|

| Species                          | Common name | Test Type | Duration (d) | Temp (°C) | Endpoint       | Age/ size  | Nom/ Meas | LC/ EC <sub>50</sub> (95% CI) (µg/L)  | MATC (µg/L) | Ref                 | Excl. |
|----------------------------------|-------------|-----------|--------------|-----------|----------------|------------|-----------|---|-------------|---------------------|-------|
| <i>Aedes aegypti</i>             | Mosquito    | S         | 24 h         | NR        | Survival       | 4th instar | NR        | 0.0108  | NR          | Chaton 2001         | 2     |
| <i>Aedes aegypti</i>             | Mosquito    | S         | 48 h         | NR        | Survival       | 4th instar | NR        | 0.066   | NR          | Chaton 2001         | 2     |
| <i>Aedes albopictus</i>          | Mosquito    | S         | 48 h         | 26        | Survival       | 1st instar | NR        | 0.0081 (0.0071-0.0090)  | NR          | Ali 1998            | 1     |
| <i>Aedes albopictus</i>          | Mosquito    | S         | 48 h         | 26        | Survival       | 4th instar | NR        | 0.023 (0.015-0.032)   | NR          | Ali 1998            | 1     |
| <i>Aedes taeniorhynchus</i>      | Mosquito    | S         | 24 h         | 26        | Survival       | 4th instar | NR        | 0.0014 (0.00119-0.00163)  | NR          | Ali 1998            | 1     |
| <i>Aedes taeniorhynchus</i>      | Mosquito    | S         | 24 h         | 26        | Survival       | 4th instar | NR        | 0.00043 (0.00034-0.00050)   | NR          | Ali 1998            | 1     |
| <i>Americamysis bahia</i>        | Mysid       | FT        | 28 d         | 25        | Survival       | <24 h      | Meas      | NR  | 0.011       | Machado 1995        | 5     |
| <i>Americamysis bahia</i>        | Mysid       | S         | 72 h         | 25        | Survival       | <24 h      | Meas      | 0.170 (0.140-0.240)   | NR          | Machado 1994        | 5     |
| <i>Americamysis bahia</i>        | Mysid       | S         | 96 h         | 25        | Survival       | <24 h      | Meas      | 0.140 (0.120-0.160)   | 0.078       | Machado 1994        | 5     |
| <i>Anopheles quadrimaculatus</i> | Mosquito    | S         | 48 h         | 26        | Survival       | 4th instar | NR        | 0.00043 (0.00009-0.00081)   | NR          | Ali 1998            | 1     |
| <i>Ceriodaphnia dubia</i>        | Daphnid     | S         | 48 h         | 25        | Immobilization | <24 h      | Nom       | Light:<br>Racemate:<br>17.9 ± 2.7<br>(+): 11.3 ± 2.0<br>(-): 35.4 ± 2.6<br>Dark:<br>Racemate:<br>17.5 ± 0.7 | NR          | Konwick et al. 2005 | 2     |

| Species                          | Common name       | Test Type | Duration (d)    | Temp (°C) | Endpoint       | Age/ size           | Nom/ Meas | LC/ EC <sub>50</sub> (95% CI) (µg/L) | MATC (µg/L) | Ref                  | Excl. |
|----------------------------------|-------------------|-----------|-----------------|-----------|----------------|---------------------|-----------|--------------------------------------|-------------|----------------------|-------|
|                                  |                   |           |                 |           |                |                     |           | (+): 9.4 ± 0.7<br>(-): 28.4 ± 2.4    |             |                      |       |
| <i>Chironomus crassicaudatus</i> | Midge             | S         | 48 h            | 26        | Survival       | 1st instar          | NR        | 0.0046<br>(0.00004-0.0087)           | NR          | Ali 1998             | 1, 2  |
| <i>Chironomus crassicaudatus</i> | Midge             | S         | 48 h            | 26        | Survival       | 4th instar          | NR        | 0.0073<br>(0.0069-0.0077)            | NR          | Ali 1998             | 1, 2  |
| <i>Corbicula fluminea</i>        | Clam              | SR        | 96 h            | 20        | Survival       | 12.4 mm shell width | Meas      | >2000                                | NR          | Putt 2003b           | 4     |
| <i>Cricotopus lebetis</i>        | Midge             | S         | 24 h            | 25        | Survival       | 8 d                 | NR        | 7.26 (4.92–10.89)                    | NR          | Stratman 2013        | 1, 3  |
| <i>Cricotopus lebetis</i>        | Midge             | S         | 48 h            | 25        | Survival       | 8 d                 | NR        | 2.61 (1.78–3.55)                     | NR          | Stratman 2013        | 1, 3  |
| <i>Cricotopus lebetis</i>        | Midge             | S         | 72 h            | 25        | Survival       | 8 d                 | NR        | 1.78 (1.18–2.47)                     | NR          | Stratman 2013        | 1, 3  |
| <i>Cricotopus lebetis</i>        | Midge             | S         | 96 h            | 25        | Survival       | 8 d                 | NR        | 1.06 (0.6–1.57)                      | NR          | Stratman 2013        | 1, 3  |
| <i>Cyprinodon variegatus</i>     | Sheepshead minnow | FT        | 110 d           | 28        | Length         | <26 h embryos       | Meas      | NR                                   | 8.8         | Dionne 2000          | 5     |
| <i>Cyprinodon variegatus</i>     | Sheepshead minnow | FT        | 28 d post hatch | 28        | Length         | <26 h embryos       | Meas      | NR                                   | 8.8         | Dionne 2000          | 5     |
| <i>Cyprinodon variegatus</i>     | Sheepshead minnow | FT        | 28 d post hatch | 25        | Weight         | <30 h embryos       | Meas      | NR                                   | LOEC 1.6    | Sousa 1998a          | 4, 5  |
| <i>Cyprinodon variegatus</i>     | Sheepshead minnow | FT        | 35 d            | 25        | Weight         | <23 h embryos       | Meas      | NR                                   | NOEC 2.9    | Sousa 1998a          | 4, 5  |
| <i>Cyprinodon variegatus</i>     | Sheepshead minnow | FT        | 24 h            | 22        | Survival       | 0.29 g 26 mm        | Meas      | 300 (240-340)                        | NR          | Machado 1993         | 5     |
| <i>Cyprinodon variegatus</i>     | Sheepshead minnow | FT        | 48 h            | 22        | Survival       | 0.29 g 26 mm        | Meas      | 180 (150-200)                        | NR          | Machado 1993         | 5     |
| <i>Cyprinodon variegatus</i>     | Sheepshead minnow | FT        | 72 h            | 22        | Survival       | 0.29 g 26 mm        | Meas      | 180 (150-200)                        | NR          | Machado 1993         | 5     |
| <i>Cyprinodon variegatus</i>     | Sheepshead minnow | FT        | 96 h            | 22        | Survival       | 0.29 g 26 mm        | Meas      | 130 (110-150)                        | NR          | Machado 1993         | 5     |
| <i>Daphnia magna</i>             | Daphnid           | S         | 48 h            | 21        | Immobilization | 1st instar          | Meas      | 42.9 (35.9-51.7)                     | NR          | Iwafune 2011         | 1, 3  |
| <i>Dunaliella tertiolecta</i>    | Alga              | S         | 96 h            | 25        | Cell density   | Algal cells         | Nom       | 631.2                                | 354         | Overmyer et al. 2007 | 5     |

| Species                       | Common name | Test Type | Duration (d) | Temp (°C) | Endpoint | Age/ size   | Nom/ Meas | LC/ EC <sub>50</sub> (95% CI) (µg/L)  | MATC (µg/L) | Ref                  | Excl. |
|-------------------------------|-------------|-----------|--------------|-----------|----------|-------------|-----------|---|-------------|----------------------|-------|
| <i>Elliptio complanata</i>    | Mussel      | SR        | 96 h         | 21        | Survival | Glochidia   | Meas      | >2000   | NR          | Bringolf 2007        | 4     |
| <i>Ephemerella excrucians</i> | Mayfly      | S         | 48 h         | 13        | Survival | NR          | Meas      | >0.436  | NR          | Weston 2014          | 3, 5  |
| <i>Glyptotendipes paripes</i> | Midge       | S         | 24 h         | 26        | Survival | 4th instar  | NR        | 0.00091<br>(0.00055-0.00055-0.00141)  | NR          | Ali 1998             | 1, 2  |
| <i>Glyptotendipes paripes</i> | Midge       | S         | 48 h         | 26        | Survival | 4th instar  | NR        | 0.00042<br>(0.00016-0.00080)  | NR          | Ali 1998             | 1, 2  |
| <i>Lampsilis fasciola</i>     | Mussel      | SR        | 96 h         | 21        | Survival | <2 m        | Meas      | >2000   | NR          | Bringolf 2007        | 4     |
| <i>Lampsilis fasciola</i>     | Mussel      | SR        | 96 h         | 21        | Survival | Glochidia   | Meas      | >2000   | NR          | Bringolf 2007        | 4     |
| <i>Lampsilis siliquoidea</i>  | Clam        | SR        | 96 h         | 21        | Survival | <2 m        | Meas      | >2000   | NR          | Bringolf 2007        | 4     |
| <i>Lampsilis siliquoidea</i>  | Clam        | SR        | 96 h         | 21        | Survival | Glochidia   | Meas      | >2000   | NR          | Bringolf 2007        | 4     |
| <i>Lumbriculus variegatus</i> | Blackworm   | SR        | 96 h         | 23        | Survival | 0.0039 g    | Meas      | >1900   | NR          | Putt 2003c           | 4     |
| <i>Mercenaria mercenaria</i>  | Clam        | S         | 96 h         | 25        | Survival | 212-350 µm  | Nom       | Racemate:<br>177.00<br>(46.00-674.00)<br>(+): 208 (137-318)<br>(-): 187.00<br>(124.00-281.00)           | NR          | Overmyer et al. 2007 | 5     |
| <i>Procambarus clarkii</i>    | Crayfish    | S         | 96 h         | 20        | Survival | 7.1-10.5 cm | Nom       | Racemate:<br>124.89<br>(87.20-179.24)<br>(+): 81.70<br>(62.90-106.10)<br>(-): 163.50<br>(124.37-214.94) | NR          | Overmyer et al. 2007 | 2     |

| Species                       | Common name    | Test Type | Duration (d) | Temp (°C) | Endpoint   | Age/ size     | Nom/ Meas | LC/ EC <sub>50</sub> (95% CI) (µg/L)   | MATC (µg/L) | Ref                  | Excl. |
|-------------------------------|----------------|-----------|--------------|-----------|------------|---------------|-----------|--|-------------|----------------------|-------|
| <i>Procambarus clarkii</i>    | Crayfish       | S         | 96 h         | 25        | Survival   | 6-9 cm        | Meas      | 14.3 (9.1)   | NR          | Schlenk 2001         | 1     |
| <i>Palaemonetes pugio</i>     | Shrimp         | S         | 96 h         | 25        | Survival   | Adults        | Nom       | Racemate:<br>0.32 (0.24-0.41)<br>(+): 0.54 (0.45-0.64)<br>(-): 0.32 (0.22-0.48)      | NR          | Overmyer et al. 2007 | 5     |
| <i>Palaemonetes pugio</i>     | Shrimp         | S         | 96 h         | 25        | Survival   | Larvae, 1-2 d | Nom       | Racemate:<br>0.68 (0.57-0.80)<br>(+): 208.0 (137.00-318.00)<br>(-): 0.35 (0.29-0.43) | NR          | Overmyer et al. 2007 | 5     |
| <i>Pimephales promelas</i>    | Fathead minnow | S         | 24 h         | 23        | Survival   | 7 d           | NR        | 398.29 (376.27-438.79)   | 324         | Beggel 2010          | 1, 3  |
| <i>Procambarus zonangulus</i> | Crayfish       | S         | 96 h         | 25        | Survival   | 6-9 cm        | Meas      | 19.5 (8.4)   | NR          | Schlenk 2001         | 1     |
| <i>Scenedesmus obliquus</i>   | Alga           | S         | 72 h         | 25        | Cell count | Algal cells   | Nom       | Racemate:<br>540 (270-1120)<br>(+): 1500 (810-2240)<br>(-): 290 (220-370)            | NR          | Qu 2014              | 1     |
| <i>Simulium vittatum</i>      | Black flies    | S         | 48 h         | 20        | Survival   | 5th instar    | Meas      | Racemate:<br>0.65 (0.60-0.70)<br>(+): 0.72 (0.66-0.78)<br>(-): 0.74 (0.69-0.81)      | NR          | Overmyer et al. 2007 | 2     |
| <i>Taenionema</i> sp.         | Stone fly      | S         | 96 h         | 8         | Survival   | NR            | Meas      | >0.184   | NR          | Weston 2014          | 4     |
| <i>Tricorythodes</i> sp.      | Mayfly         | S         | 48 h         | 18        | Survival   | NR            | Meas      | >1.229   | NR          | Weston 2014          | 4     |

| Species                    | Common name   | Test Type | Duration (d) | Temp (°C) | Endpoint       | Age/ size     | Nom/ Meas | LC/ EC <sub>50</sub> (95% CI) (µg/L)  | MATC (µg/L) | Ref                  | Excl. |
|----------------------------|---------------|-----------|--------------|-----------|----------------|---------------|-----------|---|-------------|----------------------|-------|
| <i>Villosa constricta</i>  | Mussel        | SR        | 48 h         | 21        | Survival       | Glochidia     | Meas      | >2000   | NR          | Bringolf 2007        | 4     |
| <i>Xenopus laevis</i>      | Frog          | S         | 96 h         | 25        | Survival       | Tadpoles      | Nom       | Racemate:<br>850 (660-1090)<br>(+): 910 (650-1280)<br>(-): 163.50 (124.37-214.94) | NR          | Overmyer et al. 2007 | 2     |
| <i>Americamysis bahia</i>  | Mysid         | S         | 96 h         | 25        | Survival       | NR            | Meas      | 0.077 (0.030-0.120)   | 0.047       | Putt 2000a           | 5     |
| <i>Daphnia magna</i>       | Daphnid       | S         | 48 h         | 21        | Immobilization | 1st instar    | Meas      | 28.0 (22.6-33.8)  | NR          | Iwafune 2011         | 1, 3  |
| <i>Procambarus clarkii</i> | Crayfish      | S         | 96 h         | 25        | Survival       | 6-9 cm        | Meas      | 15.5 (2.5)  | NR          | Schlenk 2001         | 1     |
| <i>Americamysis bahia</i>  | Mysid         | S         | 96 h         | 25        | Survival       | NR            | Meas      | 0.056 (0.031-0.120)   | 0.042       | Putt 2000b           | 5     |
| <i>Americamysis bahia</i>  | Mysid         | FT        | 28 d         | 27        | Weight         | <24 h         | Meas      | NR  | 0.0069      | Lima 2000            | 5     |
| <i>Daphnia magna</i>       | Daphnid       | S         | 48 h         | 21        | Immobilization | 1st instar    | Meas      | 5.17 (2.45-3.28)  | NR          | Iwafune 2011         | 1, 3  |
| <i>Procambarus clarkii</i> | Crayfish      | S         | 96 h         | 25        | Survival       | 6-9 cm        | Meas      | 11.2 (2.0)  | NR          | Schlenk 2001         | 1     |
| <i>Oncorhynchus mykiss</i> | Rainbow trout | SR        | 96 h         | 12        | Survival       | 0.85 g, 45 mm | Nom       | >100,000  | NR          | Collins 1993b        | 4     |
| <i>Americamysis bahia</i>  | Mysid         | S         | 96 h         | 25        | Survival       | NR            | Meas      | 0.1500 (0.0660-0.2500)  | 0.0926      | Putt 2000c           | 5     |
| <i>Procambarus clarkii</i> | Crayfish      | S         | 96 h         | 25        | Survival       | 6-9 cm        | Meas      | 68.6 (26.6)   | NR          | Schlenk 2001         | 1     |
| <i>Ceriodaphnia dubia</i>  | Daphnid       | S         | 48 h         | 25        | Immobilization | <24 h         | Nom       | 355 ± 9.3   | NR          | Konwick et al. 2005  | 2     |

1. Control not described and/or response not acceptable
2. Low reliability score
3. No standard method cited
4. Toxicity value not calculable
5. Seawater

Table 15 Final acute sediment toxicity data for fipronil.

All studies were rated relevant and reliable (RR).

All studies were rated relevant and reliable (RR).

| <b>Species</b>            | <b>Common name</b> | <b>Family</b> | <b>Duration (d)</b> | <b>Temp (°C)</b> | <b>Endpoint</b> | <b>Age/size</b> | <b>LC/EC<sub>50</sub> (95% CI) (µg/g OC)</b> | <b>% OC</b> | <b>Reference</b> |
|---------------------------|--------------------|---------------|---------------------|------------------|-----------------|-----------------|--|-------------|------------------|
| <i>Chironomus dilutus</i> | Chironomids        | Chironomidae  | 10 d                | 23               | Immobilization  | 4th instar      | <b>0.10</b> (0.08-0.11)                      | 0.69        | Maul 2008        |
| <i>Hyalella azteca</i>    | Amphipod           | Hyalellidae   | 10 d                | 23               | Survival        | 10 d            | <b>13.33</b> (11.48-15.19)                   | 2.7         | Picard 2015h     |

Table 16 Final acute sediment toxicity data for fipronil-sulfide.

All studies were rated relevant and reliable (RR).

| <b>Species</b>            | <b>Common name</b> | <b>Family</b> | <b>Duration (d)</b> | <b>Temp (°C)</b> | <b>Endpoint</b> | <b>Age/ size</b> | <b>LC/EC<sub>50</sub> (95% CI) (µg/g OC)</b> | <b>% OC</b> | <b>Reference</b> |
|---------------------------|--------------------|---------------|---------------------|------------------|-----------------|------------------|--|-------------|------------------|
| <i>Chironomus dilutus</i> | Chironomids        | Chironomidae  | 10 d                | 23               | Immobilization  | 4th instar       | <b>0.06</b> (0.03-0.07)                      | 0.69        | Maul 2008        |
| <i>Hyalella azteca</i>    | Amphipod           | Hyalellidae   | 10 d                | 23               | Survival        | 8 d              | <b>56</b> (48-63)                            | 2.7         | Picard 2015a     |

Table 17 Final acute sediment toxicity data for fipronil-sulfone.

All studies were rated relevant and reliable (RR).

| <b>Species</b>            | <b>Common name</b> | <b>Family</b> | <b>Duration<br/>(d)</b> | <b>Temp (°C)</b> | <b>Endpoint</b> | <b>Age/ size</b> | <b>LC/EC<sub>50</sub><br/>(95% CI)<br/>(µg/g OC)</b> | <b>% OC</b> | <b>Reference</b> |
|---------------------------|--------------------|---------------|-------------------------|------------------|-----------------|------------------|--|-------------|------------------|
| <i>Chironomus dilutus</i> | Chironomids        | Chironomidae  | 10 d                    | 23               | Growth          | 4th instar       | 0.04 (CI not reported)                               | 0.69        | Maul 2008        |
| <i>Hyalella azteca</i>    | Amphipod           | Hyalellidae   | 10 d                    | 23               | Survival        | 8 d              | <b>10</b> (10-11)                                    | 2.7         | Picard 2015b     |

Table 18 Final acute sediment toxicity data for fipronil-desulfinyl.

All studies were rated relevant and reliable (RR).

| Species                   | Common name | Family       | Duration (d) | Temp (°C) | Endpoint | Age/ size  | Nom/ Meas | LC/EC <sub>50</sub> (95% CI) (µg/g OC) | % OC | Reference    |
|---------------------------|-------------|--------------|--------------|-----------|----------|------------|-----------|--|------|--------------|
| <i>Chironomus dilutus</i> | Chironomids | Chironomidae | 10 d         | 22        | Growth   | 3rd instar | Meas      | <b>28</b> (24-31)                      | 2.3  | Putt 2001    |
| <i>Hyalella azteca</i>    | Amphipod    | Hyalellidae  | 10 d         | 23        | Survival | 8 d        | Meas      | <b>181</b> (167-200)                   | 2.7  | Picard 2015c |

Table 19 Reduced acute sediment toxicity data. All studies were rated relevant and reliable (RR).

| Species                   | Common name | Family       | Duration (d) | Temp (°C) | Endpoint | Age/size   | LC/EC <sub>50</sub> (95% CI) (µg/g OC) | % OC | Reference  | Reason for Reduction |
|---------------------------|-------------|--------------|--------------|-----------|----------|------------|--|------|------------|----------------------|
| <i>Chironomus dilutus</i> | Chironomids | Chironomidae | 10 d         | 23        | Survival | 4th instar | <b>0.13</b><br>(0.14-0.12)             | 0.69 | Maul 2008  | 1                    |
| <i>Chironomus dilutus</i> | Chironomids | Chironomidae | 10 d         | 23        | Growth   | 4th instar | MATC:<br>0.17                          | 0.69 | Maul 2008  | 2                    |
| <i>Chironomus dilutus</i> | Chironomids | Chironomidae | 10 d         | 23        | Survival | 3rd instar | MATC:<br>0.82                          | 2.8  | Putt 2003d | 2                    |
| <i>Chironomus dilutus</i> | Chironomids | Chironomidae | 10 d         | 23        | Survival | 3rd instar | 1.1                                    | 2.8  | Putt 2003d | 1                    |
| <i>Chironomus dilutus</i> | Chironomids | Chironomidae | 10 d         | 23        | Growth   | 3rd instar | 1.8                                    | 2.8  | Putt 2003d | 1                    |
| <i>Chironomus dilutus</i> | Chironomids | Chironomidae | 10 d         | 23        | Growth   | 3rd instar | MATC:<br>1.7                           | 2.8  | Putt 2003d | 2                    |
| <i>Chironomus dilutus</i> | Chironomids | Chironomidae | 10 d         | 23        | Survival | 4th instar | <b>0.16</b><br>(0.23-0.12)             | 0.69 | Maul 2008  | 1                    |
| <i>Chironomus dilutus</i> | Chironomids | Chironomidae | 10 d         | 23        | Growth   | 4th instar | MATC:<br>0.08                          | 0.69 | Maul 2008  | 2                    |
| <i>Chironomus dilutus</i> | Chironomids | Chironomidae | 10 d         | 23        | Survival | 3rd instar | 4.8                                    | 2.9  | Putt 2000d | 1                    |
| <i>Chironomus dilutus</i> | Chironomids | Chironomidae | 10 d         | 23        | Growth   | 3rd instar | 1.6                                    | 2.9  | Putt 2000d | 1                    |
| <i>Chironomus dilutus</i> | Chironomids | Chironomidae | 10 d         | 23        | Survival | 3rd instar | MATC:<br>1.4                           | 2.9  | Putt 2000d | 2                    |

| Species                   | Common name | Family       | Duration (d) | Temp (°C) | Endpoint | Age/size   | LC/EC <sub>50</sub> (95% CI) (µg/g OC) | % OC | Reference    | Reason for Reduction |
|---------------------------|-------------|--------------|--------------|-----------|----------|------------|--|------|--------------|----------------------|
| <i>Hyalella azteca</i>    | Amphipod    | Hyalellidae  | 10 d         | 23        | Survival | 8 d        | MATC: 40                               | 2.7  | Picard 2015a | 2                    |
| <i>Hyalella azteca</i>    | Amphipod    | Hyalellidae  | 10 d         | 23        | Growth   | 8 d        | MATC: 19                               | 2.7  | Picard 2015a | 2                    |
| <i>Chironomus dilutus</i> | Chironomids | Chironomidae | 10 d         | 23        | Survival | 4th instar | 0.12 (0.14-0.10)                       | 0.69 | Maul 2008    | 1                    |
| <i>Chironomus dilutus</i> | Chironomids | Chironomidae | 10 d         | 23        | Growth   | 4th instar | MATC: 0.08                             | 0.69 | Maul 2008    | 2                    |
| <i>Chironomus dilutus</i> | Chironomids | Chironomidae | 10 d         | 22        | Survival | 3rd instar | 1.5                                    | 0.69 | Putt 2000e   | 1                    |
| <i>Chironomus dilutus</i> | Chironomids | Chironomidae | 10 d         | 22        | Growth   | 3rd instar | 1.6                                    | 0.69 | Putt 2000e   | 1                    |
| <i>Chironomus dilutus</i> | Chironomids | Chironomidae | 10 d         | 22        | Survival | 3rd instar | MATC: 0.39                             | 0.69 | Putt 2000e   | 2                    |
| <i>Hyalella azteca</i>    | Amphipod    | Hyalellidae  | 10 d         | 23        | Survival | 8 d        | >13                                    | 2.7  | Picard 2015b | 1                    |
| <i>Hyalella azteca</i>    | Amphipod    | Hyalellidae  | 10 d         | 23        | Survival | 8 d        | MATC: 5                                | 2.7  | Picard 2015b | 2                    |
| <i>Chironomus dilutus</i> | Chironomids | Chironomidae | 10 d         | 22        | Survival | 3rd instar | 57 (31-100)                            | 2.3  | Putt 2001    | 1                    |
| <i>Chironomus dilutus</i> | Chironomids | Chironomidae | 10 d         | 22        | Growth   | 3rd instar | MATC: 7.78                             | 2.3  | Putt 2001    | 2                    |
| <i>Hyalella azteca</i>    | Amphipod    | Hyalellidae  | 10 d         | 23        | Growth   | 8 d        | >193                                   | 2.7  | Picard 2015c | 1                    |

| Species                | Common name | Family      | Duration (d) | Temp (°C) | Endpoint | Age/size | LC/EC <sub>50</sub><br>(95% CI)<br>(µg/g OC) | % OC | Reference       | Reason for Reduction |
|------------------------|-------------|-------------|--------------|-----------|----------|----------|--|------|-----------------|----------------------|
| <i>Hyalella azteca</i> | Amphipod    | Hyalellidae | 10 d         | 23        | Growth   | 8 d      | MATC:<br>69                                  | 2.7  | Picard<br>2015c | 2                    |
| <i>Hyalella azteca</i> | Amphipod    | Hyalellidae | 10 d         | 23        | Survival | 8 d      | MATC:<br>137                                 | 2.7  | Picard<br>2015c | 2                    |

1. More sensitive endpoint available.
2. Point estimate available (rather than MATC).

Table 20 Final chronic sediment toxicity data for fipronil-sulfide.

All studies were rated relevant and reliable (RR).

| <b>Species</b>             | <b>Common name</b> | <b>Family</b> | <b>Duration (d)</b> | <b>Temp (°C)</b> | <b>Endpoint</b>      | <b>Age/size</b> | <b>Nom/Meas</b> | <b>MATC (µg/g OC)</b> | <b>% OC</b> | <b>Reference</b> |
|----------------------------|--------------------|---------------|---------------------|------------------|----------------------|-----------------|-----------------|-----------------------|-------------|------------------|
| <i>Chironomus riparius</i> | Chironomids        | Chironomidae  | 28 d                | 20               | Cumulative emergence | 1st instar      | Nom             | <b>0.16</b>           | 2.18        | Kolk 2002        |

Table 20 Supplemental sediment toxicity values excluded from fipronil/degradates BSQC derivation

| Species                        | Common name | Family      | Duration (d) | Temp (°C) | Endpoint | Age/size | LC/EC <sub>50</sub> (95% CI) (µg/g OC) | % OC | MATC (µg/g OC) | Ref            | Excl. |
|--------------------------------|-------------|-------------|--------------|-----------|----------|----------|--|------|----------------|----------------|-------|
| <i>Leptocheirus plumulosus</i> | Amphipod    | Corophiidae | 10 d         | 25        | Survival | 2-4 mm   | 0.54 (0.49-0.56)                       | 3.9  | 0.42           | Picard 2015d   | 1     |
| <i>Mysidopsis bahia</i>        | Mysid       | Mysidae     | 28 d         | 25        | Survival | 21 d     | NR                                     | 2.7  | NOEC 0.06 µg/L | Cafarella 2005 | 1, 2  |
| <i>Leptocheirus plumulosus</i> | Amphipod    | Corophiidae | 10 d         | 23        | Survival | 2-4 mm   | 1.4 (1.3-1.4)                          | 3.9  | 1.3            | Picard 2015e   | 1     |
| <i>Leptocheirus plumulosus</i> | Amphipod    | Corophiidae | 10 d         | 25        | Survival | 2-4 mm   | 0.69 (0.69-0.72)                       | 3.9  | 0.71           | Picard 2015f   | 1     |
| <i>Leptocheirus plumulosus</i> | Amphipod    | Corophiidae | 10 d         | 24        | Survival | 2-4 mm   | 8.3 (7.6-8.6)                          | 3.9  | 5.3            | Picard 2015g   | 1     |

1. Saltwater
2. Toxicity value not based on bioavailability

Table 21 Threatened, endangered, or rare species predicted values by Web-ICE.

| Surrogate       |  |                            | Predicted            |                               |
|-----------------|--|----------------------------|----------------------|-------------------------------|
| Species         | Chemical                                     | LC <sub>50</sub><br>(µg/L) | Species              | LC <sub>50</sub> (µg/L)       |
| <i>O.mykiss</i> | Fipronil, acute                              | 248                        | <i>O.clarkii</i>     | 255.26 (203.60-320.03)        |
|                 |  |                            | <i>O.gilae</i>       | 205.14 (132.75-316.99)        |
|                 |  |                            | <i>O.kisutch</i>     | 356.67 (302.67-420.29)        |
|                 |  |                            | <i>O.nerka</i>       | 583.53 (208.32-1634.5)        |
|                 |  |                            | <i>O.tshawytscha</i> | 340.89 (241.83-480.54)        |
| <i>O.mykiss</i> | Fipronil, chronic                            | 20                         | <i>O.clarkii</i>     | 24.00 (17.74-32.47)           |
|                 |  |                            | <i>O.gilae</i>       | 12.57 (8.23-19.22)            |
|                 |  |                            | <i>O.kisutch</i>     | 29.98 (24.79-36.26)           |
|                 |  |                            | <i>O.nerka</i>       | Out of model range            |
|                 |  |                            | <i>O.tshawytscha</i> | 31.77 (19.33-52.22)           |
| <i>O.mykiss</i> | Fipronil-sulfone acute                       | 39                         | <i>O.clarkii</i>     | 44.94 (34.21-59.02)           |
|                 |  |                            | <i>O.gilae</i>       | 26.37 (18.12-38.38)           |
|                 |  |                            | <i>O.kisutch</i>     | 57.83 (48.47-68.99)           |
|                 |  |                            | <i>O.nerka</i>       | 153.18 (28.82-814.12)         |
|                 |  |                            | <i>O.tshawytscha</i> | 59.62 (38.09-93.34)           |
| <i>O.mykiss</i> | Fipronil-desulfinyl, acute                   | 31                         | <i>O.clarkii</i>     | 36.22 (27.32-48.04)           |
|                 |  |                            | <i>O.gilae</i>       | 20.44 (13.86-30.15)           |
|                 |  |                            | <i>O.kisutch</i>     | 46.14 (38.51-55.28)           |
|                 |  |                            | <i>O.nerka</i>       | 129.75 (22.25-756.40)         |
|                 |  |                            | <i>O.tshawytscha</i> | 48.02 (30.18-76.41)           |
| <i>O.mykiss</i> | Fipronil-destrifluoromethyl-sulfonate, acute | 100,000                    | <i>O.clarkii</i>     | 71353.48 (44785.76-113681.63) |
|                 |  |                            | <i>O.gilae</i>       | Out of model range            |
|                 |  |                            | <i>O.kisutch</i>     | Out of model range            |
|                 |  |                            | <i>O.nerka</i>       | Out of model range            |
|                 |  |                            | <i>O.tshawytscha</i> | 97331.23 (56437.28-167856.57) |

# **Appendix A – Aqueous Toxicity Data**

## **Summaries**

*Appendix A1 – Aqueous Toxicity Studies Rated RR*

## Water Toxicity Data Summary

*Baetis tricaudatus*

Fipronil

MB46030

Weston DP and Lydy MJ. (2014) Toxicity of the insecticide fipronil and its degradates to benthic macroinvertebrates of urban streams. *Environmental science & technology*, 48(2), 1290-1297.

Relevance

Score: 90

Rating: R

Reliability

Score: 85.5

Rating: R

Relevance points taken off for: Standard method (10). 100-10=90

| Fipronil                                     | Weston & Lydy 2014  | <i>B. tricaudatus</i> |
|--|---|-----------------------|
| Parameter                                    | Value   | Comment               |
| Test method cited                            | Not reported  |                       |
| Phylum/subphylum                             | Anthropoda  |                       |
| Class  | Insecta   |                       |
| Order  | Ephemeroptera   |                       |
| Family                                       | Baetidae  |                       |
| Genus  | <i>Baetis</i>   |                       |
| Species                                      | <i>tricaudatus</i>  |                       |
| Family native to North America?              | Yes   |                       |
| Age/size at start of test/growth phase       | Not reported  |                       |
| Source of organisms                          | Urban waterbodies with minimal development in Northern California |                       |
| Have organisms been exposed to contaminants? | Not reported  |                       |
| Animals acclimated and disease-free?         | 24 h  |                       |
| Animals randomized?                          | Not reported  |                       |
| Test vessels randomized?                     | Not reported  |                       |
| Test duration                                | 48 h  |                       |
| Data for multiple times?                     | Not reported  |                       |
| Effect 1                                     | Survival  |                       |
| Control response 1                           | 80 %  |                       |
| Effect 2                                     | Immobilization (ability to swim)                                  |                       |
| Control response 2                           | Not reported  |                       |
| Temperature                                  | 17 °C   |                       |
| Test type                                    | Static  |                       |
| Photoperiod/light intensity                  | 16l:8d; Not reported  |                       |

| Fipronil  | Weston & Lydy 2014   | <i>B. tricaudatus</i>                                   |
|---|--|---|
| Parameter   | Value  | Comment   |
| Dilution water  | Milli-Q purified, deionized watermade moderately hard by addition of salts | *According to EPA 821-R-02-012                          |
| pH  | Not reported*  |   |
| Hardness  | Not reported*  |   |
| Alkalinity  | Not reported*  |   |
| Conductivity  | Not reported*  |   |
| Dissolved Oxygen  | Not reported*  |   |
| Feeding   | Not reported   |   |
| Purity of test substance  | 99.50 %  | Not reported but author verified from chemical supplier |
| Concentrations measured?  | Yes  |   |
| Measured is what % of nominal?  | 66-113%  |   |
| Toxicity values calculated based on nominal or measured concentrations? | Measured   |   |
| Chemical method documented?   | GC   |   |
| Concentration of carrier (if any) in test solutions                     | Acetone, <36 µg/L  |   |
| Concentration 1 Nom; Meas (µg/L)  | Not reported; 4-7 concentrations tested; dilution factor of 2              | 3 reps, 4-6/rep   |
| Control   | Negative: 0; 0<br>Solvent: 0; 0  |   |
| LC <sub>50</sub> (95% CI) (µg/L)  | 0.0519 (0.0373-0.0720)   | Method: Probit  |
| EC <sub>50</sub> (95% CI) (µg/L)  | 0.105 (0.076-0.146)  | Method: Probit  |

Notes: Points were not deducted for lack of water quality parameter reporting because water was prepared according to EPA 821-R-02-012 and measured during study (but not reported).

Solubility (S) of fipronil = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Organism life stage/size (5), Nominal concentrations (3), Measured concentrations (3), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100-15 =85

Acceptability: Standard method (5), Organisms randomized (1), Temperature variation (3), Random design (2), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1). Total: 100-14 =86

**Reliability score: mean(85,86)=85.5**

## Water Toxicity Data Summary

*Baetis tricaudatus*  
Fipronil sulfide  
MB45950

Weston DP and Lydy MJ. (2014) Toxicity of the insecticide fipronil and its degradates to benthic macroinvertebrates of urban streams. *Environmental science & technology*, 48(2), 1290-1297.

Relevance  
Score: 90  
Rating: R

Reliability  
Score: 85.5  
Rating: R

Relevance points taken off for: Standard method (10). 100-10=90

| Fipronil sulfide                             | <b>Weston &amp; Lydy 2014</b>                                     | <i>B. tricaudatus</i> |
|--|---|-----------------------|
| <b>Parameter</b>                             | <b>Value</b>  | <b>Comment</b>        |
| Test method cited                            | Not reported  |                       |
| Phylum/subphylum                             | Anthropoda  |                       |
| Class  | Insecta   |                       |
| Order  | Ephemeroptera   |                       |
| Family                                       | Baetidae  |                       |
| Genus  | <i>Baetis</i>   |                       |
| Species                                      | <i>tricaudatus</i>  |                       |
| Family native to North America?              | Yes   |                       |
| Age/size at start of test/growth phase       | Not reported  |                       |
| Source of organisms                          | Urban waterbodies with minimal development in Northern California |                       |
| Have organisms been exposed to contaminants? | Not reported  |                       |
| Animals acclimated and disease-free?         | 24 h  |                       |
| Animals randomized?                          | Not reported  |                       |
| Test vessels randomized?                     | Not reported  |                       |
| Test duration                                | 48 h  |                       |
| Data for multiple times?                     | Not reported  |                       |
| Effect 1                                     | Survival  |                       |
| Control response 1                           | 87 %  |                       |
| Effect 2                                     | Immobilization (ability to swim)                                  |                       |
| Control response 2                           | Not reported  |                       |
| Temperature                                  | 12 °C   |                       |
| Test type                                    | Static  |                       |
| Photoperiod/light intensity                  | 16l:8d; Not reported  |                       |

| Fipronil sulfide  | Weston & Lydy 2014   | <i>B. tricaudatus</i>                                   |
|---|--|---|
| Parameter   | Value  | Comment   |
| Dilution water  | Milli-Q purified, deionized watermade moderately hard by addition of salts | *According to EPA 821-R-02-012                          |
| pH  | Not reported*  |   |
| Hardness  | Not reported*  |   |
| Alkalinity  | Not reported*  |   |
| Conductivity  | Not reported*  |   |
| Dissolved Oxygen  | Not reported*  |   |
| Feeding   | Not reported   |   |
| Purity of test substance  | 99.0 %   | Not reported but author verified from chemical supplier |
| Concentrations measured?  | Yes  |   |
| Measured is what % of nominal?  | 66-113%  |   |
| Toxicity values calculated based on nominal or measured concentrations? | Measured   |   |
| Chemical method documented?   | GC   |   |
| Concentration of carrier (if any) in test solutions                     | Acetone, <36 µg/L  |   |
| Concentration 1 Nom; Meas (µg/L)  | Not reported; 4-7 concentrations tested; dilution factor of 2              | 3 reps, 4-6/rep   |
| Control   | Negative: 0; 0<br>Solvent: 0; 0  |   |
| LC <sub>50</sub> (95% CI) (µg/L)  | >0.717   | Method: Probit  |
| EC <sub>50</sub> (95% CI) (µg/L)  | 0.0803 (0.0531-0.108)  | Method: Probit  |

Notes: Points were not deducted for lack of water quality parameter reporting because water was prepared according to EPA 821-R-02-012 and measured during study (but not reported).

Solubility value for fipronil sulfide (MB 45950) not available. Solubility (S) of fipronil parent compound (MB 46030) = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Organism life stage/size (5), Nominal concentrations (3), Measured concentrations (3), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100-15 =85

Acceptability: Standard method (5), Organisms randomized (1), Temperature variation (3), Random design (2), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1). Total: 100-14 =86

**Reliability score: mean(85,86)=85.5**

## Water Toxicity Data Summary

*Baetis tricaudatus*  
Fipronil sulfone  
MB46136

Weston DP and Lydy MJ. (2014) Toxicity of the insecticide fipronil and its degradates to benthic macroinvertebrates of urban streams. *Environmental science & technology*, 48(2), 1290-1297.

Relevance  
Score: 90  
Rating: R

Reliability  
Score: 85.5  
Rating: R

Relevance points taken off for: Standard method (10). 100-10=90

| Fipronil sulfone                             | <b>Weston &amp; Lydy 2014</b>                                     | <i>B. tricaudatus</i> |
|--|---|-----------------------|
| <b>Parameter</b>                             | <b>Value</b>  | <b>Comment</b>        |
| Test method cited                            | Not reported  |                       |
| Phylum/subphylum                             | Anthropoda  |                       |
| Class  | Insecta   |                       |
| Order  | Ephemeroptera   |                       |
| Family                                       | Baetidae  |                       |
| Genus  | <i>Baetis</i>   |                       |
| Species                                      | <i>tricaudatus</i>  |                       |
| Family native to North America?              | Yes   |                       |
| Age/size at start of test/growth phase       | Not reported  |                       |
| Source of organisms                          | Urban waterbodies with minimal development in Northern California |                       |
| Have organisms been exposed to contaminants? | Not reported  |                       |
| Animals acclimated and disease-free?         | 24 h  |                       |
| Animals randomized?                          | Not reported  |                       |
| Test vessels randomized?                     | Not reported  |                       |
| Test duration                                | 48 h  |                       |
| Data for multiple times?                     | Not reported  |                       |
| Effect 1                                     | Survival  |                       |
| Control response 1                           | 91 %  |                       |
| Effect 2                                     | Immobilization (ability to swim)                                  |                       |
| Control response 2                           | Not reported  |                       |
| Temperature                                  | 12 °C   |                       |
| Test type                                    | Static  |                       |
| Photoperiod/light intensity                  | 16l:8d; Not reported  |                       |

| Fipronil sulfone  | Weston & Lydy 2014   | <i>B. tricaudatus</i>                                   |
|---|--|---|
| Parameter   | Value  | Comment   |
| Dilution water  | Milli-Q purified, deionized watermade moderately hard by addition of salts | *According to EPA 821-R-02-012                          |
| pH  | Not reported*  |   |
| Hardness  | Not reported*  |   |
| Alkalinity  | Not reported*  |   |
| Conductivity  | Not reported*  |   |
| Dissolved Oxygen  | Not reported*  |   |
| Feeding   | Not reported   |   |
| Purity of test substance  | 99.3 %   | Not reported but author verified from chemical supplier |
| Concentrations measured?  | Yes  |   |
| Measured is what % of nominal?  | 66-113%  |   |
| Toxicity values calculated based on nominal or measured concentrations? | Measured   |   |
| Chemical method documented?   | GC   |   |
| Concentration of carrier (if any) in test solutions                     | Acetone, <36 µg/L  |   |
| Concentration 1 Nom; Meas (µg/L)  | Not reported; 4-7 concentrations tested; dilution factor of 2              | 3 reps, 4-6/rep   |
| Control   | Negative: 0; 0<br>Solvent: 0; 0  |   |
| LC <sub>50</sub> (95% CI) (µg/L)  | 0.535 (0.382-0.750)  | Method: Probit  |
| EC <sub>50</sub> (95% CI) (µg/L)  | 0.075 (0.0449-0.109)   | Method: Probit  |

Notes: Points were not deducted for lack of water quality parameter reporting because water was prepared according to EPA 821-R-02-012 and measured during study (but not reported).

Solubility (S) value for fipronil sulfone (MB 46136) = 160 µg/L, 2S = 320 µg/L.

Reliability points taken off for:

Documentation: Organism life stage/size (5), Nominal concentrations (3), Measured concentrations (3), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100-15 =85

Acceptability: Standard method (5), Organisms randomized (1), Temperature variation (3), Random design (2), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1). Total: 100-14 =86

**Reliability score: mean(85,86)=85.5**

## Water Toxicity Data Summary

*Ceriodaphnia dubia*

Fipronil

MB 46030

Wilson WA, Konwick BJ, Garrison AW, Avants JK and Black MC. (2008) Enantioselective chronic toxicity of fipronil to *Ceriodaphnia dubia*. *Archives of environmental contamination and toxicology*, 54(1), 36-43.

Relevance

Score: 100

Rating: R

Reliability

Score: 86.5

Rating: R

Relevance points taken off for: none.

| Fipronil                                     | Wilson et al. 2008  | <i>C. dubia</i> |
|--|---|-----------------|
| Parameter                                    | Value   | Comment         |
| Test method cited                            | USEPA 2002, Method #1002.0  |                 |
| Phylum/subphylum                             | Arthropoda  |                 |
| Class  | Branchiopoda  |                 |
| Order  | Cladocera   |                 |
| Family                                       | Daphniidae  |                 |
| Genus  | <i>Ceriodaphnia</i>   |                 |
| Species                                      | <i>dubia</i>  |                 |
| Family native to North America?              | Yes   |                 |
| Age/size at start of test/growth phase       | f <sub>1</sub> : <24 h<br>f <sub>2</sub> : <24 h  |                 |
| Source of organisms                          | Region IV Ecological Services Laboratory, Athens, Georgia                                 |                 |
| Have organisms been exposed to contaminants? | f <sub>1</sub> : No<br>f <sub>2</sub> : Yes   |                 |
| Animals acclimated and disease-free?         | Yes   |                 |
| Animals randomized?                          | Not reported  |                 |
| Test vessels randomized?                     | Yes   |                 |
| Test duration                                | f <sub>1</sub> : 8 d<br>f <sub>2</sub> : 48 h post-emergence                              |                 |
| Data for multiple times?                     | 24, 48 h  |                 |
| Effect 1                                     | f <sub>1</sub> Impaired movement  |                 |
| Control response 1                           | Qualitative endpoint; observations given in Table 2, but no effect levels were calculated |                 |

| Fipronil                            | Wilson et al. 2008  | <i>C. dubia</i> |
|-------------------------------------|---|-----------------|
| Parameter                           | Value   | Comment         |
| Effect 2                            | Mortality   |                 |
| Control response 2                  | 24 h: 5.3 (units not reported, likely %)<br>48 h: 11.8 (units not reported, likely %) |                 |
| Effect 3                            | f <sub>1</sub> Time to first brood (days)   |                 |
| Control response 3                  | Racemate: 4.1<br>(+): 4.1<br>(-): 4.1   |                 |
| Effect 4                            | f <sub>1</sub> No. of neonates/female   |                 |
| Control response 4                  | Racemate: 49.1<br>(+): 26.4<br>(-): 33.9  |                 |
| Effect 5                            | f <sub>1</sub> Average brood size   |                 |
| Control response 5                  | Racemate: 13.0<br>(+): 6.3<br>(-): 10.5   |                 |
| Effect 6                            | f <sub>1</sub> No. of broods/female   |                 |
| Control response 6                  | Racemate: 3.8<br>(+): 3.7<br>(-): 3.1   |                 |
| Effect 7                            | f <sub>1</sub> Adult survival time (days)   |                 |
| Control response 7                  | Racemate: 7.9<br>(+): 7.6<br>(-): 7.9   |                 |
| Temperature                         | 25 ± 1°C  |                 |
| Test type                           | Static  |                 |
| Photoperiod/light intensity         | 16l:8d  |                 |
| Dilution water                      | Moderately hard water   |                 |
| pH                                  | Not reported  |                 |
| Hardness                            | Not reported  |                 |
| Alkalinity                          | Not reported  |                 |
| Conductivity                        | Not reported  |                 |
| Dissolved Oxygen                    | >7.0 mg/L   |                 |
| Feeding                             | <i>P. subcapitata</i> and yeast, cereal leaves, and digested Tetramin1 mixture daily  |                 |
| Purity of test substance            | Racemate: 98 %<br>(+): Konwick 2005, 97.3 %<br>(-): Konwick 2005, 98.1 %              |                 |
| Concentrations measured?            | Not reported  |                 |
| Measured is what % of nominal?      | Not reported  |                 |
| Toxicity values calculated based on | Nominal   |                 |

| Fipronil  | Wilson et al. 2008   | <i>C. dubia</i>   |
|---|--|---|
| Parameter   | Value  | Comment   |
| nominal or measured concentrations?                 |  |   |
| Chemical method documented?                         | GCMS   |   |
| Concentration of carrier (if any) in test solutions | Acetone, 0.1 %   |   |
| Concentration 1 Nom; Meas (µg/L)                    | Racemate: 15; Not reported<br>(+): 2; Not reported<br>(-): 10; Not reported  | f <sub>1</sub> :10 reps, 1/rep<br>f <sub>2</sub> : 10 reps, various brood size/rep            |
| Concentration 2 Nom; Meas (µg/L)                    | Racemate: 30; Not reported<br>(+): 8; Not reported<br>(-): 30; Not reported  |   |
| Concentration 3 Nom; Meas (µg/L)                    | Racemate: 60; Not reported<br>(+): 32; Not reported<br>(-): 90; Not reported   |   |
| Concentration 4 Nom; Meas (µg/L)                    | Racemate: 120; Not reported<br>(+): 64; Not reported<br>(-): 270; Not reported   |   |
| Control   | Solvent<br>Negative  |   |
| LC <sub>50</sub> (standard error) (µg/L)            | f <sub>2</sub> 24 h<br>Racemate: 33.3 (3.1)<br>(+): 18.1 (4.7)<br>(-): 65.2 (15.8)<br>f <sub>2</sub> 48 h<br>Racemate: 30.3 (3.4)<br>(+): 10.3 (3.1)<br>(-): 50.1 (2.4)  | Method: Trimmed Spearman-Kärber   |
| NOEC  | f <sub>1</sub> <u>Time to first brood</u><br>Racemate: 60<br>(+): 64<br>(-): 30<br>f <sub>1</sub> <u>No. of neonates/female</u><br>Racemate: <15<br>(+): <2<br>(-): 10<br>f <sub>1</sub> <u>Average brood size</u><br>Racemate: <15<br>(+): <2<br>(-): 10<br>f <sub>1</sub> <u>No. of broods/female</u><br>Racemate: 60<br>(+): 8<br>(-): 90 | Method: Dunnett's post hoc test<br>p: 0.05<br>MSD: Not reported<br>Based on reduced offspring |

| Fipronil  | Wilson et al. 2008  | <i>C. dubia</i> |
|-----------|---|-----------------|
| Parameter | Value   | Comment         |
|           | <u>f<sub>1</sub> Adult survival time</u><br>Racemate: 60<br>(+): 64<br>(-): 90  |                 |
| LOEC      | <u>f<sub>1</sub> Time to first brood</u><br>Racemate: 120<br>(+): >64<br>(-): 90<br><u>f<sub>1</sub> No. of neonates/female</u><br>Racemate: 15<br>(+): 2<br>(-): 30<br><u>f<sub>1</sub> Average brood size</u><br>Racemate: 15<br>(+): 2<br>(-): 30<br><u>f<sub>1</sub> No. of broods/female</u><br>Racemate: 120<br>(+): 32<br>(-): 270<br><u>f<sub>1</sub> Adult survival time</u><br>Racemate: 120<br>(+): > 64<br>(-): 270   |                 |
| MATC      | <u>f<sub>1</sub> Time to first brood</u><br>Racemate: 85<br>(+): Not Calculable<br>(-): 52<br><u>f<sub>1</sub> No. of neonates/female</u><br>Racemate: Not Calculable<br>(+): Not Calculable<br>(-): 17<br><u>f<sub>1</sub> Average brood size</u><br>Racemate: Not Calculable<br>(+): Not Calculable<br>(-): 17<br><u>f<sub>1</sub> No. of broods/female</u><br>Racemate: 85<br>(+): 16<br>(-): 156<br><u>f<sub>1</sub> Adult survival time</u><br>Racemate: 85<br>(+): Not Calculable<br>(-): 156 |                 |

| Fipronil                        | Wilson et al. 2008  | <i>C. dubia</i> |
|---------------------------------|---|-----------------|
| Parameter                       | Value   | Comment         |
| % control at NOEC (tmt/control) | <u>f<sub>1</sub> Time to first brood</u><br>Racemate: 4.3/4.1=105%<br>(+): 5.8/4.1=141%<br>(-): 4.5/4.1=110%<br><u>f<sub>1</sub> No. of neonates/female</u><br>Racemate: Not Calculable<br>(+): Not Calculable<br>(-): 32.3/33.9=95%<br><u>f<sub>1</sub> Average brood size</u><br>Racemate: Not Calculable<br>(+): Not Calculable<br>(-): 10.0/10.5=95%<br><u>f<sub>1</sub> No. of broods/female</u><br>Racemate: 3.1/3.8=82%<br>(+): 2.6/3.7=70%<br>(-): 2.4/3.1=77%<br><u>f<sub>1</sub> Adult survival time</u><br>Racemate: 7.2/7.9=91%<br>(+): 6.1/7.6=80%<br>(-): 7.8/7.9=99% |                 |
| % control at LOEC (tmt/control) | <u>f<sub>1</sub> Time to first brood</u><br>Racemate: 5.6/4.1=137%<br>(+): Not Calculable<br>(-): 5.1/4.1=124%<br><u>f<sub>1</sub> No. of neonates/female</u><br>Racemate: 40.6/49.1=83%<br>(+): 15/26.4=57%<br>(-): 23.8/33.9=70%<br><u>f<sub>1</sub> Average brood size</u><br>Racemate: 10.7/13=82%<br>(+): 4.7/6.3=75%<br>(-): 7.6/10.5=72%<br><u>f<sub>1</sub> No. of broods/female</u><br>Racemate: 1.4/3.8=37%<br>(+): 1.8/3.7=49%<br>(-): 0/3.1=0%<br><u>f<sub>1</sub> Adult survival time</u><br>Racemate: 6.3/7.9=80%<br>(+): Not Calculable<br>(-): 2.6/7.9=33%          |                 |

Notes:

F2 generation hatched from f1 during the f1 exposures.

Solubility (S) of fipronil = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Measured concentrations (3), Hardness (2), Alkalinity (2), Conductivity (2), pH (3), Minimum significant difference (2). Total:  $100-14=86$

Acceptability: Measured concentrations within 20% nominal (4), Organisms randomized (1), Hardness (2), Alkalinity (2), Conductivity (1), pH (2), Minimum significant difference (1). Total:  $100-13=87$

**Reliability score:  $\text{mean}(86, 87)=86.5$**

## Water Toxicity Data Summary

*Ceriodaphnia dubia*

Fipronil

MB 46030

Wilson WA, Konwick BJ, Garrison AW, Avants JK and Black MC. (2008) Enantioselective chronic toxicity of fipronil to *Ceriodaphnia dubia*. *Archives of environmental contamination and toxicology*, 54(1), 36-43.

Relevance

Score: 100

Rating: R

Reliability

Score: 86.5

Rating: R

Relevance points taken off for: none.

| Fipronil                                     | Wilson et al. 2008  | <i>C. dubia</i>      |
|--|---|----------------------|
| Parameter                                    | Value   | Comment              |
| Test method cited                            | USEPA 2002, Method #1002.0                                |                      |
| Phylum/subphylum                             | Arthropoda  |                      |
| Class  | Branchiopoda  |                      |
| Order  | Cladocera   |                      |
| Family                                       | Daphniidae  |                      |
| Genus  | <i>Ceriodaphnia</i>                                       |                      |
| Species                                      | <i>dubia</i>  |                      |
| Family native to North America?              | Yes   |                      |
| Age/size at start of test/growth phase       | f <sub>1</sub> : <24 h<br>f <sub>2</sub> : <24 h          |                      |
| Source of organisms                          | Region IV Ecological Services Laboratory, Athens, Georgia |                      |
| Have organisms been exposed to contaminants? | No  |                      |
| Animals acclimated and disease-free?         | Yes   |                      |
| Animals randomized?                          | Not reported  |                      |
| Test vessels randomized?                     | Yes   |                      |
| Test duration                                | f <sub>1</sub> : 8 d<br>f <sub>2</sub> : 48 h post birth  |                      |
| Data for multiple times?                     | 24, 48 h  |                      |
| Effect 1                                     | f <sub>1</sub> : Impaired movement                        |                      |
| Control response 1                           | Not reported  | Qualitative endpoint |
| Effect 2                                     | Survival  |                      |
| Control response 2                           | Not reported  |                      |

| Fipronil  | Wilson et al. 2008   | <i>C. dubia</i>   |
|---|--|---|
| Parameter   | Value  | Comment   |
|   | 7.6-7.9 d<br><br>f <sub>2</sub> :<br>24 h: 5.3<br>48 h: 11.8                               |   |
| Temperature   | 25 ± 1°C   |   |
| Test type   | Static   |   |
| Photoperiod/light intensity   | 16l:8d   |   |
| Dilution water  | Moderately hard water  |   |
| pH  | Not reported   |   |
| Hardness  | Not reported   |   |
| Alkalinity  | Not reported   |   |
| Conductivity  | Not reported   |   |
| Dissolved Oxygen  | >7.0 mg/L  |   |
| Feeding   | <i>P. subcapitata</i> and yeast,<br>cereal leaves, and digested<br>Tetramin1 mixture daily |   |
| Purity of test substance  | Racemate: 98 %<br>(+): Konwick 2005, 97.3 %<br>(-): Konwick 2005, 98.1 %                   |   |
| Concentrations measured?  | Not reported   |   |
| Measured is what % of nominal?  | Not reported   |   |
| Toxicity values calculated based on nominal or measured concentrations? | Nominal  |   |
| Chemical method documented?   | GCMS   |   |
| Concentration of carrier (if any) in test solutions                     | Acetone, 0.1 %   |   |
| Concentration 1 Nom; Meas (µg/L)  | Racemate: 15; Not reported<br>(+): 2; Not reported<br>(-): 10; Not reported                | f <sub>1</sub> :10 reps, 1/rep<br>f <sub>2</sub> : 10 reps, various<br>brood size/rep |
| Concentration 2 Nom; Meas (µg/L)  | Racemate: 30; Not reported<br>(+): 8; Not reported<br>(-): 30; Not reported                |   |
| Concentration 3 Nom; Meas (µg/L)  | Racemate: 60; Not reported<br>(+): 32; Not reported<br>(-): 90; Not reported               |   |
| Concentration 4 Nom; Meas (µg/L)  | Racemate: 120; Not reported<br>(+): 64; Not reported<br>(-): 270; Not reported             |   |
| Control   | Solvent<br>Negative  |   |

| Fipronil                         | Wilson et al. 2008  | <i>C. dubia</i>   |
|----------------------------------|---|---|
| Parameter                        | Value   | Comment   |
| LC <sub>50</sub> (95% CI) (µg/L) | f <sub>2</sub> :<br>24 h:<br>Racemate: 33.3 (3.1)<br>(+): 18.1 (4.7)<br>(-): 65.2 (15.8)<br>48 h:<br>Racemate: 30.3 (3.4)<br>(+): 10.3 (3.1)<br>(-): 50.1 (2.4) | Method: Trimmed Spearman-Kärber   |
| NOEC                             | f <sub>1</sub> :<br>Racemate: 30<br>(+): 8<br>(-): 30   | Method: Dunnett's post hoc test<br>p: 0.05<br>MSD: Not reported<br>Based on reduced offspring   |
| LOEC                             | f <sub>1</sub> :<br>Racemate: 60<br>(+): 32<br>(-): 90  |   |
| MATC                             | f <sub>1</sub> :<br>Racemate: 42<br>(+): 16<br>(-): 52  |   |
| % control at NOEC                | f <sub>1</sub> :<br>Racemate: 143 %<br>(+): 53 %<br>(-): 70 %   | Racemate: 32.6 (tmt) / 22.8 (control) = 143 %<br><br>(+): 14.1 (tmt) / 26.4 (control) = 53 %<br><br>(-): 23.8 (tmt) / 33.9 (control) = 70 % |
| % control at LOEC                | f <sub>1</sub> :<br>Racemate: 46 %<br>(+): 28 %<br>(-): 30 %  | Racemate: 22.8 (tmt) / 49.1 (control) = 46 %<br>(+): 7.4 (tmt) / 26.4 (control) = 28 %<br>(-): 10.3 (tmt) / 33.9 (control) = 30 %           |

Notes: Solubility (S) of fipronil = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Measured concentrations (3), Hardness (2), Alkalinity (2), Conductivity (2), pH (3), Minimum significant difference (2). Total:  $100-14=86$

Acceptability: Measured concentrations within 20% nominal (4), Organisms randomized (1), Hardness (2), Alkalinity (2), Conductivity (1), pH (2), Minimum significant difference (1). Total:  $100-13=87$

**Reliability score:  $\text{mean}(86, 87)=86.5$**

## Water Toxicity Data Summary

*Chironomus dilutus*

Fipronil

MB46030

Weston DP and Lydy MJ. (2014) Toxicity of the insecticide fipronil and its degradates to benthic macroinvertebrates of urban streams. *Environmental science & technology*, 48(2), 1290-1297.

Relevance

Score: 90

Rating: R

Reliability

Score: 85.5

Rating: R

Relevance points taken off for: Standard method (10). 100-10=90

| Fipronil                                     | Weston & Lydy 2014                              | <i>C. dilutus</i> |
|--|---|-------------------|
| Parameter                                    | Value   | Comment           |
| Test method cited                            | Not reported                                    |                   |
| Phylum/subphylum                             | Anthropoda                                      |                   |
| Class  | Insecta   |                   |
| Order  | Diptera   |                   |
| Family                                       | Chironomidae                                    |                   |
| Genus  | <i>Chironomus</i>                               |                   |
| Species                                      | <i>dilutus</i>                                  |                   |
| Family native to North America?              | Yes   |                   |
| Age/size at start of test/growth phase       | Not reported                                    |                   |
| Source of organisms                          | University of California Berkeley lab culture   |                   |
| Have organisms been exposed to contaminants? | No  |                   |
| Animals acclimated and disease-free?         | Yes   |                   |
| Animals randomized?                          | Not reported                                    |                   |
| Test vessels randomized?                     | Not reported                                    |                   |
| Test duration                                | 96 h  |                   |
| Data for multiple times?                     | Not reported                                    |                   |
| Effect 1                                     | Survival  |                   |
| Control response 1                           | 100 %   |                   |
| Effect 2                                     | Immobilization (ability to thrash when prodded) |                   |
| Control response 2                           | Not reported                                    |                   |
| Temperature                                  | 23 °C   |                   |
| Test type                                    | Static  |                   |
| Photoperiod/light intensity                  | 16l:8d; Not reported                            |                   |
| Dilution water                               | Milli-Q purified, deionized                     | *According to     |

| Fipronil  | Weston & Lydy 2014                             | <i>C. dilutus</i>                                       |
|---|--|---|
| Parameter   | Value  | Comment   |
|   | watermade moderately hard by addition of salts | EPA 821-R-02-012  |
| pH  | Not reported*                                  |   |
| Hardness  | Not reported*                                  |   |
| Alkalinity  | Not reported*                                  |   |
| Conductivity  | Not reported*                                  |   |
| Dissolved Oxygen  | Not reported*                                  |   |
| Feeding   | 0.5 mL Tetrafin fish food slurry               |   |
| Purity of test substance  | 99.50 %  | Not reported but author verified from chemical supplier |
| Concentrations measured?  | Yes  |   |
| Measured is what % of nominal?  | 66-113%  |   |
| Toxicity values calculated based on nominal or measured concentrations? | Measured                                       |   |
| Chemical method documented?   | GC   |   |
| Concentration of carrier (if any) in test solutions                     | Acetone, <36 µg/L                              |   |
| Purity of test substance  | 99.50 %  | Not reported but author verified from chemical supplier |
| Control   | Negative: 0; 0<br>Solvent: 0; 0                |   |
| LC <sub>50</sub> (95% CI) (µg/L)  | 0.0300 (0.0233-0.0360)                         | Method: Probit  |
| EC <sub>50</sub> (95% CI) (µg/L)  | >0.0815  | Method: Probit  |

Notes: Two tests performed; lowest values included. Points were not deducted for lack of water quality parameter reporting because water was prepared according to EPA 821-R-02-012 and measured during study (but not reported).

Solubility (S) of fipronil = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Organism life stage/size (5), Nominal concentrations (3), Measured concentrations (3), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100-15 =85

Acceptability: Standard method (5), Organisms randomized (1), Temperature variation (3), Random design (2), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1). Total: 100-14 =86

**Reliability score: mean(85,86)=85.5**

## Water Toxicity Data Summary

*Chironomus dilutus*

Fipronil sulfide

MB45950

Weston DP and Lydy MJ. (2014) Toxicity of the insecticide fipronil and its degradates to benthic macroinvertebrates of urban streams. *Environmental science & technology*, 48(2), 1290-1297.

Relevance

Score: 90

Rating: R

Reliability

Score: 85.5

Rating: R

Relevance points taken off for: Standard method (10). 100-10=90

| Fipronil sulfide                             | <b>Weston &amp; Lydy 2014</b>                   | <i>C. dilutus</i> |
|--|---|-------------------|
| <b>Parameter</b>                             | <b>Value</b>                                    | <b>Comment</b>    |
| Test method cited                            | Not reported                                    |                   |
| Phylum/subphylum                             | Anthropoda                                      |                   |
| Class  | Insecta   |                   |
| Order  | Diptera   |                   |
| Family                                       | Chironomidae                                    |                   |
| Genus  | <i>Chironomus</i>                               |                   |
| Species                                      | <i>dilutus</i>                                  |                   |
| Family native to North America?              | Yes   |                   |
| Age/size at start of test/growth phase       | Not reported                                    |                   |
| Source of organisms                          | University of California Berkeley lab culture   |                   |
| Have organisms been exposed to contaminants? | No  |                   |
| Animals acclimated and disease-free?         | Yes   |                   |
| Animals randomized?                          | Not reported                                    |                   |
| Test vessels randomized?                     | Not reported                                    |                   |
| Test duration                                | 96 h  |                   |
| Data for multiple times?                     | Not reported                                    |                   |
| Effect 1                                     | Survival  |                   |
| Control response 1                           | 77 %  |                   |
| Effect 2                                     | Immobilization (ability to thrash when prodded) |                   |
| Control response 2                           | Not reported                                    |                   |
| Temperature                                  | 23 °C   |                   |
| Test type                                    | Static  |                   |
| Photoperiod/light intensity                  | 16l:8d; Not reported                            |                   |
| Dilution water                               | Milli-Q purified, deionized                     | *According to     |

| Fipronil sulfide  | Weston & Lydy 2014                             | <i>C. dilutus</i>                                       |
|---|--|---|
| Parameter   | Value  | Comment   |
|   | watermade moderately hard by addition of salts | EPA 821-R-02-012  |
| pH  | Not reported*                                  |   |
| Hardness  | Not reported*                                  |   |
| Alkalinity  | Not reported*                                  |   |
| Conductivity  | Not reported*                                  |   |
| Dissolved Oxygen  | Not reported*                                  |   |
| Feeding   | 0.5 mL Tetrafin fish food slurry               |   |
| Purity of test substance  | 99.0 %   | Not reported but author verified from chemical supplier |
| Concentrations measured?  | Yes  |   |
| Measured is what % of nominal?  | 66-113%  |   |
| Toxicity values calculated based on nominal or measured concentrations? | Measured                                       |   |
| Chemical method documented?   | GC   |   |
| Concentration of carrier (if any) in test solutions                     | Acetone, <36 µg/L                              |   |
| Purity of test substance  | 99.50 %  | Not reported but author verified from chemical supplier |
| Control   | Negative: 0; 0<br>Solvent: 0; 0                |   |
| LC <sub>50</sub> (95% CI) (µg/L)  | >0.0624  | Method: Probit  |
| EC <sub>50</sub> (95% CI) (µg/L)  | 0.0093 (0.0076-0.0114)                         | Method: Probit  |

Notes: Two tests performed; lowest values included. Points were not deducted for lack of water quality parameter reporting because water was prepared according to EPA 821-R-02-012 and measured during study (but not reported).

Solubility value for fipronil sulfide (MB 45950) not available. Solubility (S) of fipronil parent compound (MB 46030) = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Organism life stage/size (5), Nominal concentrations (3), Measured concentrations (3), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100-15 =85

Acceptability: Standard method (5), Organisms randomized (1), Temperature variation (3), Random design (2), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1). Total: 100-14 =86

**Reliability score: mean(85,86)=85.5**

## Water Toxicity Data Summary

*Chironomus dilutus*

Fipronil sulfone

MB46136

Weston DP and Lydy MJ. (2014) Toxicity of the insecticide fipronil and its degradates to benthic macroinvertebrates of urban streams. *Environmental science & technology*, 48(2), 1290-1297.

Relevance

Score: 90

Rating: R

Reliability

Score: 85.5

Rating: R

Relevance points taken off for: Standard method (10). 100-10=90

| Fipronil sulfone                             | Weston & Lydy 2014                              | <i>C. dilutus</i> |
|--|---|-------------------|
| Parameter                                    | Value   | Comment           |
| Test method cited                            | Not reported                                    |                   |
| Phylum/subphylum                             | Anthropoda                                      |                   |
| Class  | Insecta   |                   |
| Order  | Diptera   |                   |
| Family                                       | Chironomidae                                    |                   |
| Genus  | <i>Chironomus</i>                               |                   |
| Species                                      | <i>dilutus</i>                                  |                   |
| Family native to North America?              | Yes   |                   |
| Age/size at start of test/growth phase       | Not reported                                    |                   |
| Source of organisms                          | University of California Berkeley lab culture   |                   |
| Have organisms been exposed to contaminants? | No  |                   |
| Animals acclimated and disease-free?         | Yes   |                   |
| Animals randomized?                          | Not reported                                    |                   |
| Test vessels randomized?                     | Not reported                                    |                   |
| Test duration                                | 96 h  |                   |
| Data for multiple times?                     | Not reported                                    |                   |
| Effect 1                                     | Survival  |                   |
| Control response 1                           | 86 %  |                   |
| Effect 2                                     | Immobilization (ability to thrash when prodded) |                   |
| Control response 2                           | Not reported                                    |                   |
| Temperature                                  | 23 °C   |                   |
| Test type                                    | Static  |                   |
| Photoperiod/light intensity                  | 16l:8d; Not reported                            |                   |
| Dilution water                               | Milli-Q purified, deionized                     | *According to     |

| Fipronil sulfone  | Weston & Lydy 2014                             | <i>C. dilutus</i>                                       |
|---|--|---|
| Parameter   | Value  | Comment   |
|   | watermade moderately hard by addition of salts | EPA 821-R-02-012  |
| pH  | Not reported*                                  |   |
| Hardness  | Not reported*                                  |   |
| Alkalinity  | Not reported*                                  |   |
| Conductivity  | Not reported*                                  |   |
| Dissolved Oxygen  | Not reported*                                  |   |
| Feeding   | 0.5 mL Tetrafin fish food slurry               |   |
| Purity of test substance  | 99.0 %   | Not reported but author verified from chemical supplier |
| Concentrations measured?  | Yes  |   |
| Measured is what % of nominal?  | 66-113%  |   |
| Toxicity values calculated based on nominal or measured concentrations? | Measured                                       |   |
| Chemical method documented?   | GC   |   |
| Concentration of carrier (if any) in test solutions                     | Acetone, <36 µg/L                              |   |
| Purity of test substance  | 99.3 %   | Not reported but author verified from chemical supplier |
| Control   | Negative: 0; 0<br>Solvent: 0; 0                |   |
| LC <sub>50</sub> (95% CI) (µg/L)  | >0.102   | Method: Probit  |
| EC <sub>50</sub> (95% CI) (µg/L)  | 0.0079 (0.0050-0.0103)                         | Method: Probit  |

Notes: Two tests performed; lowest values included. Points were not deducted for lack of water quality parameter reporting because water was prepared according to EPA 821-R-02-012 and measured during study (but not reported).

Solubility (S) value for fipronil sulfone (MB 46136) = 160 µg/L, 2S = 320 µg/L.

Reliability points taken off for:

Documentation: Organism life stage/size (5), Nominal concentrations (3), Measured concentrations (3), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100-15 =85

Acceptability: Standard method (5), Organisms randomized (1), Temperature variation (3), Random design (2), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1). Total: 100-14 =86

**Reliability score: mean(85,86)=85.5**

## Water Toxicity Data Summary

*Chironomus riparius*  
Fipronil carboxamide  
RPA 200766

Funk M, Grote C. (2004) Effect of reg. no. 5300605 (metabolite of BAS 350 I, RPA 200766) on the mortality of *Chironomus riparius* in a 48 hours static, acute toxicity test. BASF Agricultural Center Limburgerhof. Limburgerhof, Germany. Study code 198235. Submitted to BASF Aktiengesellschaft, Limburgerhof, Germany. US EPA MRID 46376701.

Relevance  
Score: 100  
Rating: R

Reliability  
Score: 91.5  
Rating: R

Relevance points taken off for: none.

| Fipronil carboxamide                         | <b>Funk 2004</b>                    | <i>C. riparius</i> |
|--|-------------------------------------|--------------------|
| <b>Parameter</b>                             | <b>Value</b>                        | <b>Comment</b>     |
| Test method cited                            | OECD Guideline 202                  |                    |
| Phylum/subphylum                             | Anthropoda                          |                    |
| Class  | Insecta                             |                    |
| Order  | Diptera                             |                    |
| Family                                       | Chironomidae                        |                    |
| Genus  | <i>Chironomus</i>                   |                    |
| Species                                      | <i>riparius</i>                     |                    |
| Family native to North America?              | Yes                                 |                    |
| Age/size at start of test/growth phase       | <3 d, 1 <sup>st</sup> instar larvae |                    |
| Source of organisms                          | Laboratory cultures                 |                    |
| Have organisms been exposed to contaminants? | Yes                                 |                    |
| Animals acclimated and disease-free?         | Yes                                 |                    |
| Animals randomized?                          | Not reported                        |                    |
| Test vessels randomized?                     | Not reported                        |                    |
| Test duration                                | 48 h                                |                    |
| Data for multiple times?                     | 24, 48 h                            |                    |
| Effect 1                                     | Survival                            |                    |
| Control response 1                           | 100 %                               |                    |
| Temperature                                  | 21 ± 1 °C                           |                    |
| Test type                                    | Static                              |                    |
| Photoperiod/light intensity                  | 16l:8d/130-200 lux                  |                    |
| Dilution water                               | M4 Elendt medium                    |                    |
| pH   | 7.97                                |                    |
| Hardness                                     | 2.55 mmol/L CaCO <sub>3</sub>       |                    |

| Fipronil carboxamide  | <b>Funk 2004</b>                    | <i>C. riparius</i>   |
|---|-------------------------------------|--|
| <b>Parameter</b>  | <b>Value</b>                        | <b>Comment</b>   |
| Alkalinity  | 0.89 mmol/L CaCO <sub>3</sub>       |  |
| Conductivity  | 617 µmhos/cm                        |  |
| Dissolved Oxygen  | 8.72 mg/L                           | 98 %   |
| Feeding   | Pinch of Tetramin and quartz sand   |  |
| Purity of test substance  | 99.8 %                              |  |
| Concentrations measured?  | Yes                                 |  |
| Measured is what % of nominal?  | 75.8-92..0 %                        |  |
| Toxicity values calculated based on nominal or measured concentrations? | Measured                            |  |
| Chemical method documented?   | HPLC, LC-MS/MS                      |  |
| Concentration of carrier (if any) in test solutions                     | Acetone, concentration not reported |  |
| Concentration 1 Nom; Meas (µg/L)  | 10; 8                               | 4 reps, 5/rep  |
| Concentration 2 Nom; Meas (µg/L)  | 33; 26                              |  |
| Concentration 3 Nom; Meas (µg/L)  | 100; 90                             |  |
| Concentration 4 Nom; Meas (µg/L)  | 330; 260                            |  |
| Concentration 5 Nom; Meas (µg/L)  | 1000; 870                           |  |
| Concentration 6 Nom; Meas (µg/L)  | 3000; 2560                          |  |
| Control   | Negative: 0; 0<br>Solvent: 0; 0     |  |
| LC <sub>50</sub> (95% CI) (µg/L)  | 250 (100-630)                       | Method: Spearman-Kärber                                      |
| NOEC  | 8                                   | Method: Not reported<br>p: Not reported<br>MSD: Not reported |
| LOEC  | 90                                  | Not reported; See Table 3                                    |
| MATC (GeoMean NOEC, LOEC)   | 27                                  |  |
| % control at NOEC   | 100 %                               |  |
| % control at LOEC   | 75 %                                |  |

Notes:

Solubility value for fipronil carboxamide (RPA 200766) not available. Solubility (S) of fipronil parent compound (MB 46030) = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Statistical significance (2), Significance level (2), Minimum significant difference (2). Total: 100-6 =94

Acceptability: Carrier solvent (4), Organisms randomized (1), Feeding (3), Random design (2), Minimum significant difference (1). Total: 100-11 =89

**Reliability score: mean(94, 89)=91.5**

## Water Toxicity Data Summary

*Daphnia magna*

Fipronil

MB 46030

McNamara PC. (1990a) Acute toxicity to daphnids (*Daphnia magna*) during a 48-hour flow-through exposure. Springborn Laboratories, Inc., Wareham, Massachusetts. Laboratory study number 10566.1089.6146.115. Submitted to Rhone-Poulenc Ag Company, Research Triangle, North Carolina. USEPA MRID 42918625. CA DPR 157282 (1990) and 157283 (1996 duplicate).

Relevance

Score: 100

Rating: R

Reliability

Score: 94

Rating: R

Relevance points taken off for: none.

| Fipronil                                     | McNamara 1990a                 | <i>D. magna</i> |
|--|--------------------------------|-----------------|
| Parameter                                    | Value                          | Comment         |
| Test method cited                            | FIFRA Guidelines 72-2          |                 |
| Phylum/subphylum                             | Arthropoda/Crustacea           |                 |
| Class  | Branchiopoda                   |                 |
| Order  | Cladocera                      |                 |
| Family                                       | Daphniidae                     |                 |
| Genus  | <i>Daphnia</i>                 |                 |
| Species                                      | <i>magna</i>                   |                 |
| Family native to North America?              | Yes                            |                 |
| Age/size at start of test/growth phase       | <24 h                          |                 |
| Source of organisms                          | Laboratory cultures            |                 |
| Have organisms been exposed to contaminants? | No                             |                 |
| Animals acclimated and disease-free?         | Yes                            |                 |
| Animals randomized?                          | Yes                            |                 |
| Test vessels randomized?                     | Yes                            |                 |
| Test duration                                | 48 h                           |                 |
| Data for multiple times?                     | 24, 48 h                       |                 |
| Effect 1                                     | Immobilization                 |                 |
| Control response 1                           | Negative: 0 %<br>Solvent: 10 % |                 |
| Temperature                                  | 20 ± 1 °C                      |                 |
| Test type                                    | Flow through                   |                 |
| Photoperiod/light intensity                  | 16l:8d/40-70 footcandles       |                 |
| Dilution water                               | Fortified well water           |                 |

| Fipronil  | McNamara 1990a                    | <i>D. magna</i>  |
|---|-----------------------------------|--|
| Parameter   | Value                             | Comment  |
| pH  | 8.2-8.3                           |  |
| Hardness  | 170 mg/L CaCO <sub>3</sub>        |  |
| Alkalinity  | 120 mg/L CaCO <sub>3</sub>        |  |
| Conductivity  | 500 µmhos/cm                      |  |
| Dissolved Oxygen  | 7.7-8.6 mg/L                      | 85-95 %  |
| Feeding   | Not reported                      |  |
| Purity of test substance  | 100 %                             |  |
| Concentrations measured?  | Yes                               |  |
| Measured is what % of nominal?  | 67-85 %                           |  |
| Toxicity values calculated based on nominal or measured concentrations? | Measured                          |  |
| Chemical method documented?   | HPLC                              |  |
| Concentration of carrier (if any) in test solutions                     | Acetone, 90 µL/L                  |  |
| Concentration 1 Nom; Meas (µg/L)  | 47; 34                            | 2 reps, 10/rep   |
| Concentration 2 Nom; Meas (µg/L)  | 78; 52                            |  |
| Concentration 3 Nom; Meas (µg/L)  | 130; 110                          |  |
| Concentration 4 Nom; Meas (µg/L)  | 220; 160                          |  |
| Concentration 5 Nom; Meas (µg/L)  | 360; 280                          |  |
| Control   | Negative: 0; 0<br>Solvent: 0; 0   |  |
| EC <sub>50</sub> (95% CI) (µg/L)  | 24 h: >280<br>48 h: 190 (110-280) | Method: non-linear interpolation and binomial probability    |
| NOEC  | 52                                | Method: Not reported<br>p: Not reported<br>MSD: Not reported |
| % control at NOEC   | 100 %                             | 0.5 (tmt) / 0.5 (mean controls) = 100%                       |

Notes:

Solubility (S) of fipronil = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Statistical significance (2), Significance level (2), Minimum significant difference (2). Total: 100-6=94

Acceptability: Feeding (3), Adequate replication (2), Minimum significant difference (1). Total: 100-6 =94

**Reliability score: mean(94, 94)=94**

## Water Toxicity Data Summary

*Daphnia magna*  
Fipronil sulfide  
MB 45950

McNamara PC. (1990b) (M & B 45950)-Acute toxicity to daphnids (*Daphnia magna*) during a 48-hour flow-through exposure. Springborn Laboratories, Inc., Wareham, Massachusetts. Laboratory study number 10566.1089.6147.115. Submitted to Rhone-Poulenc Ag Company, Research Triangle, North Carolina. USEPA MRID 42918669. CA DPR 157307.

Relevance  
Score: 100  
Rating: R

Reliability  
Score: 94.5  
Rating: R

Relevance points taken off for: none.

| Fipronil sulfide                             | <b>McNamara 1990b</b>          | <i>D. magna</i> |
|--|--------------------------------|-----------------|
| <b>Parameter</b>                             | <b>Value</b>                   | <b>Comment</b>  |
| Test method cited                            | FIFRA 72-2                     |                 |
| Phylum/subphylum                             | Arthropoda/Crustacea           |                 |
| Class  | Branchiopoda                   |                 |
| Order  | Cladocera                      |                 |
| Family                                       | Daphniidae                     |                 |
| Genus  | <i>Daphnia</i>                 |                 |
| Species                                      | <i>magna</i>                   |                 |
| Family native to North America?              | Yes                            |                 |
| Age/size at start of test/growth phase       | <24 h                          |                 |
| Source of organisms                          | Laboratory cultures            |                 |
| Have organisms been exposed to contaminants? | No                             |                 |
| Animals acclimated and disease-free?         | Yes                            |                 |
| Animals randomized?                          | Yes                            |                 |
| Test vessels randomized?                     | Yes                            |                 |
| Test duration                                | 48 h                           |                 |
| Data for multiple times?                     | 24, 48 h                       |                 |
| Effect 1                                     | Immobilization                 |                 |
| Control response 1 (mean)                    | 2.5 %                          |                 |
| Temperature                                  | 20 ± 1 °C                      |                 |
| Test type                                    | Flow through                   |                 |
| Photoperiod/light intensity                  | 16l:8d/70-120 footcandles      |                 |
| Dilution water                               | Fortified well water           |                 |
| pH   | 8.1-8.3                        |                 |
| Hardness                                     | 170-180 mg/L CaCO <sub>3</sub> |                 |

| Parameter   | Value                                     | Comment  |
|---|---|--|
| Fipronil sulfide  | McNamara 1990b                            | <i>D. magna</i>  |
| Alkalinity  | 120 mg/L CaCO <sub>3</sub>                |  |
| Conductivity  | 500 µmhos/cm                              |  |
| Dissolved Oxygen  | 8.1-9.1 mg/L                              | 90-100 %   |
| Feeding   | Not fed                                   |  |
| Purity of test substance  | 100 %                                     |  |
| Concentrations measured?  | Yes                                       |  |
| Measured is what % of nominal?  | 72-89 %                                   |  |
| Toxicity values calculated based on nominal or measured concentrations? | Measured                                  |  |
| Chemical method documented?   | HPLC                                      |  |
| Concentration of carrier (if any) in test solutions                     | 0.090 mL/L acetone                        |  |
| Concentration 1 Nom; Meas (µg/L)  | 47; 34                                    | 2 reps, /rep   |
| Concentration 2 Nom; Meas (µg/L)  | 78; 60                                    |  |
| Concentration 3 Nom; Meas (µg/L)  | 130; 100                                  |  |
| Concentration 4 Nom; Meas (µg/L)  | 220; 180                                  |  |
| Concentration 5 Nom; Meas (µg/L)  | 360; 320                                  |  |
| Control   | Negative: 0; 0<br>Solvent: 0; 0           |  |
| EC <sub>50</sub> (95% CI) (µg/L)  | 24 h: 320 (210-950)<br>48 h: 100 (81-130) | Method: probit   |
| NOEC  | <34                                       | Method: Not reported<br>p: Not reported<br>MSD: Not reported |

Notes: Solubility value for fipronil sulfide (MB 45950) not available. Solubility (S) of fipronil parent compound (MB 46030) = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Statistical significance (2), Significance level (2), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100- 8=92

Acceptability: Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1). Total: 100- 3=97

**Reliability score: mean(92, 97)=94.5**

## Water Toxicity Data Summary

*Daphnia magna*  
Fipronil sulfone  
MB46136

McNamara PC. (1990c) (M&B 46136)-Acute toxicity to daphnids (*Daphnia magna*) during a 48-hour flow-through exposure. Springborn Laboratories, Inc., Wareham, Massachusetts. Laboratory study number 10566.1089.6148.115. Submitted to Rhone-Poulenc Ag Company, Research Triangle, North Carolina. USEPA MRID 42918671. CA DPR 157304.

Relevance  
Score: 100  
Rating: R

Reliability  
Score: 96  
Rating: R

Relevance points taken off for: none.

| Fipronil sulfone                             | <b>McNamara 1990c</b>      | <i>D. magna</i>                  |
|--|----------------------------|----------------------------------|
| <b>Parameter</b>                             | <b>Value</b>               | <b>Comment</b>                   |
| Test method cited                            | FIFRA Guideline 72-2       |                                  |
| Phylum/subphylum                             | Arthropoda/Crustacea       |                                  |
| Class  | Branchiopoda               |                                  |
| Order  | Cladocera                  |                                  |
| Family                                       | Daphniidae                 |                                  |
| Genus  | <i>Daphnia</i>             |                                  |
| Species                                      | <i>magna</i>               |                                  |
| Family native to North America?              | Yes                        |                                  |
| Age/size at start of test/growth phase       | <24 h                      |                                  |
| Source of organisms                          | Laboratory cultures        |                                  |
| Have organisms been exposed to contaminants? | No                         |                                  |
| Animals acclimated and disease-free?         | Yes                        |                                  |
| Animals randomized?                          | Yes                        |                                  |
| Test vessels randomized?                     | Yes                        |                                  |
| Test duration                                | 48 h                       |                                  |
| Data for multiple times?                     | 24, 48 h                   |                                  |
| Effect 1                                     | Immobilization             |                                  |
| Control response 1(mean)                     | 24 h: 0%<br>48 h: 5 %      |                                  |
| Temperature                                  | 20 ± 2 °C                  |                                  |
| Test type                                    | Flow through               |                                  |
| Photoperiod/light intensity                  | 16l:8d/120-150 footcandles |                                  |
| Dilution water                               | Fortified well water       | ASTM method for hard water, 1980 |

| Fipronil sulfone  | McNamara 1990c                         | <i>D. magna</i>  |
|---|--|--|
| Parameter   | Value                                  | Comment  |
| pH  | 8.1                                    |  |
| Hardness  | 160-170 mg/L CaCO <sub>3</sub>         |  |
| Alkalinity  | 120 mg/L CaCO <sub>3</sub>             |  |
| Conductivity  | 500 µmhos/cm                           |  |
| Dissolved Oxygen  | >5.5 mg/L                              | >60%   |
| Feeding   | Not fed                                |  |
| Purity of test substance  | 100 %                                  |  |
| Concentrations measured?  | Yes                                    |  |
| Measured is what % of nominal?  | 60-65 %                                |  |
| Toxicity values calculated based on nominal or measured concentrations? | Measured                               |  |
| Chemical method documented?   | HPLC                                   |  |
| Concentration of carrier (if any) in test solutions                     | 0.060 mL/L acetone                     |  |
| Concentration 1 Nom; Meas (µg/L)  | 31; 19                                 | 2 reps, 10/rep   |
| Concentration 2 Nom; Meas (µg/L)  | 52; 31                                 |  |
| Concentration 3 Nom; Meas (µg/L)  | 86; 56                                 |  |
| Concentration 4 Nom; Meas (µg/L)  | 140; 89                                |  |
| Concentration 5 Nom; Meas (µg/L)  | 240; 150                               |  |
| Control   | Negative: 0; 0<br>Solvent: 0; 0        |  |
| EC <sub>50</sub> (95% CI) (µg/L)  | 24 h: 110 (82-170)<br>48 h: 29 (20-38) | Method: Probit   |
| NOEC  | 48 h: <19                              | Method: Not reported<br>p: Not reported<br>MSD: Not reported |
| % control at NOEC   | Not calculable                         |  |

Notes: Dilution water TOC = 0.59 mg/L

Solubility (S) value for fipronil sulfone (MB 46136) = 160 µg/L, 2S = 320 µg/L.

Reliability points taken off for:

Documentation: Minimum significant difference (2). Total: 100-2 =98

Acceptability: Temperature variation (3), Measured concentrations within 20% nominal (4), Random design (2), Adequate Minimum significant difference (1). Total: 100-10 =90

**Reliability score: mean(98, 94)=96**

## Water Toxicity Data Summary

*Daphnia magna*

Fipronil-destrifluoromethyl-sulfonate

RPA 104615

Collins MK. (1993) RPA 104615-Acute toxicity to daphnids (*Daphnia magna*) under static. Springborn Laboratories, Inc., Wareham, Massachusetts. Laboratory study number 10566.0792.6245.110. Submitted to Rhone-Poulenc Ag Company, Research Triangle, North Carolina. USEPA MRID 43291719.

Relevance

Score: 85

Rating: L

Reliability

Score: 89.5

Rating: R

Relevance points taken off for: Toxicity value (15). 100-15=85

| Fipronil-destrifluoromethyl-sulfonate        | Collins 1993          | <i>D. magna</i> |
|--|-----------------------|-----------------|
| Parameter                                    | Value                 | Comment         |
| Test method cited                            | FIFRA 72-2            |                 |
| Phylum/subphylum                             | Arthropoda/Crustacea  |                 |
| Class  | Branchiopoda          |                 |
| Order  | Cladocera             |                 |
| Family                                       | Daphniidae            |                 |
| Genus  | <i>Daphnia</i>        |                 |
| Species                                      | <i>magna</i>          |                 |
| Family native to North America?              | Yes                   |                 |
| Age/size at start of test/growth phase       | <24 h                 |                 |
| Source of organisms                          | Laboratory cultures   |                 |
| Have organisms been exposed to contaminants? | No                    |                 |
| Animals acclimated and disease-free?         | Yes                   |                 |
| Animals randomized?                          | Yes                   |                 |
| Test vessels randomized?                     | Yes                   |                 |
| Test duration                                | 48 h                  |                 |
| Data for multiple times?                     | 24, 48 h              |                 |
| Effect 1                                     | Immobilization        |                 |
| Control response 1                           | Not reported          |                 |
| Temperature                                  | 22 ± 1 °C             |                 |
| Test type                                    | Static                |                 |
| Photoperiod/light intensity                  | 16l:8d/70 footcandles |                 |
| Dilution water                               | Fortified well water  |                 |
| pH   | 8.1                   |                 |

|   |                            |   |
|---|----------------------------|---|
| Fipronil-destrifluoromethyl-sulfonate                                   | <b>Collins 1993</b>        | <i>D. magna</i>   |
| <b>Parameter</b>  | <b>Value</b>               | <b>Comment</b>  |
| Hardness  | 170 mg/L CaCO <sub>3</sub> |   |
| Alkalinity  | 120 mg/L CaCO <sub>3</sub> |   |
| Conductivity  | 400-600 µmhos/cm           |   |
| Dissolved Oxygen  | > 5.2 mg/L                 | >60 %   |
| Feeding   | Not fed                    |   |
| Purity of test substance  | 94.7 %                     |   |
| Concentrations measured?  | No                         |   |
| Measured is what % of nominal?  | Not reported               |   |
| Toxicity values calculated based on nominal or measured concentrations? | Nominal                    |   |
| Chemical method documented?   | Not reported               |   |
| Concentration of carrier (if any) in test solutions                     | Not used                   |   |
| Concentration 1 Nom; Meas (µg/L)  | 13,000; Not reported       | 4 reps, 5/rep   |
| Concentration 2 Nom; Meas (µg/L)  | 22,000; Not reported       |   |
| Concentration 3 Nom; Meas (µg/L)  | 36,000; Not reported       |   |
| Concentration 4 Nom; Meas (µg/L)  | 60,000; Not reported       |   |
| Concentration 5 Nom; Meas (µg/L)  | 100,000; Not reported      |   |
| Control   | Negative: 0; 0             |   |
| EC <sub>50</sub> (95% CI) (µg/L)  | 48 h: >100,000             | Method:<br>Empirically<br>estimated                               |
| NOEC  | 22,000                     | Method: Not<br>reported.*<br>p: Not reported<br>MSD: Not reported |

Notes: \*No mortality observed in any treatment.

Solubility value for fipronil-destrifluoromethyl-sulfonate (RPA 104615) not available. Solubility (S) of fipronil parent compound (MB 46030) = 1650.8 µg/L, 2S = 3301.6 µg/L. Exposure concentrations exceed 2S of parent compound (fipronil, MB46030).

Reliability points taken off for:

Documentation: Measured concentrations (3), Statistical significance (2), Significance level (2), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100- 11=89

Acceptability: Measured concentrations within 20% nominal (4), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1), Point estimates (3). Total: 100-10=90

**Reliability score: mean(89, 90)=89.5**

## Water Toxicity Data Summary

*Daphnia magna*

Fipronil

MB46030

McNamara PC. (1990d) The chronic toxicity of M&B 46030 to *Daphnia magna* under flow-through conditions. Springborn Laboratories, Inc., Wareham, Massachusetts. Laboratory study number 10566.1089.6146.130. Submitted to Rhone-Poulenc Ag Company, Research Triangle, North Carolina. USEPA MRID 42918626. CA DPR 157288.

Relevance

Score: 100

Rating: R

Reliability

Score: 96

Rating: R

Relevance points taken off for: none.

| Fipronil                                     | McNamara 1990d                  | <i>D. magna</i> |
|--|---------------------------------|-----------------|
| Parameter                                    | Value                           | Comment         |
| Test method cited                            | FIFRA 72-2                      |                 |
| Phylum/subphylum                             | Arthropoda/Crustacea            |                 |
| Class  | Branchiopoda                    |                 |
| Order  | Cladocera                       |                 |
| Family                                       | Daphniidae                      |                 |
| Genus  | <i>Daphnia</i>                  |                 |
| Species                                      | <i>magna</i>                    |                 |
| Family native to North America?              | Yes                             |                 |
| Age/size at start of test/growth phase       | <24 h                           |                 |
| Source of organisms                          | Laboratory cultures             |                 |
| Have organisms been exposed to contaminants? | No                              |                 |
| Animals acclimated and disease-free?         | Yes                             |                 |
| Animals randomized?                          | Yes                             |                 |
| Test vessels randomized?                     | Not reported                    |                 |
| Test duration                                | 21 d                            |                 |
| Data for multiple times?                     | 1, 2,4, d then 3/wk days 7-21   |                 |
| Effect 1                                     | Survival                        |                 |
| Control response 1(mean)                     | 21 d: 74 %                      |                 |
| Effect 2                                     | Reproduction                    |                 |
| Control response 2 (mean)                    | 111 cumulative offspring/female |                 |
| Effect 3                                     | Growth                          |                 |

| Fipronil  | McNamara 1990d  | <i>D. magna</i>  |
|---|---|--|
| Parameter   | Value   | Comment  |
| Control response 3 (mean)   | 4.6 mm  |  |
| Temperature   | 20 ± 2 °C   |  |
| Test type   | Flow through  |  |
| Photoperiod/light intensity   | 16l:8d/40-90 footcandles  |  |
| Dilution water  | Fortified well water  | ASTM method for hard water, 1980   |
| pH  | 7.9-8.3   |  |
| Hardness  | 160-180 mg/L CaCO <sub>3</sub>  |  |
| Alkalinity  | 110-130 mg/L CaCO <sub>3</sub>  |  |
| Conductivity  | 400-600 µmhos/cm  |  |
| Dissolved Oxygen  | 7-8.4 mg/L  |  |
| Feeding   | Trout food, green algae, and Selco suspension 2-3/d                                       |  |
| Purity of test substance  | 100 %   |  |
| Concentrations measured?  | Yes   |  |
| Measured is what % of nominal?  | 68-79 %   |  |
| Toxicity values calculated based on nominal or measured concentrations? | Measured  |  |
| Chemical method documented?   | HPLC  |  |
| Concentration of carrier (if any) in test solutions                     | 17 µL/L acetone   |  |
| Concentration 1 Nom; Meas (µg/L)  | 6.3; 5.0  | 4 reps, 10/rep   |
| Concentration 2 Nom; Meas (µg/L)  | 13; 9.8   |  |
| Concentration 3 Nom; Meas (µg/L)  | 25; 20  |  |
| Concentration 4 Nom; Meas (µg/L)  | 50; 34  |  |
| Concentration 5 Nom; Meas (µg/L)  | 100; 79   |  |
| Control   | Negative: 0; 0<br>Solvent: 0; 0   |  |
| EC <sub>50</sub> (95% CI) (µg/L)  | 1, 2 d: >79<br>4 d: 61 (34-79)<br>7 d: 53 (34-79)<br>14 d: 41 (34-79)<br>21 d: 39 (34-79) | Method: Moving average, Probit, or non-linear interpolation                          |
| NOEC  | 9.8   | Method: William Test or Dunnett's Test<br>p:<br>MSD: Not reported<br>Based on growth |
| LOEC  | 20  | Based on growth  |
| MATC (GeoMean NOEC, LOEC)   | 14  | Based on growth  |
| % control at NOEC   | Growth: 104 %   | Growth: 4.8 (tmt) /  |

| Fipronil          | McNamara 1990d | <i>D. magna</i>                                      |
|-------------------|----------------|--|
| Parameter         | Value          | Comment  |
|                   |                | 4.6 (mean controls)<br>= 104 %                       |
| % control at LOEC | Growth: 96 %   | Growth: 4.4 (tmt) /<br>4.6 (mean controls)<br>= 96 % |

Notes:

Solubility (S) of fipronil = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Minimum significant difference (2). Total: 100-2 =98

Acceptability: Temperature variation (3), Random design (2), Adequate Minimum significant difference (1). Total: 100-6 =94

**Reliability score: mean(98, 94)=96**

## Water Toxicity Data Summary

*Daphnia magna*  
Fipronil sulfide  
MB45950

McNamara PC. (1990e) (M&B 45950)-Chronic toxicity to daphnids (*Daphnia magna*) under flow-through conditions. Springborn Laboratories, Inc., Wareham, Massachusetts. Laboratory study number 10566.1089.6147.130. Submitted to Rhone-Poulenc Ag Company, Research Triangle, North Carolina. USEPA MRID 42918670. CA DPR 157308.

Relevance  
Score: 100  
Rating: R

Reliability  
Score: 94  
Rating: R

Relevance points taken off for: none.

| Fipronil sulfide                             | <b>McNamara 1990e</b>           | <i>D. magna</i> |
|--|---------------------------------|-----------------|
| <b>Parameter</b>                             | <b>Value</b>                    | <b>Comment</b>  |
| Test method cited                            | OECD Guideline #202             |                 |
| Phylum/subphylum                             | Arthropoda/Crustacea            |                 |
| Class  | Branchiopoda                    |                 |
| Order  | Cladocera                       |                 |
| Family                                       | Daphniidae                      |                 |
| Genus  | <i>Daphnia</i>                  |                 |
| Species                                      | <i>magna</i>                    |                 |
| Family native to North America?              | Yes                             |                 |
| Age/size at start of test/growth phase       | <24 h                           |                 |
| Source of organisms                          | Laboratory cultures             |                 |
| Have organisms been exposed to contaminants? | No                              |                 |
| Animals acclimated and disease-free?         | Yes                             |                 |
| Animals randomized?                          | Yes                             |                 |
| Test vessels randomized?                     | Not reported                    |                 |
| Test duration                                | 21 d                            |                 |
| Data for multiple times?                     | 1, 2,4, d then 3/wk days 7-21   |                 |
| Effect 1                                     | Survival                        |                 |
| Control response 1(mean)                     | 21 d: 88 %                      |                 |
| Effect 2                                     | Reproduction                    |                 |
| Control response 2 (mean)                    | 124 cumulative offspring/female |                 |
| Effect 3                                     | Growth                          |                 |
| Control response 3 (mean)                    | 4.8 mm                          |                 |

|   |   |  |
|---|---|--|
| Fipronil sulfide  | <b>McNamara 1990e</b>                               | <i>D. magna</i>  |
| <b>Parameter</b>  | <b>Value</b>  | <b>Comment</b>   |
| Temperature   | 20 ± 2 °C   |  |
| Test type   | Flow through  |  |
| Photoperiod/light intensity   | 16l:8d/60-100 footcandles                           |  |
| Dilution water  | Fortified well water                                | ASTM method for hard water, 1980   |
| pH  | 7.9-8.3   |  |
| Hardness  | 160-180 mg/L CaCO <sub>3</sub>                      |  |
| Alkalinity  | 110-130 mg/L CaCO <sub>3</sub>                      |  |
| Conductivity  | 400-600 µmhos/cm                                    |  |
| Dissolved Oxygen  | >5.5 mg/L   | >60%   |
| Feeding   | Trout food, green algae, and Selco suspension 2-3/d |  |
| Purity of test substance  | 100 %   |  |
| Concentrations measured?  | Yes   |  |
| Measured is what % of nominal?  | 100-130 %   |  |
| Toxicity values calculated based on nominal or measured concentrations? | Measured  |  |
| Chemical method documented?   | HPLC  |  |
| Concentration of carrier (if any) in test solutions                     | 17 µL/L acetone                                     |  |
| Concentration 1 Nom; Meas (µg/L)  | 3.1; 4.0  | 2 reps, 20/rep   |
| Concentration 2 Nom; Meas (µg/L)  | 6.3; 7.0  |  |
| Concentration 3 Nom; Meas (µg/L)  | 13; 13  |  |
| Concentration 4 Nom; Meas (µg/L)  | 22; 25  |  |
| Concentration 5 Nom; Meas (µg/L)  | 45; 50  |  |
| Control   | Negative: 0; 0<br>Solvent: 0; 0                     |  |
| EC <sub>50</sub> (95% CI) (µg/L)  | 21 d: 27 (22-45)                                    | Method: Non-linear interpolation and binomial probability                              |
| NOEC  | 21 d: 13  | Method: William's Test<br>p: 0.05<br>MSD: Not reported<br>Based on growth/reproduction |
| LOEC  | 21 d: 22  | Based on growth/reproduction   |
| MATC (GeoMean NOEC, LOEC)   | 21 d: 17  | Based on growth/reproduction   |
| % control at NOEC   | 21 d:<br>Growth: 96 %                               | 21 d:<br>Growth: 4.6 (tmt) /   |

| Fipronil sulfide  | McNamara 1990e   | <i>D. magna</i>  |
|-------------------|--|--|
| Parameter         | Value  | Comment  |
|                   | Survival: 106 %<br>Reproduction: 92 %                        | 4.8 (mean controls) = 96 %<br><br>Survival: 93 (tmt) / 88 (mean controls) = 106 %<br><br>Reproduction: 114 (tmt) / 124 (mean controls) = 92 %                            |
| % control at LOEC | 21 d:<br>Growth: 88%<br>Survival: 83 %<br>Reproduction: 19 % | 21 d:<br>Growth: 4.2 (tmt) / 4.8 (mean controls) = 88 %<br><br>Survival: 73 (tmt) / 88 (mean controls) = 83 %<br><br>Reproduction: 23 (tmt) / 124 (mean controls) = 19 % |

Notes:

Solubility value for fipronil sulfide (MB 45950) not available. Solubility (S) of fipronil parent compound (MB 46030) = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Minimum significant difference (2). Total: 100-2 =98

Acceptability: Temperature variation (3), Measured concentrations within 20% nominal (4), Random design (2), Adequate Minimum significant difference (1). Total: 100-10 =90

**Reliability score: mean(98, 90)=94**

## Water Toxicity Data Summary

*Daphnia magna*  
Fipronil sulfone  
MB46136

McNamara PC. (1992) (M&B 46136)-Chronic toxicity to daphnids (*Daphnia magna*) under flow-through conditions. Springborn Laboratories, Inc., Wareham, Massachusetts. Laboratory study number 10566.1090.6175.130. Submitted to Rhone-Poulenc Ag Company, Research Triangle, North Carolina. CA DPR 157305.

Relevance  
Score: 100  
Rating: R

Reliability  
Score: 96.5  
Rating: R

Relevance points taken off for: none.

| Fipronil sulfone                             | <b>McNamara 1992</b>            | <i>D. magna</i> |
|--|---------------------------------|-----------------|
| <b>Parameter</b>                             | <b>Value</b>                    | <b>Comment</b>  |
| Test method cited                            | FIFRA 72-4                      |                 |
| Phylum/subphylum                             | Arthropoda/Crustacea            |                 |
| Class  | Branchiopoda                    |                 |
| Order  | Cladocera                       |                 |
| Family                                       | Daphniidae                      |                 |
| Genus  | <i>Daphnia</i>                  |                 |
| Species                                      | <i>magna</i>                    |                 |
| Family native to North America?              | Yes                             |                 |
| Age/size at start of test/growth phase       | <24 h                           |                 |
| Source of organisms                          | Laboratory cultures             |                 |
| Have organisms been exposed to contaminants? | No                              |                 |
| Animals acclimated and disease-free?         | Yes                             |                 |
| Animals randomized?                          | Yes                             |                 |
| Test vessels randomized?                     | Not reported                    |                 |
| Test duration                                | 21 d                            |                 |
| Data for multiple times?                     | 1, 2,4, d then 3/wk days 7-21   |                 |
| Effect 1                                     | Survival                        |                 |
| Control response 1(mean)                     | 21 d: 94 %                      |                 |
| Effect 2                                     | Reproduction                    |                 |
| Control response 2 (mean)                    | 170 cumulative offspring/female |                 |
| Effect 3                                     | Growth                          |                 |
| Control response 3 (mean)                    | 5.2 mm                          |                 |

| Fipronil sulfone  | McNamara 1992   | <i>D. magna</i>   |
|---|---|---|
| Parameter   | Value   | Comment   |
|   | 1.89 mg   |   |
| Temperature   | 20 ± 1 °C   |   |
| Test type   | Flow through  |   |
| Photoperiod/light intensity   | 16l:8d/50-85 footcandles  |   |
| Dilution water  | Fortified well water  | ASTM method for hard water, 1980  |
| pH  | 7.9-8.3   |   |
| Hardness  | 160-180 mg/L CaCO <sub>3</sub>  |   |
| Alkalinity  | 110-130 mg/L CaCO <sub>3</sub>  |   |
| Conductivity  | 400-500 µmhos/cm  |   |
| Dissolved Oxygen  | ≥7.8 mg/L   | >86%  |
| Feeding   | Trout food, green algae, and Selco suspension 2-3/d                             |   |
| Purity of test substance  | 99.2 %  |   |
| Concentrations measured?  | Yes   |   |
| Measured is what % of nominal?  | 84-100 %  |   |
| Toxicity values calculated based on nominal or measured concentrations? | Measured  |   |
| Chemical method documented?   | HPLC  |   |
| Concentration of carrier (if any) in test solutions                     | 17 µL/L acetone   |   |
| Concentration 1 Nom; Meas (µg/L)  | 0.75; 0.63  | 2 reps, 10/rep  |
| Concentration 2 Nom; Meas (µg/L)  | 1.5; 1.5  |   |
| Concentration 3 Nom; Meas (µg/L)  | 3.0; 2.6  |   |
| Concentration 4 Nom; Meas (µg/L)  | 6.0; 5.8  |   |
| Concentration 5 Nom; Meas (µg/L)  | 12; 12  |   |
| Control   | Negative: 0; 0<br>Solvent: 0; 0   |   |
| EC <sub>50</sub> (95% CI) (µg/L)  | 21 d: 4.5 (3.9-5.2)   | Method: Moving average  |
| NOEC  | 0.63  | Method: William's Test<br>p: 0.05<br>MSD: Not reported<br>Based on growth |
| LOEC  | 1.5   | Based on growth   |
| MATC (GeoMean NOEC, LOEC)   | 0.97  | Based on growth   |
| % control at NOEC   | 21 d:<br>Length: 100 %<br>Weight: 99 %<br>Survival: 104 %<br>Reproduction: 99 % | 21 d:<br>Length: 5.2 (tmt) /<br>5.2 (mean controls)<br>= 100 %            |

| Fipronil sulfone  | McNamara 1992   | <i>D. magna</i>   |
|-------------------|---|---|
| Parameter         | Value   | Comment   |
|                   |   | Weight: 1.90 (tmt) / 189 (mean controls) = 99 %<br><br>Survival: 98 (tmt) / 94 (mean controls) = 104 %<br><br>Reproduction: 169 (tmt) / 170 (mean controls) = 99 %  |
| % control at LOEC | 21 d:<br>Length: 96%<br>Weight: 86 %<br>Survival: 101 %<br>Reproduction: 68 % | 21 d:<br>Length: 5.0 (tmt) / 5.2 (mean controls) = 86 %<br><br>Weight: 1.63 (tmt) / 1.89 (mean controls) = %<br><br>Survival: 95 (tmt) / 94 (mean controls) = 101 %<br><br>Reproduction: 115 (tmt) / 170 (mean controls) = 68 % |

Notes: Dilution water TOC = 0.60-0.67 mg/L.

Solubility (S) value for fipronil sulfone (MB 46136) = 160 µg/L, 2S = 320 µg/L.

Reliability points taken off for:

Documentation: Minimum significant difference (2). Total: 100-2 =98

Acceptability: Random design (2), Adequate replication (2), Minimum significant difference (1). Total: 100-5 =95

**Reliability score: mean(98, 95)=96.5**

## Water Toxicity Data Summary

*Daphnia magna*  
Fipronil sulfone  
MB 46136

Janson, GM. (2014) Chronic toxicity of the BAS 350 I metabolite MB46136 (Reg. No. 4673253) to *Daphnia magna* Straus in a 21 day semi-static test. BASF SE, Limburgerhof, Germany. Study code 367103. Submitted to BASF Corporation, Research Triangle Park, North Carolina. CA DPR277084.

Relevance  
Score: 100  
Rating: R

Reliability  
Score: 90.5  
Rating: R

Relevance points taken off for: none.

| Fipronil sulfone                             | <b>Janson 2014</b>                | <i>D. magna</i> |
|--|-----------------------------------|-----------------|
| <b>Parameter</b>                             | <b>Value</b>                      | <b>Comment</b>  |
| Test method cited                            | OECD 211, OPPTS 850.133           |                 |
| Phylum/subphylum                             | Arthropoda/Crustacea              |                 |
| Class  | Branchiopoda                      |                 |
| Order  | Cladocera                         |                 |
| Family                                       | Daphniidae                        |                 |
| Genus  | <i>Daphnia</i>                    |                 |
| Species                                      | <i>magna</i>                      |                 |
| Family native to North America?              | Yes                               |                 |
| Age/size at start of test/growth phase       | Neonates: 2-24 h<br>Parents: 14 d |                 |
| Source of organisms                          | Laboratory cultures               |                 |
| Have organisms been exposed to contaminants? | No                                |                 |
| Animals acclimated and disease-free?         | Yes                               |                 |
| Animals randomized?                          | Not reported                      |                 |
| Test vessels randomized?                     | Not reported                      |                 |
| Test duration                                | 21 d                              |                 |
| Data for multiple times?                     | No                                |                 |
| Effect 1                                     | Survival                          |                 |
| Control response 1                           | 100 %                             |                 |
| Effect 2                                     | Cumulative offspring/female       |                 |
| Control response 2                           | 143                               |                 |
| Effect 3                                     | Age at first brood                |                 |
| Control response 3                           | 9                                 |                 |
| Effect 4                                     | Growth                            |                 |

| Fipronil sulfone  | Janson 2014  | <i>D. magna</i>  |
|---|--|--|
| Parameter   | Value  | Comment  |
| Control response 4  | Weight: 0.74 mg<br>Length: 4.7 mm<br>Growth rate: 0.37   |  |
| Temperature   | 21.5 ± 0.5 °C  |  |
| Test type   | Flow through   |  |
| Photoperiod/light intensity   | 16l:8d/220-885 lux   |  |
| Dilution water  | M4 (Elendt medium)   | Prepared with ultrapure deionized water  |
| pH  | 7.51-8.19  |  |
| Hardness  | 2.50-2.62 mmol/L CaCO <sub>3</sub>   |  |
| Alkalinity  | 0.86-0.89 mmol/L CaCO <sub>3</sub>   |  |
| Conductivity  | 654-671 µmhos/cm   |  |
| Dissolved Oxygen  | 7.17-9.26 mg/L   | 80-104 %, not aerated  |
| Feeding   | Algae ( <i>Desmodesmus subspicatus</i> )   |  |
| Purity of test substance  | 99.7 %   |  |
| Concentrations measured?  | Yes  |  |
| Measured is what % of nominal?  | 88-102 %   |  |
| Toxicity values calculated based on nominal or measured concentrations? | Time weighted mean measured  |  |
| Chemical method documented?   | LC/MS  |  |
| Concentration of carrier (if any) in test solutions                     | Acetone, 10 µL/L   |  |
| Concentration 1 Nom; Time weighted average (µg/L)                       | 0.25; 0.22   | 10 reps, 1/rep   |
| Concentration 2 Nom; Time weighted average (µg/L)                       | 0.5; 0.45  |  |
| Concentration 3 Nom; Time weighted average (µg/L)                       | 1.0; 0.93  |  |
| Concentration 4 Nom; Time weighted average (µg/L)                       | 2.0; 1.85  |  |
| Concentration 5 Nom; Time weighted average (µg/L)                       | 4.0; 3.76  |  |
| Control   | Negative: 0; 0<br>Solvent: 0; 0  |  |
| NOEC  | Parent survival: 1.85<br>Cumulative offspring/female: 0.93<br>Age at first brood: 1.85<br>Weight: 1.85 | Method: Dunnett's test (reproduction, growth), Fisher's exact test (survival)<br>p: 0.05 |

| Fipronil sulfone          | Janson 2014   | <i>D. magna</i>   |
|---------------------------|---|---|
| Parameter                 | Value   | Comment   |
|                           | Length: 0.45<br>Growth rate: 0.93   | MSD:  |
| LOEC                      | Parent survival: 3.76<br>Cumulative offspring/female: 1.85<br>Age at first brood: 3.76<br>Parent eight: 3.76<br>Parent length: 0.93<br>Parent growth rate: 1.85   |   |
| MATC (GeoMean NOEC, LOEC) | Parent survival: 2.63<br>Cumulative offspring/female: 1.31<br>Age at first brood: 2.63<br>Parent weight: 2.63<br>Parent length: 0.65<br>Parent growth rate: 1.31  |   |
| % control at NOEC         | Parent survival: 70 %<br><br>Cumulative offspring/female: 93 %<br><br>Age at first brood: 106 %<br><br>Weight: 68 %<br><br>Length: 97 %<br><br>Growth rate: 101 % | Parent survival: 7 (tmt) / 10 (mean controls) = 70 %<br><br>Cumulative offspring/female: 133 (tmt) / 143 (mean controls) = 93 %<br><br>Age at first brood: 9.6 (tmt) / 9.05 (mean controls) = 106 %<br><br>Weight: 0.617 (tmt) / 0.74 (mean controls) = 68 %<br><br>Length: 4.5 (tmt) / 4.65 (mean controls) = 97 %<br><br>Growth rate: 0.373 (tmt) / 0.370 (mean controls) = 101 % |
| % control at LOEC         | Parent survival: 20 %   | Parent survival: 2 (tmt) / 10 (mean   |

| Fipronil sulfone | Janson 2014                       | <i>D. magna</i>  |
|------------------|-----------------------------------|--|
| Parameter        | Value                             | Comment  |
|                  | Cumulative offspring/female: 48 % | controls) = 20 %   |
|                  | Age at first brood: 139 %         | Cumulative offspring/female: 68 (tmt) / 143 (mean controls) = 48 % |
|                  | Weight: 60 %                      |  |
|                  | Length: 95 %                      |  |
|                  | Growth rate: 86 %                 | Age at first brood: 12.5 (tmt) / 9.05 (mean controls) = 139 %      |
|                  |                                   | Weight: 0.440 (tmt) / 0.74 (mean controls) = 60 %                  |
|                  |                                   | Length: 4.4 (tmt) / 4.65 (mean controls) = 95 %                    |
|                  |                                   | Growth rate: 0.319 (tmt) / 0.370 (mean controls) = 86 %            |

Notes: Measured concentrations not reported, only 'time weighted average' concentrations.

Solubility (S) value for fipronil sulfone (MB 46136) = 160 µg/L, 2S = 320 µg/L.

Reliability points taken off for:

Documentation: Minimum significant difference (2), Point estimates (8). Total: 100-10 =90

Acceptability: Organisms randomized (1), Adequate organisms per rep (2), Random design (2), Minimum significant difference (1), Point estimates (3). Total: 100-9 =91

**Reliability score: mean(90, 91)=90.5**

## Water Toxicity Data Summary

*Daphnia magna*  
Fipronil desulfinyl  
MB46513

Putt AE. (1992a) MB46513-Chronic toxicity to daphnids (*Daphnia magna*) under static renewal conditions. Springborn Laboratories, Inc., Wareham, Massachusetts. Laboratory study number 10566.1090.6176.130. Submitted to Rhone-Poulenc Ag Company, Research Triangle, North Carolina. USEPA MRID 43279704. CA DPR 157300.

Relevance  
Score: 100  
Rating: R

Reliability  
Score: 98.5  
Rating: R

Relevance points taken off for: none.

| Fipronil desulfinyl                          | <b>Putt 1992a</b>               | <i>D. magna</i> |
|--|---------------------------------|-----------------|
| <b>Parameter</b>                             | <b>Value</b>                    | <b>Comment</b>  |
| Test method cited                            | FIFRA 72-4                      |                 |
| Phylum/subphylum                             | Arthropoda/Crustacea            |                 |
| Class  | Branchiopoda                    |                 |
| Order  | Cladocera                       |                 |
| Family                                       | Daphniidae                      |                 |
| Genus  | <i>Daphnia</i>                  |                 |
| Species                                      | <i>magna</i>                    |                 |
| Family native to North America?              | Yes                             |                 |
| Age/size at start of test/growth phase       | <24 h                           |                 |
| Source of organisms                          | Laboratory cultures             |                 |
| Have organisms been exposed to contaminants? | No                              |                 |
| Animals acclimated and disease-free?         | Yes                             |                 |
| Animals randomized?                          | Yes                             |                 |
| Test vessels randomized?                     | Yes                             |                 |
| Test duration                                | 21 d                            |                 |
| Data for multiple times?                     | 1, 2,4, d then 3/wk days 7-21   |                 |
| Effect 1                                     | Survival                        |                 |
| Control response 1(mean)                     | 21 d: 94 %                      |                 |
| Effect 2                                     | Reproduction                    |                 |
| Control response 2 (mean)                    | 187 cumulative offspring/female |                 |
| Effect 3                                     | Growth                          |                 |
| Control response 3 (mean)                    | 5.2 mm<br>1.99 mg               |                 |

|   |  |  |
|---|--|--|
| Fipronil desulfinyl   | <b>Putt 1992a</b>                              | <i>D. magna</i>  |
| <b>Parameter</b>  | <b>Value</b>                                   | <b>Comment</b>   |
| Temperature   | 20 ± 2 °C                                      |  |
| Test type   | Static renewal                                 |  |
| Photoperiod/light intensity   | 16l:8d/30-38 footcandles                       |  |
| Dilution water  | Fortified well water                           | ASTM method for hard water, 1980   |
| pH  | 7.9-8.3  |  |
| Hardness  | 160-180 mg/L CaCO <sub>3</sub>                 |  |
| Alkalinity  | 110-130 mg/L CaCO <sub>3</sub>                 |  |
| Conductivity  | 400-600 µmhos/cm                               |  |
| Dissolved Oxygen  | 7.2-8.4 mg/L                                   | 82-96 %  |
| Feeding   | Trout food, green algae, and Selco suspension  |  |
| Purity of test substance  | 97.81 %  |  |
| Concentrations measured?  | Yes  |  |
| Measured is what % of nominal?  | 84-106 %                                       |  |
| Toxicity values calculated based on nominal or measured concentrations? | Measured                                       |  |
| Chemical method documented?   | HPLC   |  |
| Concentration of carrier (if any) in test solutions                     | ≤0.1 mL/L acetone                              |  |
| Concentration 1 Nom; Meas (µg/L)  | 6.4; 6.6                                       | 4 reps, 10/rep   |
| Concentration 2 Nom; Meas (µg/L)  | 16; 17   |  |
| Concentration 3 Nom; Meas (µg/L)  | 40; 41   |  |
| Concentration 4 Nom; Meas (µg/L)  | 100; 100                                       |  |
| Concentration 5 Nom; Meas (µg/L)  | 250; 260                                       |  |
| Control   | Negative: 0; 0<br>Solvent: 0; 0                |  |
| EC <sub>50</sub> (95% CI) (µg/L)  | 1, 2, 4, 7, 14, d: >260<br>21 d: 230 (100-260) | Method: Non-linear interpolation   |
| NOEC  | 41   | Method: William Test (survival, growth) or Kruskal-Wallis Test (reproduction)<br>p: 0.05<br>MSD: Not reported<br>Based on growth |

| Fipronil desulfanyl       | Putt 1992a                     | <i>D. magna</i>  |
|---------------------------|--------------------------------|--|
| Parameter                 | Value                          | Comment  |
| LOEC                      | 100                            | Based on growth  |
| MATC (GeoMean NOEC, LOEC) | 64                             | Based on growth  |
| % control at NOEC         | Length: 100 %<br>Weight: 100 % | Length: 5.2 (tmt) /<br>5.2 (mean controls)<br>= 100 %<br><br>Weight: 5.2 (tmt) /<br>5.2 (mean controls)<br>= 100 % |
| % control at LOEC         | Length: 98 %<br>Weight: 88 %   | Length: 5.1 (tmt) /<br>5.2 (mean controls)<br>= 98 %<br><br>Weight: 1.75 (tmt) /<br>1.99 (mean<br>controls) = 88 % |

Notes: Dilution water TOC = 0.60 mg/L

Solubility (S) value for fipronil desulfanyl (MB 46513) = 950 µg/L, 2S = 1900 µg/L.

Reliability points taken off for:

Documentation: Minimum significant difference (2). Total: 100-2 =98

Acceptability: Minimum significant difference (1). Total: 100- 1=99

**Reliability score: mean(98, 99)=98.5**

## Water Toxicity Data Summary

*Dipheter hageni*

Fipronil

MB46030

Weston DP and Lydy MJ. (2014) Toxicity of the insecticide fipronil and its degradates to benthic macroinvertebrates of urban streams. *Environmental science & technology*, 48(2), 1290-1297.

Relevance

Score: 90

Rating: R

Reliability

Score: 85.5

Rating: R

Relevance points taken off for: Standard method (10). 100-10=90

| Fipronil                                     | Weston & Lydy 2014  | <i>D. hageni</i> |
|--|---|------------------|
| Parameter                                    | Value   | Comment          |
| Test method cited                            | Not reported  |                  |
| Phylum/subphylum                             | Anthropoda  |                  |
| Class  | Insecta   |                  |
| Order  | Ephemeroptera   |                  |
| Family                                       | Baetidae  |                  |
| Genus  | <i>Dipheter</i>   |                  |
| Species                                      | <i>hageni</i>   |                  |
| Family native to North America?              | Yes   |                  |
| Age/size at start of test/growth phase       | Not reported  |                  |
| Source of organisms                          | Urban waterbodies with minimal development in Northern California |                  |
| Have organisms been exposed to contaminants? | Not reported  |                  |
| Animals acclimated and disease-free?         | 24 h  |                  |
| Animals randomized?                          | Not reported  |                  |
| Test vessels randomized?                     | Not reported  |                  |
| Test duration                                | 48 h  |                  |
| Data for multiple times?                     | Not reported  |                  |
| Effect 1                                     | Survival  |                  |
| Control response 1                           | 90 %  |                  |
| Effect 2                                     | Immobilization (ability to swim)                                  |                  |
| Control response 2                           | Not reported  |                  |
| Temperature                                  | 18 °C   |                  |
| Test type                                    | Static  |                  |
| Photoperiod/light intensity                  | 16l:8d; Not reported  |                  |

| Fipronil  | Weston & Lydy 2014   | <i>D. hageni</i>  |
|---|--|---|
| Parameter   | Value  | Comment   |
| Dilution water  | Milli-Q purified, deionized watermade moderately hard by addition of salts | *According to EPA 821-R-02-012                          |
| pH  | Not reported*  |   |
| Hardness  | Not reported*  |   |
| Alkalinity  | Not reported*  |   |
| Conductivity  | Not reported*  |   |
| Dissolved Oxygen  | Not reported*  |   |
| Feeding   | Not reported   |   |
| Purity of test substance  | 99.50 %  | Not reported but author verified from chemical supplier |
| Concentrations measured?  | Yes  |   |
| Measured is what % of nominal?  | 66-113%  |   |
| Toxicity values calculated based on nominal or measured concentrations? | Measured   |   |
| Chemical method documented?   | GC   |   |
| Concentration of carrier (if any) in test solutions                     | Acetone, <36 µg/L  |   |
| Concentration 1 Nom; Meas (µg/L)  | Not reported; 4-7 concentrations tested; dilution factor of 2              | 3 reps, 4-6/rep   |
| Control   | Negative: 0; 0<br>Solvent: 0; 0  |   |
| LC <sub>50</sub> (95% CI) (µg/L)  | 0.163 (0.107-0.208)  | Method: Probit  |
| EC <sub>50</sub> (95% CI) (µg/L)  | 0.347 (0.196-0.568)  | Method: Probit  |

Notes: Points were not deducted for lack of water quality parameter reporting because water was prepared according to EPA 821-R-02-012 and measured during study (but not reported).

Solubility (S) of fipronil = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Organism life stage/size (5), Nominal concentrations (3), Measured concentrations (3), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100-15 =85

Acceptability: Standard method (5), Organisms randomized (1), Temperature variation (3), Random design (2), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1). Total: 100-14 =86

**Reliability score: mean(85,86)=85.5**

## Water Toxicity Data Summary

*Dipheter hageni*  
Fipronil sulfone  
MB46136

Weston DP and Lydy MJ. (2014) Toxicity of the insecticide fipronil and its degradates to benthic macroinvertebrates of urban streams. *Environmental science & technology*, 48(2), 1290-1297.

Relevance  
Score: 90  
Rating: R

Reliability  
Score: 85.5  
Rating: R

Relevance points taken off for: Standard method (10). 100-10=90

| Fipronil sulfone                             | Weston & Lydy 2014  | <i>D. hageni</i> |
|--|---|------------------|
| Parameter                                    | Value   | Comment          |
| Test method cited                            | Not reported  |                  |
| Phylum/subphylum                             | Anthropoda  |                  |
| Class  | Insecta   |                  |
| Order  | Ephemeroptera   |                  |
| Family                                       | Baetidae  |                  |
| Genus  | <i>Dipheter</i>   |                  |
| Species                                      | <i>hageni</i>   |                  |
| Family native to North America?              | Yes   |                  |
| Age/size at start of test/growth phase       | Not reported  |                  |
| Source of organisms                          | Urban waterbodies with minimal development in Northern California |                  |
| Have organisms been exposed to contaminants? | Not reported  |                  |
| Animals acclimated and disease-free?         | 24 h  |                  |
| Animals randomized?                          | Not reported  |                  |
| Test vessels randomized?                     | Not reported  |                  |
| Test duration                                | 48 h  |                  |
| Data for multiple times?                     | Not reported  |                  |
| Effect 1                                     | Survival  |                  |
| Control response 1                           | 87 %  |                  |
| Effect 2                                     | Immobilization (ability to swim)                                  |                  |
| Control response 2                           | Not reported  |                  |
| Temperature                                  | 18 °C   |                  |
| Test type                                    | Static  |                  |
| Photoperiod/light intensity                  | 16l:8d; Not reported  |                  |

| Fipronil sulfone  | Weston & Lydy 2014   | <i>D. hageni</i>  |
|---|--|---|
| Parameter   | Value  | Comment   |
| Dilution water  | Milli-Q purified, deionized watermade moderately hard by addition of salts | *According to EPA 821-R-02-012                          |
| pH  | Not reported*  |   |
| Hardness  | Not reported*  |   |
| Alkalinity  | Not reported*  |   |
| Conductivity  | Not reported*  |   |
| Dissolved Oxygen  | Not reported*  |   |
| Feeding   | Not reported   |   |
| Purity of test substance  | 99.3 %   | Not reported but author verified from chemical supplier |
| Concentrations measured?  | Yes  |   |
| Measured is what % of nominal?  | 66-113%  |   |
| Toxicity values calculated based on nominal or measured concentrations? | Measured   |   |
| Chemical method documented?   | GC   |   |
| Concentration of carrier (if any) in test solutions                     | Acetone, <36 µg/L  |   |
| Concentration 1 Nom; Meas (µg/L)  | Not reported; 4-7 concentrations tested; dilution factor of 2              | 3 reps, 4-6/rep   |
| Control   | Negative: 0; 0<br>Solvent: 0; 0  |   |
| LC <sub>50</sub> (95% CI) (µg/L)  | 0.330 (0.188-0.536)  | Method: Probit  |
| EC <sub>50</sub> (95% CI) (µg/L)  | 0.0926 (0.0565-0.128)  | Method: Probit  |

Notes: Points were not deducted for lack of water quality parameter reporting because water was prepared according to EPA 821-R-02-012 and measured during study (but not reported).

Solubility (S) value for fipronil sulfone (MB 46136) = 160 µg/L, 2S = 320 µg/L.

Reliability points taken off for:

Documentation: Organism life stage/size (5), Nominal concentrations (3), Measured concentrations (3), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100-15 =85

Acceptability: Standard method (5), Organisms randomized (1), Temperature variation (3), Random design (2), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1). Total: 100-14 =86

**Reliability score: mean(85,86)=85.5**

## Water Toxicity Data Summary

*Fallceon quilleri*

Fipronil

MB46030

Weston DP and Lydy MJ. (2014) Toxicity of the insecticide fipronil and its degradates to benthic macroinvertebrates of urban streams. *Environmental science & technology*, 48(2), 1290-1297.

Relevance

Score: 90

Rating: R

Reliability

Score: 85.5

Rating: R

Relevance points taken off for: Standard method (10). 100-10=90

|  | <b>Weston 2014</b>  | <b><i>F. quilleri</i></b> |
|--|---|---------------------------|
| <b>Parameter</b>                             | <b>Value</b>  | <b>Comment</b>            |
| Test method cited                            | Not reported  |                           |
| Phylum/subphylum                             | Anthropoda  |                           |
| Class  | Insecta   |                           |
| Order  | Ephemeroptera   |                           |
| Family                                       | Baetidae  |                           |
| Genus  | <i>Fallceon</i>   |                           |
| Species                                      | <i>quilleri</i>   |                           |
| Family native to North America?              | Yes   |                           |
| Age/size at start of test/growth phase       | Not reported  |                           |
| Source of organisms                          | Urban waterbodies with minimal development in Northern California |                           |
| Have organisms been exposed to contaminants? | Not reported  |                           |
| Animals acclimated and disease-free?         | 24 h  |                           |
| Animals randomized?                          | Not reported  |                           |
| Test vessels randomized?                     | Not reported  |                           |
| Test duration                                | 48 h  |                           |
| Data for multiple times?                     | Not reported  |                           |
| Effect 1                                     | Survival  |                           |
| Control response 1                           | 77 %  |                           |
| Effect 2                                     | Immobilization (ability to swim)                                  |                           |
| Control response 2                           | Not reported  |                           |
| Temperature                                  | 23 °C   |                           |
| Test type                                    | Static  |                           |
| Photoperiod/light intensity                  | 16l:8d; Not reported  |                           |

|   | <b>Weston 2014</b>   | <b><i>F. quilleri</i></b>                               |
|---|--|---|
| <b>Parameter</b>  | <b>Value</b>   | <b>Comment</b>  |
| Dilution water  | Milli-Q purified, deionized watermade moderately hard by addition of salts | *According to EPA 821-R-02-012                          |
| pH  | Not reported*  |   |
| Hardness  | Not reported*  |   |
| Alkalinity  | Not reported*  |   |
| Conductivity  | Not reported*  |   |
| Dissolved Oxygen  | Not reported*  |   |
| Feeding   | Not reported   |   |
| Purity of test substance  | 99.50 %  | Not reported but author verified from chemical supplier |
| Concentrations measured?  | Yes  |   |
| Measured is what % of nominal?  | 66-113%  |   |
| Toxicity values calculated based on nominal or measured concentrations? | Measured   |   |
| Chemical method documented?   | GC   |   |
| Concentration of carrier (if any) in test solutions                     | Acetone, <36 µg/L  |   |
| Concentration 1 Nom; Meas (µg/L)  | Not reported; 4-7 concentrations tested; dilution factor of 2              | 3 reps, 4-6/rep   |
| Control   | Negative: 0; 0<br>Solvent: 0; 0  |   |
| LC <sub>50</sub> (95% CI) (µg/L)  | 0.0707 (0.0.365-0.0935)  | Method: Probit  |
| EC <sub>50</sub> (95% CI) (µg/L)  | >0.187   | Method: Probit  |

Notes: Points were not deducted for lack of water quality parameter reporting because water was prepared according to EPA 821-R-02-012 and measured during study (but not reported).

Solubility (S) of fipronil = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Organism life stage/size (5), Nominal concentrations (3), Measured concentrations (3), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100-15 =85

Acceptability: Standard method (5), Organisms randomized (1), Temperature variation (3), Random design (2), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1). Total: 100-14 =86

**Reliability score: mean(85,86)=85.5**

## Water Toxicity Data Summary

*Fallceon quilleri*  
Fipronil sulfone  
MB46136

Weston DP and Lydy MJ. (2014) Toxicity of the insecticide fipronil and its degradates to benthic macroinvertebrates of urban streams. *Environmental science & technology*, 48(2), 1290-1297.

Relevance  
Score: 90  
Rating: R

Reliability  
Score: 85.5  
Rating: R

Relevance points taken off for: Standard method (10). 100-10=90

|  | <b>Weston 2014</b>  | <b><i>F. quilleri</i></b> |
|--|---|---------------------------|
| <b>Parameter</b>                             | <b>Value</b>  | <b>Comment</b>            |
| Test method cited                            | Not reported  |                           |
| Phylum/subphylum                             | Anthropoda  |                           |
| Class  | Insecta   |                           |
| Order  | Ephemeroptera   |                           |
| Family                                       | Baetidae  |                           |
| Genus  | <i>Fallceon</i>   |                           |
| Species                                      | <i>quilleri</i>   |                           |
| Family native to North America?              | Yes   |                           |
| Age/size at start of test/growth phase       | Not reported  |                           |
| Source of organisms                          | Urban waterbodies with minimal development in Northern California |                           |
| Have organisms been exposed to contaminants? | Not reported  |                           |
| Animals acclimated and disease-free?         | 24 h  |                           |
| Animals randomized?                          | Not reported  |                           |
| Test vessels randomized?                     | Not reported  |                           |
| Test duration                                | 48 h  |                           |
| Data for multiple times?                     | Not reported  |                           |
| Effect 1                                     | Survival  |                           |
| Control response 1                           | 95 %  |                           |
| Effect 2                                     | Immobilization (ability to swim)                                  |                           |
| Control response 2                           | Not reported  |                           |
| Temperature                                  | 23 °C   |                           |
| Test type                                    | Static  |                           |
| Photoperiod/light intensity                  | 16l:8d; Not reported  |                           |

|   | <b>Weston 2014</b>   | <b><i>F. quilleri</i></b>                               |
|---|--|---|
| <b>Parameter</b>  | <b>Value</b>   | <b>Comment</b>  |
| Dilution water  | Milli-Q purified, deionized watermade moderately hard by addition of salts | *According to EPA 821-R-02-012                          |
| pH  | Not reported*  |   |
| Hardness  | Not reported*  |   |
| Alkalinity  | Not reported*  |   |
| Conductivity  | Not reported*  |   |
| Dissolved Oxygen  | Not reported*  |   |
| Feeding   | Not reported   |   |
| Purity of test substance  | 99.3 %   | Not reported but author verified from chemical supplier |
| Concentrations measured?  | Yes  |   |
| Measured is what % of nominal?  | 66-113%  |   |
| Toxicity values calculated based on nominal or measured concentrations? | Measured   |   |
| Chemical method documented?   | GC   |   |
| Concentration of carrier (if any) in test solutions                     | Acetone, <36 µg/L  |   |
| Concentration 1 Nom; Meas (µg/L)  | Not reported; 4-7 concentrations tested; dilution factor of 2              | 3 reps, 4-6/rep   |
| Control   | Negative: 0; 0<br>Solvent: 0; 0  |   |
| LC <sub>50</sub> (95% CI) (µg/L)  | >0.196   | Method: Probit  |
| EC <sub>50</sub> (95% CI) (µg/L)  | 0.0717 (0.0523-0.0906)   | Method: Probit  |

Notes: Points were not deducted for lack of water quality parameter reporting because water was prepared according to EPA 821-R-02-012 and measured during study (but not reported).

Solubility (S) of fipronil = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Organism life stage/size (5), Nominal concentrations (3), Measured concentrations (3), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100-15 =85

Acceptability: Standard method (5), Organisms randomized (1), Temperature variation (3), Random design (2), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1). Total: 100-14 =86

**Reliability score: mean(85,86)=85.5**

## Water Toxicity Data Summary

*Hyalella azteca*  
Fipronil sulfide  
MB45950

Weston DP and Lydy MJ. (2014) Toxicity of the insecticide fipronil and its degradates to benthic macroinvertebrates of urban streams. *Environmental science & technology*, 48(2), 1290-1297.

Relevance  
Score: 90  
Rating: R

Reliability  
Score: 85.5  
Rating: R

Relevance points taken off for: Standard method (10). 100-10=90

|  | <b>Weston 2014</b>                            | <b><i>H. azteca</i></b> |
|--|---|-------------------------|
| <b>Parameter</b>                             | <b>Value</b>                                  | <b>Comment</b>          |
| Test method cited                            | Not reported                                  |                         |
| Phylum/subphylum                             | Arthropoda                                    |                         |
| Class  | Crustacea                                     |                         |
| Order  | Malacostraca                                  |                         |
| Family                                       | Hyalellidae                                   |                         |
| Genus  | <i>Hyalella</i>                               |                         |
| Species                                      | <i>azteca</i>                                 |                         |
| Family native to North America?              | Yes   |                         |
| Age/size at start of test/growth phase       | Not reported                                  |                         |
| Source of organisms                          | University of California Berkeley lab culture |                         |
| Have organisms been exposed to contaminants? | No  |                         |
| Animals acclimated and disease-free?         | Yes   |                         |
| Animals randomized?                          | Not reported                                  |                         |
| Test vessels randomized?                     | Not reported                                  |                         |
| Test duration                                | 96 h  |                         |
| Data for multiple times?                     | Not reported                                  |                         |
| Effect 1                                     | Survival                                      |                         |
| Control response 1 (mean tests)              | 99 %  |                         |
| Effect 2                                     | Immobilization (ability to swim)              |                         |
| Control response 2                           | Not reported                                  |                         |
| Temperature                                  | 23 °C   |                         |
| Test type                                    | Static  |                         |
| Photoperiod/light intensity                  | 16l:8d; Not reported                          |                         |
| Dilution water                               | Milli-Q purified, deionized                   | *According to           |

|   | <b>Weston 2014</b>   | <b><i>H. azteca</i></b>                                 |
|---|--|---|
| <b>Parameter</b>  | <b>Value</b>   | <b>Comment</b>  |
|   | watermade moderately hard by addition of salts                         | EPA 821-R-02-012  |
| pH  | Not reported*  |   |
| Hardness  | Not reported*  |   |
| Alkalinity  | Not reported*  |   |
| Conductivity  | Not reported*  |   |
| Dissolved Oxygen  | Not reported*  |   |
| Feeding   | 1 mL: yeast/cerophyll/trout food on second day then 80% water replaced |   |
| Purity of test substance  | 99.0 %   | Not reported but author verified from chemical supplier |
| Concentrations measured?  | Yes  |   |
| Measured is what % of nominal?  | 66-113%  |   |
| Toxicity values calculated based on nominal or measured concentrations? | Measured   |   |
| Chemical method documented?   | GC   |   |
| Concentration of carrier (if any) in test solutions                     | Acetone, <36 µg/L  |   |
| Concentration 1 Nom; Meas (µg/L)  | Not reported; 4-7 concentrations tested; dilution factor of 2          | 3 reps, 10/rep  |
| Control   | Negative: 0; 0<br>Solvent: 0; 0  |   |
| LC <sub>50</sub> (95% CI) (µg/L)  | 1.356 (1.092-1.635)  | Method: Probit  |
| EC <sub>50</sub> (95% CI) (µg/L)  | 0.375 (0.325-0.433)  | Method: Probit  |

Notes: Two tests performed; lowest values included. Points were not deducted for lack of water quality parameter reporting because water was prepared according to EPA 821-R-02-012 and measured during study (but not reported).

Solubility value for fipronil sulfide (MB 45950) not available. Solubility (S) of fipronil parent compound (MB 46030) = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Organism life stage/size (5), Nominal concentrations (3), Measured concentrations (3), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100-15 =85

Acceptability: Standard method (5), Organisms randomized (1), Temperature variation (3), Random design (2), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1). Total: 100-14 =86

**Reliability score: mean(85,86)=85.5**

## Water Toxicity Data Summary

*Hyadella azteca*

Fipronil

MB46030

Weston DP and Lydy MJ. (2014) Toxicity of the insecticide fipronil and its degradates to benthic macroinvertebrates of urban streams. *Environmental science & technology*, 48(2), 1290-1297.

Relevance

Score: 90

Rating: R

Reliability

Score: 85.5

Rating: R

Relevance points taken off for: Standard method (10). 100-10=90

|  | <b>Weston 2014</b>                            | <b><i>H. azteca</i></b> |
|--|---|-------------------------|
| <b>Parameter</b>                             | <b>Value</b>                                  | <b>Comment</b>          |
| Test method cited                            | Not reported                                  |                         |
| Phylum/subphylum                             | Arthropoda                                    |                         |
| Class  | Crustacea                                     |                         |
| Order  | Malacostraca                                  |                         |
| Family                                       | Hyaellidae                                    |                         |
| Genus  | <i>Hyaella</i>                                |                         |
| Species                                      | <i>azteca</i>                                 |                         |
| Family native to North America?              | Yes   |                         |
| Age/size at start of test/growth phase       | Not reported                                  |                         |
| Source of organisms                          | University of California Berkeley lab culture |                         |
| Have organisms been exposed to contaminants? | No  |                         |
| Animals acclimated and disease-free?         | Yes   |                         |
| Animals randomized?                          | Not reported                                  |                         |
| Test vessels randomized?                     | Not reported                                  |                         |
| Test duration                                | 96 h  |                         |
| Data for multiple times?                     | Not reported                                  |                         |
| Effect 1                                     | Survival                                      |                         |
| Control response 1                           | 100 %   |                         |
| Effect 2                                     | Immobilization (ability to swim)              |                         |
| Control response 2                           | Not reported                                  |                         |
| Temperature                                  | 23 °C   |                         |
| Test type                                    | Static  |                         |
| Photoperiod/light intensity                  | 16l:8d; Not reported                          |                         |
| Dilution water                               | Milli-Q purified, deionized                   | *According to           |

|   | <b>Weston 2014</b>   | <b><i>H. azteca</i></b>                                 |
|---|--|---|
| <b>Parameter</b>  | <b>Value</b>   | <b>Comment</b>  |
|   | watermade moderately hard by addition of salts                         | EPA 821-R-02-012  |
| pH  | Not reported*  |   |
| Hardness  | Not reported*  |   |
| Alkalinity  | Not reported*  |   |
| Conductivity  | Not reported*  |   |
| Dissolved Oxygen  | Not reported*  |   |
| Feeding   | 1 mL: yeast/cerophyll/trout food on second day then 80% water replaced |   |
| Purity of test substance  | 99.50 %  | Not reported but author verified from chemical supplier |
| Concentrations measured?  | Yes  |   |
| Measured is what % of nominal?  | 66-113%  |   |
| Toxicity values calculated based on nominal or measured concentrations? | Measured   |   |
| Chemical method documented?   | GC   |   |
| Concentration of carrier (if any) in test solutions                     | Acetone, <36 µg/L  |   |
| Concentration 1 Nom; Meas (µg/L)  | Not reported; 4-7 concentrations tested; dilution factor of 2          | 3 reps, 10/rep  |
| Control   | Negative: 0; 0<br>Solvent: 0; 0  |   |
| LC <sub>50</sub> (95% CI) (µg/L)  | 1.593 (1.343-1.889)  | Method: Probit  |
| EC <sub>50</sub> (95% CI) (µg/L)  | 0.727 (0.648-0.816)  | Method: Probit  |

Notes: Two tests performed; lowest values included. Points were not deducted for lack of water quality parameter reporting because water was prepared according to EPA 821-R-02-012 and measured during study (but not reported).

Solubility (S) of fipronil = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Organism life stage/size (5), Nominal concentrations (3), Measured concentrations (3), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100-15 =85

Acceptability: Standard method (5), Organisms randomized (1), Temperature variation (3), Random design (2), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1). Total: 100-14 =86

**Reliability score: mean(85,86)=85.5**

## Water Toxicity Data Summary

*Helicopsyche* sp.  
Fipronil sulfide  
MB45950

Weston DP and Lydy MJ. (2014) Toxicity of the insecticide fipronil and its degradates to benthic macroinvertebrates of urban streams. *Environmental science & technology*, 48(2), 1290-1297.

Relevance  
Score: 90  
Rating: R

Reliability  
Score: 85.5  
Rating: R

Relevance points taken off for: Standard method (10). 100-10=90

|  | <b>Weston 2014</b>  | <b><i>Helicopsyche</i></b> |
|--|---|----------------------------|
| <b>Parameter</b>                             | <b>Value</b>  | <b>Comment</b>             |
| Test method cited                            | Not reported  |                            |
| Phylum/subphylum                             | Anthropoda  |                            |
| Class  | Insecta   |                            |
| Order  | Trichoptera   |                            |
| Family                                       | Helicopsychidae   |                            |
| Genus  | <i>Helicopsyche</i>   |                            |
| Species                                      | sp.   |                            |
| Family native to North America?              | Yes   |                            |
| Age/size at start of test/growth phase       | Not reported  |                            |
| Source of organisms                          | Urban waterbodies with minimal development in Northern California |                            |
| Have organisms been exposed to contaminants? | Not reported  |                            |
| Animals acclimated and disease-free?         | 24 h  |                            |
| Animals randomized?                          | Not reported  |                            |
| Test vessels randomized?                     | Not reported  |                            |
| Test duration                                | 96 h  |                            |
| Data for multiple times?                     | Not reported  |                            |
| Effect 1                                     | Survival  |                            |
| Control response 1                           | 100 %   |                            |
| Effect 2                                     | Immobilization (ability to swim)                                  |                            |
| Control response 2                           | Not reported  |                            |
| Temperature                                  | 13 °C   |                            |
| Test type                                    | Static  |                            |
| Photoperiod/light intensity                  | 16l:8d; Not reported  |                            |

|   | <b>Weston 2014</b>   | <b><i>Helicopsyche</i></b>                              |
|---|--|---|
| <b>Parameter</b>  | <b>Value</b>   | <b>Comment</b>  |
| Dilution water  | Milli-Q purified, deionized watermade moderately hard by addition of salts | *According to EPA 821-R-02-012                          |
| pH  | Not reported*  |   |
| Hardness  | Not reported*  |   |
| Alkalinity  | Not reported*  |   |
| Conductivity  | Not reported*  |   |
| Dissolved Oxygen  | Not reported*  |   |
| Feeding   | Not reported   |   |
| Purity of test substance  | 99.0 %   | Not reported but author verified from chemical supplier |
| Concentrations measured?  | Yes  |   |
| Measured is what % of nominal?  | 66-113%  |   |
| Toxicity values calculated based on nominal or measured concentrations? | Measured   |   |
| Chemical method documented?   | GC   |   |
| Concentration of carrier (if any) in test solutions                     | Acetone, <36 µg/L  |   |
| Concentration 1 Nom; Meas (µg/L)  | Not reported; 4-7 concentrations tested; dilution factor of 2              | 3 reps, 4-6/rep   |
| Control   | Negative: 0; 0<br>Solvent: 0; 0  |   |
| LC <sub>50</sub> (95% CI) (µg/L)  | >0.551   | Method: Probit  |
| EC <sub>50</sub> (95% CI) (µg/L)  | 0.177 (0.146-0.216)  | Method: Probit  |

Notes: Points were not deducted for lack of water quality parameter reporting because water was prepared according to EPA 821-R-02-012 and measured during study (but not reported).

Solubility value for fipronil sulfide (MB 45950) not available. Solubility (S) of fipronil parent compound (MB 46030) = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Organism life stage/size (5), Nominal concentrations (3), Measured concentrations (3), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100-15 =85

Acceptability: Standard method (5), Organisms randomized (1), Temperature variation (3), Random design (2), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1). Total: 100-14 =86

**Reliability score: mean(85,86)=85.5**

## Water Toxicity Data Summary

*Helicopsyche* sp.

Fipronil

MB46030

Weston DP and Lydy MJ. (2014) Toxicity of the insecticide fipronil and its degradates to benthic macroinvertebrates of urban streams. *Environmental science & technology*, 48(2), 1290-1297.

Relevance

Score: 90

Rating: R

Reliability

Score: 85.5

Rating: R

Relevance points taken off for: Standard method (10). 100-10=90

|  | <b>Weston 2014</b>  | <b><i>Helicopsyche</i></b> |
|--|---|----------------------------|
| <b>Parameter</b>                             | <b>Value</b>  | <b>Comment</b>             |
| Test method cited                            | Not reported  |                            |
| Phylum/subphylum                             | Anthropoda  |                            |
| Class  | Insecta   |                            |
| Order  | Trichoptera   |                            |
| Family                                       | Helicopsychidae   |                            |
| Genus  | <i>Helicopsyche</i>   |                            |
| Species                                      | sp.   |                            |
| Family native to North America?              | Yes   |                            |
| Age/size at start of test/growth phase       | Not reported  |                            |
| Source of organisms                          | Urban waterbodies with minimal development in Northern California |                            |
| Have organisms been exposed to contaminants? | Not reported  |                            |
| Animals acclimated and disease-free?         | 24 h  |                            |
| Animals randomized?                          | Not reported  |                            |
| Test vessels randomized?                     | Not reported  |                            |
| Test duration                                | 96 h  |                            |
| Data for multiple times?                     | Not reported  |                            |
| Effect 1                                     | Survival  |                            |
| Control response 1                           | 100 %   |                            |
| Effect 2                                     | Immobilization (ability to swim)                                  |                            |
| Control response 2                           | Not reported  |                            |
| Temperature                                  | 13 °C   |                            |
| Test type                                    | Static  |                            |
| Photoperiod/light intensity                  | 16l:8d; Not reported  |                            |

|   | <b>Weston 2014</b>   | <b><i>Helicopsyche</i></b>                              |
|---|--|---|
| <b>Parameter</b>  | <b>Value</b>   | <b>Comment</b>  |
| Dilution water  | Milli-Q purified, deionized watermade moderately hard by addition of salts | *According to EPA 821-R-02-012                          |
| pH  | Not reported*  |   |
| Hardness  | Not reported*  |   |
| Alkalinity  | Not reported*  |   |
| Conductivity  | Not reported*  |   |
| Dissolved Oxygen  | Not reported*  |   |
| Feeding   | Not reported   |   |
| Purity of test substance  | 99.50 %  | Not reported but author verified from chemical supplier |
| Concentrations measured?  | Yes  |   |
| Measured is what % of nominal?  | 66-113%  |   |
| Toxicity values calculated based on nominal or measured concentrations? | Measured   |   |
| Chemical method documented?   | GC   |   |
| Concentration of carrier (if any) in test solutions                     | Acetone, <36 µg/L  |   |
| Concentration 1 Nom; Meas (µg/L)  | Not reported; 4-7 concentrations tested; dilution factor of 2              | 3 reps, 4-6/rep   |
| Control   | Negative: 0; 0<br>Solvent: 0; 0  |   |
| LC <sub>50</sub> (95% CI) (µg/L)  | 0.267 (0.210-0.338)  | Method: Probit  |
| EC <sub>50</sub> (95% CI) (µg/L)  | >0.842   | Method: Probit  |

Notes: Points were not deducted for lack of water quality parameter reporting because water was prepared according to EPA 821-R-02-012 and measured during study (but not reported).

Solubility (S) of fipronil = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Organism life stage/size (5), Nominal concentrations (3), Measured concentrations (3), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100-15 =85

Acceptability: Standard method (5), Organisms randomized (1), Temperature variation (3), Random design (2), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1). Total: 100-14 =86

**Reliability score: mean(85,86)=85.5**

## Water Toxicity Data Summary

*Helicopsyche* sp.  
Fipronil sulfone  
MB46136

Weston DP and Lydy MJ. (2014) Toxicity of the insecticide fipronil and its degradates to benthic macroinvertebrates of urban streams. *Environmental science & technology*, 48(2), 1290-1297.

Relevance  
Score: 90  
Rating: R

Reliability  
Score: 85.5  
Rating: R

Relevance points taken off for: Standard method (10). 100-10=90

|  | <b>Weston 2014</b>  | <i>Helicopsyche</i> |
|--|---|---------------------|
| <b>Parameter</b>                             | <b>Value</b>  | <b>Comment</b>      |
| Test method cited                            | Not reported  |                     |
| Phylum/subphylum                             | Anthropoda  |                     |
| Class  | Insecta   |                     |
| Order  | Trichoptera   |                     |
| Family                                       | Helicopsychidae   |                     |
| Genus  | <i>Helicopsyche</i>   |                     |
| Species                                      | sp.   |                     |
| Family native to North America?              | Yes   |                     |
| Age/size at start of test/growth phase       | Not reported  |                     |
| Source of organisms                          | Urban waterbodies with minimal development in Northern California |                     |
| Have organisms been exposed to contaminants? | Not reported  |                     |
| Animals acclimated and disease-free?         | 24 h  |                     |
| Animals randomized?                          | Not reported  |                     |
| Test vessels randomized?                     | Not reported  |                     |
| Test duration                                | 96 h  |                     |
| Data for multiple times?                     | Not reported  |                     |
| Effect 1                                     | Survival  |                     |
| Control response 1                           | 100 %   |                     |
| Effect 2                                     | Immobilization (ability to swim)                                  |                     |
| Control response 2                           | Not reported  |                     |
| Temperature                                  | 13 °C   |                     |
| Test type                                    | Static  |                     |
| Photoperiod/light intensity                  | 16l:8d; Not reported  |                     |

|   | <b>Weston 2014</b>   | <b><i>Helicopsyche</i></b>                              |
|---|--|---|
| <b>Parameter</b>  | <b>Value</b>   | <b>Comment</b>  |
| Dilution water  | Milli-Q purified, deionized watermade moderately hard by addition of salts | *According to EPA 821-R-02-012                          |
| pH  | Not reported*  |   |
| Hardness  | Not reported*  |   |
| Alkalinity  | Not reported*  |   |
| Conductivity  | Not reported*  |   |
| Dissolved Oxygen  | Not reported*  |   |
| Feeding   | Not reported   |   |
| Purity of test substance  | 99.3 %   | Not reported but author verified from chemical supplier |
| Concentrations measured?  | Yes  |   |
| Measured is what % of nominal?  | 66-113%  |   |
| Toxicity values calculated based on nominal or measured concentrations? | Measured   |   |
| Chemical method documented?   | GC   |   |
| Concentration of carrier (if any) in test solutions                     | Acetone, <36 µg/L  |   |
| Concentration 1 Nom; Meas (µg/L)  | Not reported; 4-7 concentrations tested; dilution factor of 2              | 3 reps, 4-6/rep   |
| Control   | Negative: 0; 0<br>Solvent: 0; 0  |   |
| LC <sub>50</sub> (95% CI) (µg/L)  | >0.626   | Method: Probit  |
| EC <sub>50</sub> (95% CI) (µg/L)  | 0.0738 (0.0386-0.140)  | Method: Probit  |

Notes: Points were not deducted for lack of water quality parameter reporting because water was prepared according to EPA 821-R-02-012 and measured during study (but not reported).

Solubility (S) value for fipronil sulfone (MB 46136) = 160 µg/L, 2S = 320 µg/L.

Reliability points taken off for:

Documentation: Organism life stage/size (5), Nominal concentrations (3), Measured concentrations (3), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100-15 =85

Acceptability: Standard method (5), Organisms randomized (1), Temperature variation (3), Random design (2), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1). Total: 100-14 =86

**Reliability score: mean(85,86)=85.5**

## Water Toxicity Data Summary

*Hexagenia* sp.

Fipronil

MB46030

Putt AE. (2003a) Fipronil-Acute toxicity to mayfly nymphs (*Hexagenia* sp.) under static-renewal conditions. Springborn Smithers Laboratories, Wareham, Massachusetts. Laboratory study number 986.6160. Submitted to BSF, Research Triangle, North Carolina. USEPA MRID 46329902.

Relevance

Score: 100

Rating: R

Reliability

Score: 96.5

Rating: R

Relevance points taken off for: none.

|  | <b>Putt 2003</b>                          | <b><i>Hexagenia</i> sp.</b> |
|--|---|-----------------------------|
| <b>Parameter</b>                             | <b>Value</b>                              | <b>Comment</b>              |
| Test method cited                            | ASTM Guideline E-729                      |                             |
| Phylum/subphylum                             | Anthropoda                                |                             |
| Class  | Insecta                                   |                             |
| Order  | Ephemeroptera                             |                             |
| Family                                       | Ephemeridae                               |                             |
| Genus  | <i>Hexagenia</i>                          |                             |
| Species                                      | Not reported                              |                             |
| Family native to North America?              | Yes                                       |                             |
| Age/size at start of test/growth phase       | 60 d<br>6.7 mm                            |                             |
| Source of organisms                          | University of Windsor,<br>Ontario, Canada |                             |
| Have organisms been exposed to contaminants? | No  |                             |
| Animals acclimated and disease-free?         | Yes                                       |                             |
| Animals randomized?                          | Yes                                       |                             |
| Test vessels randomized?                     | Not reported                              |                             |
| Test duration                                | 96 h                                      |                             |
| Data for multiple times?                     | 24, 48, 72, 96 h                          |                             |
| Effect 1                                     | Survival                                  |                             |
| Control response 1                           | 100 %                                     |                             |
| Temperature                                  | 22 ± 1 °C                                 |                             |
| Test type                                    | Static renewal                            | Renewal at 48 h             |
| Photoperiod/light intensity                  | 16l:8d/60-77 footcandles                  |                             |
| Dilution water                               | Well water                                |                             |
| pH   | 8.1                                       |                             |

|   | <b>Putt 2003</b>                | <b><i>Hexagenia sp.</i></b>                                  |
|---|---------------------------------|--|
| <b>Parameter</b>  | <b>Value</b>                    | <b>Comment</b>   |
| Hardness  | 170 mg/L CaCO <sub>3</sub>      |  |
| Alkalinity  | 120 mg/L CaCO <sub>3</sub>      |  |
| Conductivity  | 500 µmhos/com                   |  |
| Dissolved Oxygen  | 7.4-9.1 mg/L                    | 85-104 %   |
| Feeding   | Not reported                    |  |
| Purity of test substance  | 99.7 %                          |  |
| Concentrations measured?  | Yes                             |  |
| Measured is what % of nominal?  | 94-110%                         |  |
| Toxicity values calculated based on nominal or measured concentrations? | Measured                        |  |
| Chemical method documented?   | GC/ECD                          |  |
| Concentration of carrier (if any) in test solutions                     | Acetone, 0.40 mL/L              |  |
| Concentration 1 Nom; Meas (µg/L)  | 0.063; 0.059                    | Replicates not reported, 5/rep                               |
| Concentration 2 Nom; Meas (µg/L)  | 0.13; 0.14                      |  |
| Concentration 3 Nom; Meas (µg/L)  | 0.25; 0.24                      |  |
| Concentration 4 Nom; Meas (µg/L)  | 0.50; 0.52                      |  |
| Concentration 5 Nom; Meas (µg/L)  | 1.0; 1.1                        |  |
| Control   | Negative: 0; 0<br>Solvent: 0; 0 |  |
| LC <sub>50</sub> (95% CI) (µg/L)  | 0.44 (0.39-0.49)                | Method: Log-log analysis                                     |
| NOEC (µg/L)   | 0.14                            | Method: Not reported<br>p: Not reported<br>MSD: Not reported |
| LOEC(µg/L)  | 0.24                            | Not reported; See Table 3                                    |
| MATC  | 0.18                            |  |
| % control at NOEC   | 100 %                           |  |
| % control at LOEC   | 80 %                            |  |

Notes:

Solubility (S) of fipronil = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Minimum significant difference (2). Total: 100- 2=98

Acceptability: Random design (2), Adequate replication (2), Minimum significant difference (1).

Total: 100- 5=95

**Reliability score: mean(98, 95)=96.5**

## Water Toxicity Data Summary

*Hexagenia* sp.

Fipronil

MB46030

Weston DP and Lydy MJ. (2014) Toxicity of the insecticide fipronil and its degradates to benthic macroinvertebrates of urban streams. *Environmental science & technology*, 48(2), 1290-1297.

Relevance

Score: 90

Rating: R

Reliability

Score: 85.5

Rating: R

Relevance points taken off for: Standard method (10). 100-10=90

|  | <b>Weston 2014</b>                                 | <b><i>Hexagenia</i> sp.</b> |
|--|--|-----------------------------|
| <b>Parameter</b>                             | <b>Value</b>                                       | <b>Comment</b>              |
| Test method cited                            | Not reported                                       |                             |
| Phylum/subphylum                             | Anthropoda   |                             |
| Class  | Insecta  |                             |
| Order  | Ephemeroptera                                      |                             |
| Family                                       | Ephemeridae  |                             |
| Genus  | <i>Hexagenia</i>                                   |                             |
| Species                                      | Not reported                                       |                             |
| Family native to North America?              | Yes  |                             |
| Age/size at start of test/growth phase       | Not reported                                       |                             |
| Source of organisms                          | Aquatic Research Organisms, Hampton, New Hampshire |                             |
| Have organisms been exposed to contaminants? | No   |                             |
| Animals acclimated and disease-free?         | Yes  |                             |
| Animals randomized?                          | Not reported                                       |                             |
| Test vessels randomized?                     | Not reported                                       |                             |
| Test duration                                | 96 h   |                             |
| Data for multiple times?                     | Not reported                                       |                             |
| Effect 1                                     | Survival   |                             |
| Control response 1                           | 93 %   |                             |
| Effect 2                                     | Immobilization (ability to swim)                   |                             |
| Control response 2                           | Not reported                                       |                             |
| Temperature                                  | 18 °C  |                             |
| Test type                                    | Static   |                             |
| Photoperiod/light intensity                  | 16l:8d; Not reported                               |                             |

|   | <b>Weston 2014</b>   | <b><i>Hexagenia sp.</i></b>                             |
|---|--|---|
| <b>Parameter</b>  | <b>Value</b>   | <b>Comment</b>  |
| Dilution water  | Milli-Q purified, deionized watermade moderately hard by addition of salts | *According to EPA 821-R-02-012                          |
| pH  | Not reported*  |   |
| Hardness  | Not reported*  |   |
| Alkalinity  | Not reported*  |   |
| Conductivity  | Not reported*  |   |
| Dissolved Oxygen  | Not reported*  |   |
| Feeding   | Not reported   |   |
| Purity of test substance  | 99.50 %  | Not reported but author verified from chemical supplier |
| Concentrations measured?  | Yes  |   |
| Measured is what % of nominal?  | 66-113%  |   |
| Toxicity values calculated based on nominal or measured concentrations? | Measured   |   |
| Chemical method documented?   | GC   |   |
| Concentration of carrier (if any) in test solutions                     | Acetone, <36 µg/L  |   |
| Concentration 1 Nom; Meas (µg/L)  | Not reported; 4-7 concentrations tested; dilution factor of 2              | 3 reps, 10/rep  |
| Control   | Negative: 0; 0<br>Solvent: 0; 0  |   |
| LC <sub>50</sub> (95% CI) (µg/L)  | 0.480 (0.348-0.603)  | Method: Probit  |
| EC <sub>50</sub> (95% CI) (µg/L)  | 1.231 (0.769-01.667)   | Method: Probit  |

Notes: Points were not deducted for lack of water quality parameter reporting because water was prepared according to EPA 821-R-02-012 and measured during study (but not reported).

Solubility (S) of fipronil = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Organism life stage/size (5), Nominal concentrations (3), Measured concentrations (3), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100-15 =85

Acceptability: Standard method (5), Organisms randomized (1), Temperature variation (3), Random design (2), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1). Total: 100-14 =86

**Reliability score: mean(85,86)=85.5**

## Water Toxicity Data Summary

*Hexagenia* sp.  
Fipronil sulfone  
MB46136

Weston DP and Lydy MJ. (2014) Toxicity of the insecticide fipronil and its degradates to benthic macroinvertebrates of urban streams. *Environmental science & technology*, 48(2), 1290-1297.

Relevance  
Score: 90  
Rating: R

Reliability  
Score: 85.5  
Rating: R

Relevance points taken off for: Standard method (10). 100-10=90

|  | <b>Weston 2014</b>                                 | <b><i>Hexagenia</i> sp.</b> |
|--|--|-----------------------------|
| <b>Parameter</b>                             | <b>Value</b>                                       | <b>Comment</b>              |
| Test method cited                            | Not reported                                       |                             |
| Phylum/subphylum                             | Anthropoda   |                             |
| Class  | Insecta  |                             |
| Order  | Ephemeroptera                                      |                             |
| Family                                       | Ephemeridae  |                             |
| Genus  | <i>Hexagenia</i>                                   |                             |
| Species                                      | Not reported                                       |                             |
| Family native to North America?              | Yes  |                             |
| Age/size at start of test/growth phase       | Not reported                                       |                             |
| Source of organisms                          | Aquatic Research Organisms, Hampton, New Hampshire |                             |
| Have organisms been exposed to contaminants? | No   |                             |
| Animals acclimated and disease-free?         | Yes  |                             |
| Animals randomized?                          | Not reported                                       |                             |
| Test vessels randomized?                     | Not reported                                       |                             |
| Test duration                                | 96 h   |                             |
| Data for multiple times?                     | Not reported                                       |                             |
| Effect 1                                     | Survival   |                             |
| Control response 1                           | 93 %   |                             |
| Effect 2                                     | Immobilization (ability to swim)                   |                             |
| Control response 2                           | Not reported                                       |                             |
| Temperature                                  | 18 °C  |                             |
| Test type                                    | Static   |                             |
| Photoperiod/light intensity                  | 16l:8d; Not reported                               |                             |

|   | <b>Weston 2014</b>   | <b><i>Hexagenia</i> sp.</b>                             |
|---|--|---|
| <b>Parameter</b>  | <b>Value</b>   | <b>Comment</b>  |
| Dilution water  | Milli-Q purified, deionized watermade moderately hard by addition of salts | *According to EPA 821-R-02-012                          |
| pH  | Not reported*  |   |
| Hardness  | Not reported*  |   |
| Alkalinity  | Not reported*  |   |
| Conductivity  | Not reported*  |   |
| Dissolved Oxygen  | Not reported*  |   |
| Feeding   | Not reported   |   |
| Purity of test substance  | 99.3 %   | Not reported but author verified from chemical supplier |
| Concentrations measured?  | Yes  |   |
| Measured is what % of nominal?  | 66-113%  |   |
| Toxicity values calculated based on nominal or measured concentrations? | Measured   |   |
| Chemical method documented?   | GC   |   |
| Concentration of carrier (if any) in test solutions                     | Acetone, <36 µg/L  |   |
| Concentration 1 Nom; Meas (µg/L)  | Not reported; 4-7 concentrations tested; dilution factor of 2              | 3 reps, 10/rep  |
| Control   | Negative: 0; 0<br>Solvent: 0; 0  |   |
| LC <sub>50</sub> (95% CI) (µg/L)  | 0.257 (0.109-0.362)  | Method: Probit  |
| EC <sub>50</sub> (95% CI) (µg/L)  | 0.163 (0.051-0.223)  | Method: Probit  |

Notes: Points were not deducted for lack of water quality parameter reporting because water was prepared according to EPA 821-R-02-012 and measured during study (but not reported).

Solubility (S) value for fipronil sulfone (MB 46136) = 160 µg/L, 2S = 320 µg/L.

Reliability points taken off for:

Documentation: Organism life stage/size (5), Nominal concentrations (3), Measured concentrations (3), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100-15 =85

Acceptability: Standard method (5), Organisms randomized (1), Temperature variation (3), Random design (2), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1). Total: 100-14 =86

**Reliability score: mean(85,86)=85.5**

## Water Toxicity Data Summary

*Hydropsyche* sp.

Fipronil

MB46030

Weston DP and Lydy MJ. (2014) Toxicity of the insecticide fipronil and its degradates to benthic macroinvertebrates of urban streams. *Environmental science & technology*, 48(2), 1290-1297.

Relevance

Score: 90

Rating: R

Reliability

Score: 85.5

Rating: R

Relevance points taken off for: Standard method (10). 100-10=90

|  | <b>Weston 2014</b>  | <i>Hydropsyche</i> |
|--|---|--------------------|
| <b>Parameter</b>                             | <b>Value</b>  | <b>Comment</b>     |
| Test method cited                            | Not reported  |                    |
| Phylum/subphylum                             | Anthropoda  |                    |
| Class  | Insecta   |                    |
| Order  | Trichoptera   |                    |
| Family                                       | Hydropsychidae  |                    |
| Genus  | <i>Hydropsyche</i>  |                    |
| Species                                      | sp.   |                    |
| Family native to North America?              | Yes   |                    |
| Age/size at start of test/growth phase       | Not reported  |                    |
| Source of organisms                          | Urban waterbodies with minimal development in Northern California |                    |
| Have organisms been exposed to contaminants? | Not reported  |                    |
| Animals acclimated and disease-free?         | 24 h  |                    |
| Animals randomized?                          | Not reported  |                    |
| Test vessels randomized?                     | Not reported  |                    |
| Test duration                                | 96 h  |                    |
| Data for multiple times?                     | Not reported  |                    |
| Effect 1                                     | Survival  |                    |
| Control response 1                           | 94 %  |                    |
| Effect 2                                     | Immobilization (ability to thrash when prodded)                   |                    |
| Control response 2                           | Not reported  |                    |
| Temperature                                  | 12 °C   |                    |
| Test type                                    | Static  |                    |
| Photoperiod/light intensity                  | 16l:8d; Not reported  |                    |

|   | <b>Weston 2014</b>   | <b><i>Hydropsyche</i></b>                               |
|---|--|---|
| <b>Parameter</b>  | <b>Value</b>   | <b>Comment</b>  |
| Dilution water  | Milli-Q purified, deionized watermade moderately hard by addition of salts | *According to EPA 821-R-02-012                          |
| pH  | Not reported*  |   |
| Hardness  | Not reported*  |   |
| Alkalinity  | Not reported*  |   |
| Conductivity  | Not reported*  |   |
| Dissolved Oxygen  | Not reported*  |   |
| Feeding   | Not reported   |   |
| Purity of test substance  | 99.50 %  | Not reported but author verified from chemical supplier |
| Concentrations measured?  | Yes  |   |
| Measured is what % of nominal?  | 66-113%  |   |
| Toxicity values calculated based on nominal or measured concentrations? | Measured   |   |
| Chemical method documented?   | GC   |   |
| Concentration of carrier (if any) in test solutions                     | Acetone, <36 µg/L  |   |
| Concentration 1 Nom; Meas (µg/L)  | Not reported; 4-7 concentrations tested; dilution factor of 2              | 3 reps, 4-6/rep   |
| Control   | Negative: 0; 0<br>Solvent: 0; 0  |   |
| LC <sub>50</sub> (95% CI) (µg/L)  | 0.602 (0.417-0.788)  | Method: Probit  |
| EC <sub>50</sub> (95% CI) (µg/L)  | 2.107 (1.218-2.668)  | Method: Probit  |

Notes: Points were not deducted for lack of water quality parameter reporting because water was prepared according to EPA 821-R-02-012 and measured during study (but not reported).

Solubility (S) of fipronil = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Organism life stage/size (5), Nominal concentrations (3), Measured concentrations (3), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100-15 =85

Acceptability: Standard method (5), Organisms randomized (1), Temperature variation (3), Random design (2), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1). Total: 100-14 =86

**Reliability score: mean(85,86)=85.5**

## Water Toxicity Data Summary

*Hydropsyche* sp.  
Fipronil sulfone  
MB46136

Weston DP and Lydy MJ. (2014) Toxicity of the insecticide fipronil and its degradates to benthic macroinvertebrates of urban streams. *Environmental science & technology*, 48(2), 1290-1297.

Relevance  
Score: 90  
Rating: R

Reliability  
Score: 85.5  
Rating: R

Relevance points taken off for: Standard method (10). 100-10=90

|  | <b>Weston 2014</b>  | <i>Hydropsyche</i> |
|--|---|--------------------|
| <b>Parameter</b>                             | <b>Value</b>  | <b>Comment</b>     |
| Test method cited                            | Not reported  |                    |
| Phylum/subphylum                             | Anthropoda  |                    |
| Class  | Insecta   |                    |
| Order  | Trichoptera   |                    |
| Family                                       | Hydropsychidae  |                    |
| Genus  | <i>Hydropsyche</i>  |                    |
| Species                                      | sp.   |                    |
| Family native to North America?              | Yes   |                    |
| Age/size at start of test/growth phase       | Not reported  |                    |
| Source of organisms                          | Urban waterbodies with minimal development in Northern California |                    |
| Have organisms been exposed to contaminants? | Not reported  |                    |
| Animals acclimated and disease-free?         | 24 h  |                    |
| Animals randomized?                          | Not reported  |                    |
| Test vessels randomized?                     | Not reported  |                    |
| Test duration                                | 96 h  |                    |
| Data for multiple times?                     | Not reported  |                    |
| Effect 1                                     | Survival  |                    |
| Control response 1                           | 75 %  |                    |
| Effect 2                                     | Immobilization (ability to thrash when prodded)                   |                    |
| Control response 2                           | Not reported  |                    |
| Temperature                                  | 12 °C   |                    |
| Test type                                    | Static  |                    |
| Photoperiod/light intensity                  | 16l:8d; Not reported  |                    |

|   | <b>Weston 2014</b>   | <b><i>Hydropsyche</i></b>                               |
|---|--|---|
| <b>Parameter</b>  | <b>Value</b>   | <b>Comment</b>  |
| Dilution water  | Milli-Q purified, deionized watermade moderately hard by addition of salts | *According to EPA 821-R-02-012                          |
| pH  | Not reported*  |   |
| Hardness  | Not reported*  |   |
| Alkalinity  | Not reported*  |   |
| Conductivity  | Not reported*  |   |
| Dissolved Oxygen  | Not reported*  |   |
| Feeding   | Not reported   |   |
| Purity of test substance  | 99.3 %   | Not reported but author verified from chemical supplier |
| Concentrations measured?  | Yes  |   |
| Measured is what % of nominal?  | 66-113%  |   |
| Toxicity values calculated based on nominal or measured concentrations? | Measured   |   |
| Chemical method documented?   | GC   |   |
| Concentration of carrier (if any) in test solutions                     | Acetone, <36 µg/L  |   |
| Concentration 1 Nom; Meas (µg/L)  | Not reported; 4-7 concentrations tested; dilution factor of 2              | 3 reps, 4-6/rep   |
| Control   | Negative: 0; 0<br>Solvent: 0; 0  |   |
| LC <sub>50</sub> (95% CI) (µg/L)  | >824   | Method: Probit  |
| EC <sub>50</sub> (95% CI) (µg/L)  | 0.0729 (0.0565-0.0940)   | Method: Probit  |

Notes: Points were not deducted for lack of water quality parameter reporting because water was prepared according to EPA 821-R-02-012 and measured during study (but not reported).

Solubility (S) value for fipronil sulfone (MB 46136) = 160 µg/L, 2S = 320 µg/L.

Reliability points taken off for:

Documentation: Organism life stage/size (5), Nominal concentrations (3), Measured concentrations (3), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100-15 =85

Acceptability: Standard method (5), Organisms randomized (1), Temperature variation (3), Random design (2), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1). Total: 100-14 =86

**Reliability score: mean(85,86)=85.5**

## Water Toxicity Data Summary

*Ictalurus punctatus*

Fipronil

MB 46030

Dionne E. (1997) Fipronil technical-acute toxicity to channel catfish (*Ictalurus punctatus*) under flow-through conditions. Springborn Laboratories, Inc., Wareham, Massachusetts. Laboratory study number 10566.1096.6408.107. Submitted to Rhone-Poulenc Ag Company, Research Triangle, North Carolina. USEPA MRID 44299401. CA DPR 157281.

Relevance

Score: 100

Rating: R

Reliability

Score: 92.5

Rating: R

Relevance points taken off for: none.

|  | <b>Dionne 1997</b>                        | <b><i>I. punctatus</i></b> |
|--|---|----------------------------|
| <b>Parameter</b>                             | <b>Value</b>                              | <b>Comment</b>             |
| Test method cited                            | FIFRA 72-1                                |                            |
| Phylum/subphylum                             | Chordata                                  |                            |
| Class  | Actinopterygii                            |                            |
| Order  | Siluriformes                              |                            |
| Family                                       | Ictaluridae                               |                            |
| Genus  | <i>Ictalurus</i>                          |                            |
| Species                                      | <i>Punctatus</i>                          |                            |
| Family native to North America?              | Yes                                       |                            |
| Age/size at start of test/growth phase       | 1.7 g<br>59 mm                            |                            |
| Source of organisms                          | Osage Catfisheries, Osage Beach, Missouri |                            |
| Have organisms been exposed to contaminants? | No  |                            |
| Animals acclimated and disease-free?         | 14 d                                      |                            |
| Animals randomized?                          | Yes                                       |                            |
| Test vessels randomized?                     | Not reported                              |                            |
| Test duration                                | 96 h                                      |                            |
| Data for multiple times?                     | 24, 48, 72, 96 h                          |                            |
| Effect 1                                     | Survival                                  |                            |
| Control response 1                           | 100 %                                     |                            |
| Temperature                                  | 22.5 ± 0.5 °C                             |                            |
| Test type                                    | Flow through                              |                            |
| Photoperiod/light intensity                  | 16l:8d/20-90 footcandles                  |                            |
| Dilution water                               | Well water                                |                            |
| pH   | 7.2-7.4                                   |                            |

|   | <b>Dionne 1997</b>                                   | <b><i>I. punctatus</i></b>                      |
|---|--|---|
| <b>Parameter</b>  | <b>Value</b>   | <b>Comment</b>                                  |
| Hardness  | 38 mg/L CaCO <sub>3</sub>                            |   |
| Alkalinity  | 26-28 mg/L CaCO <sub>3</sub>                         |   |
| Conductivity  | 130-160 µmhos/cm                                     |   |
| Dissolved Oxygen  | 6.2-7.6 mg/L   | 72-88 %   |
| Feeding   | Not fed  |   |
| Purity of test substance  | 97.8 %   |   |
| Concentrations measured?  | Yes  |   |
| Measured is what % of nominal?  | 80-95 %  |   |
| Toxicity values calculated based on nominal or measured concentrations? | Measured   |   |
| Chemical method documented?   | HPLC   |   |
| Concentration of carrier (if any) in test solutions                     | Acetone, 510 mL/L                                    |   |
| Concentration 1 Nom; Meas (µg/L)  | 94; 89   | 2 reps, 10/rep                                  |
| Concentration 2 Nom; Meas (µg/L)  | 190; 170   |   |
| Concentration 3 Nom; Meas (µg/L)  | 380; 320   |   |
| Concentration 4 Nom; Meas (µg/L)  | 750; 610   |   |
| Concentration 5 Nom; Meas (µg/L)  | 1500; 1200   |   |
| Control   | Negative: 0; 0<br>Solvent: 0; 0                      |   |
| LC <sub>50</sub> (95% CI) (µg/L)  | 560 (320-1200)                                       | Method: Nonlinear interpolation                 |
| NOEC  | 320  | Method: Not reported<br>p: Not reported<br>MSD: |
| LOEC  | 610  | Not reported; See Table 4                       |
| MATC (GeoMean NOEC, LOEC)   | 442  |   |
| % control at NOEC   | All times: 100 %                                     |   |
| % control at LOEC   | 24 h: 70 %<br>48 h: 40 %<br>72 h: 40 %<br>96 h: 40 % |   |

Notes: Solubility (S) of fipronil = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Statistical significance (2), Significance level (2), Minimum significant difference (2). Total: 100- 6=94

Acceptability: Carrier solvent (4), Random design (2), Adequate replication (2), Minimum significant difference (1). Total:  $100-9=91$

**Reliability score:  $\text{mean}(94, 91)=92.5$**

## Water Toxicity Data Summary

*Isoperla quinquepunctata*

Fipronil sulfide

MB45950

Weston DP and Lydy MJ. (2014) Toxicity of the insecticide fipronil and its degradates to benthic macroinvertebrates of urban streams. *Environmental science & technology*, 48(2), 1290-1297.

Relevance

Score: 90

Rating: R

Reliability

Score: 85.5

Rating: R

Relevance points taken off for: Standard method (10). 100-10=90

|  | <b>Weston 2014</b>  | <b><i>I. quinquepunctata</i></b> |
|--|---|----------------------------------|
| <b>Parameter</b>                             | <b>Value</b>  | <b>Comment</b>                   |
| Test method cited                            | Not reported  |                                  |
| Phylum/subphylum                             | Anthropoda  |                                  |
| Class  | Insecta   |                                  |
| Order  | Plecoptera  |                                  |
| Family                                       | Perlodidae  |                                  |
| Genus  | <i>Isoperla</i>   |                                  |
| Species                                      | <i>quinquepunctata</i>  |                                  |
| Family native to North America?              | Yes   |                                  |
| Age/size at start of test/growth phase       | Not reported  |                                  |
| Source of organisms                          | Urban waterbodies with minimal development in Northern California |                                  |
| Have organisms been exposed to contaminants? | Not reported  |                                  |
| Animals acclimated and disease-free?         | 24 h  |                                  |
| Animals randomized?                          | Not reported  |                                  |
| Test vessels randomized?                     | Not reported  |                                  |
| Test duration                                | 96 h  |                                  |
| Data for multiple times?                     | Not reported  |                                  |
| Effect 1                                     | Survival  |                                  |
| Control response 1                           | 100 %   |                                  |
| Effect 2                                     | Immobilization (ability to cling)                                 |                                  |
| Control response 2                           | Not reported  |                                  |
| Temperature                                  | 13 °C   |                                  |
| Test type                                    | Static  |                                  |
| Photoperiod/light intensity                  | 16l:8d; Not reported  |                                  |

|   | <b>Weston 2014</b>   | <b><i>I. quinquepunctata</i></b>                        |
|---|--|---|
| <b>Parameter</b>  | <b>Value</b>   | <b>Comment</b>  |
| Dilution water  | Milli-Q purified, deionized watermade moderately hard by addition of salts | *According to EPA 821-R-02-012                          |
| pH  | Not reported*  |   |
| Hardness  | Not reported*  |   |
| Alkalinity  | Not reported*  |   |
| Conductivity  | Not reported*  |   |
| Dissolved Oxygen  | Not reported*  |   |
| Feeding   | Not reported   |   |
| Purity of test substance  | 99.0 %   | Not reported but author verified from chemical supplier |
| Concentrations measured?  | Yes  |   |
| Measured is what % of nominal?  | 66-113%  |   |
| Toxicity values calculated based on nominal or measured concentrations? | Measured   |   |
| Chemical method documented?   | GC   |   |
| Concentration of carrier (if any) in test solutions                     | Acetone, <36 µg/L  |   |
| Concentration 1 Nom; Meas (µg/L)  | Not reported; 4-7 concentrations tested; dilution factor of 2              | 3 reps, 4-6/rep   |
| Control   | Negative: 0; 0<br>Solvent: 0; 0  |   |
| LC <sub>50</sub> (95% CI) (µg/L)  | 0.0945 (0.0668-0.175)  | Method: Probit  |
| EC <sub>50</sub> (95% CI) (µg/L)  | 0.0422 (0.0371-0.0474)   | Method: Probit  |

Notes: Points were not deducted for lack of water quality parameter reporting because water was prepared according to EPA 821-R-02-012 and measured during study (but not reported).

Solubility value for fipronil sulfide (MB 45950) not available. Solubility (S) of fipronil parent compound (MB 46030) = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Organism life stage/size (5), Nominal concentrations (3), Measured concentrations (3), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100-15 =85

Acceptability: Standard method (5), Organisms randomized (1), Temperature variation (3), Random design (2), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1). Total: 100-14 =86

**Reliability score: mean(85,86)=85.5**

## Water Toxicity Data Summary

*Isoperla quinquepunctata*

Fipronil

MB46030

Weston DP and Lydy MJ. (2014) Toxicity of the insecticide fipronil and its degradates to benthic macroinvertebrates of urban streams. *Environmental science & technology*, 48(2), 1290-1297.

Relevance

Score: 90

Rating: R

Reliability

Score: 85.5

Rating: R

Relevance points taken off for: Standard method (10). 100-10=90

|  | <b>Weston 2014</b>  | <b><i>I. quinquepunctata</i></b> |
|--|---|----------------------------------|
| <b>Parameter</b>                             | <b>Value</b>  | <b>Comment</b>                   |
| Test method cited                            | Not reported  |                                  |
| Phylum/subphylum                             | Anthropoda  |                                  |
| Class  | Insecta   |                                  |
| Order  | Plecoptera  |                                  |
| Family                                       | Perlodidae  |                                  |
| Genus  | <i>Isoperla</i>   |                                  |
| Species                                      | <i>quinquepunctata</i>  |                                  |
| Family native to North America?              | Yes   |                                  |
| Age/size at start of test/growth phase       | Not reported  |                                  |
| Source of organisms                          | Urban waterbodies with minimal development in Northern California |                                  |
| Have organisms been exposed to contaminants? | Not reported  |                                  |
| Animals acclimated and disease-free?         | 24 h  |                                  |
| Animals randomized?                          | Not reported  |                                  |
| Test vessels randomized?                     | Not reported  |                                  |
| Test duration                                | 96 h  |                                  |
| Data for multiple times?                     | Not reported  |                                  |
| Effect 1                                     | Survival  |                                  |
| Control response 1                           | 69 %  |                                  |
| Effect 2                                     | Immobilization (ability to cling)                                 |                                  |
| Control response 2                           | Not reported  |                                  |
| Temperature                                  | 13 °C   |                                  |
| Test type                                    | Static  |                                  |
| Photoperiod/light intensity                  | 16l:8d; Not reported  |                                  |

|   | <b>Weston 2014</b>   | <b><i>I. quinquepunctata</i></b>                        |
|---|--|---|
| <b>Parameter</b>  | <b>Value</b>   | <b>Comment</b>  |
| Dilution water  | Milli-Q purified, deionized watermade moderately hard by addition of salts | *According to EPA 821-R-02-012                          |
| pH  | Not reported*  |   |
| Hardness  | Not reported*  |   |
| Alkalinity  | Not reported*  |   |
| Conductivity  | Not reported*  |   |
| Dissolved Oxygen  | Not reported*  |   |
| Feeding   | Not reported   |   |
| Purity of test substance  | 99.50 %  | Not reported but author verified from chemical supplier |
| Concentrations measured?  | Yes  |   |
| Measured is what % of nominal?  | 66-113%  |   |
| Toxicity values calculated based on nominal or measured concentrations? | Measured   |   |
| Chemical method documented?   | GC   |   |
| Concentration of carrier (if any) in test solutions                     | Acetone, <36 µg/L  |   |
| Concentration 1 Nom; Meas (µg/L)  | Not reported; 4-7 concentrations tested; dilution factor of 2              | 3 reps, 4-6/rep   |
| Control   | Negative: 0; 0<br>Solvent: 0; 0  |   |
| LC <sub>50</sub> (95% CI) (µg/L)  | 0.101 (0.0846-0.119)   | Method: Probit  |
| EC <sub>50</sub> (95% CI) (µg/L)  | 0.113 (0.0942-0.135)   | Method: Probit  |

Notes: Points were not deducted for lack of water quality parameter reporting because water was prepared according to EPA 821-R-02-012 and measured during study (but not reported).

Solubility (S) of fipronil = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Organism life stage/size (5), Nominal concentrations (3), Measured concentrations (3), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100-15 =85

Acceptability: Standard method (5), Organisms randomized (1), Temperature variation (3), Random design (2), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1). Total: 100-14 =86

**Reliability score: mean(85,86)=85.5**

## Water Toxicity Data Summary

*Isoperla quinquepunctata*

Fipronil sulfone

MB46136

Weston DP and Lydy MJ. (2014) Toxicity of the insecticide fipronil and its degradates to benthic macroinvertebrates of urban streams. *Environmental science & technology*, 48(2), 1290-1297.

Relevance

Score: 90

Rating: R

Reliability

Score: 85.5

Rating: R

Relevance points taken off for: Standard method (10). 100-10=90

|  | <b>Weston 2014</b>  | <b><i>I. quinquepunctata</i></b> |
|--|---|----------------------------------|
| <b>Parameter</b>                             | <b>Value</b>  | <b>Comment</b>                   |
| Test method cited                            | Not reported  |                                  |
| Phylum/subphylum                             | Anthropoda  |                                  |
| Class  | Insecta   |                                  |
| Order  | Plecoptera  |                                  |
| Family                                       | Perlodidae  |                                  |
| Genus  | <i>Isoperla</i>   |                                  |
| Species                                      | <i>quinquepunctata</i>  |                                  |
| Family native to North America?              | Yes   |                                  |
| Age/size at start of test/growth phase       | Not reported  |                                  |
| Source of organisms                          | Urban waterbodies with minimal development in Northern California |                                  |
| Have organisms been exposed to contaminants? | Not reported  |                                  |
| Animals acclimated and disease-free?         | 24 h  |                                  |
| Animals randomized?                          | Not reported  |                                  |
| Test vessels randomized?                     | Not reported  |                                  |
| Test duration                                | 96 h  |                                  |
| Data for multiple times?                     | Not reported  |                                  |
| Effect 1                                     | Survival  |                                  |
| Control response 1                           | 85 %  |                                  |
| Effect 2                                     | Immobilization (ability to cling)                                 |                                  |
| Control response 2                           | Not reported  |                                  |
| Temperature                                  | 13 °C   |                                  |
| Test type                                    | Static  |                                  |
| Photoperiod/light intensity                  | 16l:8d; Not reported  |                                  |

|   | <b>Weston 2014</b>   | <b><i>I. quinquepunctata</i></b>                        |
|---|--|---|
| <b>Parameter</b>  | <b>Value</b>   | <b>Comment</b>  |
| Dilution water  | Milli-Q purified, deionized watermade moderately hard by addition of salts | *According to EPA 821-R-02-012                          |
| pH  | Not reported*  |   |
| Hardness  | Not reported*  |   |
| Alkalinity  | Not reported*  |   |
| Conductivity  | Not reported*  |   |
| Dissolved Oxygen  | Not reported*  |   |
| Feeding   | Not reported   |   |
| Purity of test substance  | 99.3 %   | Not reported but author verified from chemical supplier |
| Concentrations measured?  | Yes  |   |
| Measured is what % of nominal?  | 66-113%  |   |
| Toxicity values calculated based on nominal or measured concentrations? | Measured   |   |
| Chemical method documented?   | GC   |   |
| Concentration of carrier (if any) in test solutions                     | Acetone, <36 µg/L  |   |
| Concentration 1 Nom; Meas (µg/L)  | Not reported; 4-7 concentrations tested; dilution factor of 2              | 3 reps, 4-6/rep   |
| Control   | Negative: 0; 0<br>Solvent: 0; 0  |   |
| LC <sub>50</sub> (95% CI) (µg/L)  | 0.0500 (0.0431-0.0581)   | Method: Probit  |
| EC <sub>50</sub> (95% CI) (µg/L)  | 0.0474 (0.0402-0.0559)   | Method: Probit  |

Notes: Points were not deducted for lack of water quality parameter reporting because water was prepared according to EPA 821-R-02-012 and measured during study (but not reported).

Solubility (S) value for fipronil sulfone (MB 46136) = 160 µg/L, 2S = 320 µg/L.

Reliability points taken off for:

Documentation: Organism life stage/size (5), Nominal concentrations (3), Measured concentrations (3), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100-15 =85

Acceptability: Standard method (5), Organisms randomized (1), Temperature variation (3), Random design (2), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1). Total: 100-14 =86

**Reliability score: mean(85,86)=85.5**

## Water Toxicity Data Summary

*Lepomis macrochirus*  
Fipronil desulfinyl  
MB46513

Collins MK. (1993a) MB46513-Acute toxicity to bluegill sunfish (*Lepomis macrochirus*) under static renewal conditions. Springborn Laboratories, Inc., Wareham, Massachusetts. Laboratory study number 10566.0492.6242.100. Submitted to Rhone-Poulenc Ag Company, Research Triangle, North Carolina. USEPA MRID 43279702.

Relevance  
Score: 100  
Rating: R

Reliability  
Score: 95.5  
Rating: R

Relevance points taken off for: none.

|  | <b>Collins 1993</b>                            | <b><i>L. macrochirus</i></b> |
|--|--|------------------------------|
| <b>Parameter</b>                             | <b>Value</b>                                   | <b>Comment</b>               |
| Test method cited                            | FIFRA Guideline 72-1                           |                              |
| Phylum/subphylum                             | Chordata                                       |                              |
| Class  | Actinopterygii                                 |                              |
| Order  | Perciformes                                    |                              |
| Family                                       | Centrarchidae                                  |                              |
| Genus  | <i>Lepomis</i>                                 |                              |
| Species                                      | <i>macrochirus</i>                             |                              |
| Family native to North America?              | Yes  |                              |
| Age/size at start of test/growth phase       | 0.51 g<br>32 mm                                |                              |
| Source of organisms                          | Bybrook Bass Hatchery,<br>Ashford, Connecticut |                              |
| Have organisms been exposed to contaminants? | No   |                              |
| Animals acclimated and disease-free?         | Yes, 14 d                                      |                              |
| Animals randomized?                          | Yes  |                              |
| Test vessels randomized?                     | Yes  |                              |
| Test duration                                | 96 h   |                              |
| Data for multiple times?                     | 24, 48, 72, 96 h                               |                              |
| Effect 1                                     | Survival                                       |                              |
| Control response 1 (mean)                    | 100 %  |                              |
| Temperature                                  | 22 ± 1 °C                                      |                              |
| Test type                                    | Static renewal                                 |                              |
| Photoperiod/light intensity                  | 16l:8d/1100 lux                                |                              |
| Dilution water                               | Reconstituted from                             | ASTM 1980                    |

|   | <b>Collins 1993</b>  | <b><i>L. macrochirus</i></b>                                 |
|---|--|--|
| <b>Parameter</b>  | <b>Value</b>   | <b>Comment</b>   |
|   | deionized  |  |
| pH  | 7.5  |  |
| Hardness  | 36 mg/L CaCO <sub>3</sub>  |  |
| Alkalinity  | 24 mg/L CaCO <sub>3</sub>  |  |
| Conductivity  | 130 µmhos/cm   |  |
| Dissolved Oxygen  | 4.6-9.4 mg/L   | 53-108%<br>Not aerated                                       |
| Feeding   | Not fed  |  |
| Purity of test substance  | 98.6 %   |  |
| Concentrations measured?  | Yes  |  |
| Measured is what % of nominal?  | 72-89 %  |  |
| Toxicity values calculated based on nominal or measured concentrations? | Measured   |  |
| Chemical method documented?   | HPLC   |  |
| Concentration of carrier (if any) in test solutions                     | 0.50 mL/L, acetone   |  |
| Concentration 1 Nom; Meas (µg/L)  | 6.5; 4.7   | 1 reps, 10/rep   |
| Concentration 2 Nom; Meas (µg/L)  | 11; 9.6  |  |
| Concentration 3 Nom; Meas (µg/L)  | 18; 16   |  |
| Concentration 4 Nom; Meas (µg/L)  | 30; 26   |  |
| Concentration 5 Nom; Meas (µg/L)  | 50; 43   |  |
| Control   | Negative: 0; 0<br>Solvent: 0; 0  |  |
| LC <sub>50</sub> (95% CI) (µg/L)  | 24 h: 32 (26-43)<br>48 h: 28 (16-43)<br>72 h: 22 (18-27)<br>96 h: 20 (17-25) | Method: Moving average angle analysis                        |
| NOEC (µg/L)   | 96 h: 9.6  | Method: Not reported<br>p: Not reported<br>MSD: Not reported |
| LOEC (µg/L)   | 96 h: 16   | Not reported; See table 3                                    |
| MATC  | 96 h: 12   |  |
| % control at NOEC   | 100 % survival   |  |
| % control at LOEC   | 80 % survival  |  |

Notes: Dilution water TOC = 2.2 mg/L

Solubility (S) value for fipronil desulfinyl (MB 46513) = 950 µg/L, 2S = 1900 µg/L.

Reliability points taken off for:

Documentation: Statistical significance (2), Significance level (2), Minimum significant difference (2). Total:  $100 - 6 = 94$

Acceptability: Adequate replication (2), Minimum significant difference (1). Total:  $100 - 3 = 97$

**Reliability score:  $\text{mean}(94, 97) = 95.5$**

## Water Toxicity Data Summary

*Lepomis macrochirus*

Fipronil sulfone

MB46136

Bettencourt MJ. (1992a) (M&B 46136)-Acute toxicity to bluegill sunfish(*Lepomis macrochirus*) under flow-through conditions. Springborn Laboratories, Inc., Wareham, Massachusetts. Laboratory study number 10566.0.391.6207.105. Submitted to Rhone-Poulenc Ag Company, Research Triangle, North Carolina. USEPA MRID 42918674. CA DPR 157302.

Relevance

Score: 100

Rating: R

Reliability

Score: 94.5

Rating: R

Relevance points taken off for: none.

|  | <b>Bettencourt 1992</b>                        | <i>L. macrochirus</i> |
|--|--|-----------------------|
| <b>Parameter</b>                             | <b>Value</b>                                   | <b>Comment</b>        |
| Test method cited                            | FIFRA Guideline 72-1                           |                       |
| Phylum/subphylum                             | Chordata                                       |                       |
| Class  | Actinopterygii                                 |                       |
| Order  | Perciformes                                    |                       |
| Family                                       | Centrarchidae                                  |                       |
| Genus  | <i>Lepomis</i>                                 |                       |
| Species                                      | <i>macrochirus</i>                             |                       |
| Family native to North America?              | Yes  |                       |
| Age/size at start of test/growth phase       | 1.5 g<br>45 mm                                 |                       |
| Source of organisms                          | Bybrook Bass Hatchery,<br>Ashford, Connecticut |                       |
| Have organisms been exposed to contaminants? | No   |                       |
| Animals acclimated and disease-free?         | Yes  |                       |
| Animals randomized?                          | Yes  |                       |
| Test vessels randomized?                     | Yes  |                       |
| Test duration                                | 96 h   |                       |
| Data for multiple times?                     | 24, 48, 72, 96 h                               |                       |
| Effect 1                                     | Survival                                       |                       |
| Control response 1 (mean)                    | 100 %  |                       |
| Temperature                                  | 22 ± 1 °C                                      |                       |
| Test type                                    | Flow through                                   |                       |
| Photoperiod/light intensity                  | 16l:8d/22-56 footcandles                       |                       |
| Dilution water                               | Well water                                     |                       |

|   | <b>Bettencourt 1992</b>  | <i>L. macrochirus</i>  |
|---|--|--|
| <b>Parameter</b>  | <b>Value</b>   | <b>Comment</b>   |
| pH  | 7.0-7.2  |  |
| Hardness  | 30 mg/L CaCO <sub>3</sub>  |  |
| Alkalinity  | 24-27 mg/L CaCO <sub>3</sub>   |  |
| Conductivity  | 110-130 µmhos/cm   |  |
| Dissolved Oxygen  | 7.8-9.2 mg/L   | 89-105 %<br>Not aerated                                      |
| Feeding   | Not fed  |  |
| Purity of test substance  | 99.2 %   |  |
| Concentrations measured?  | Yes  |  |
| Measured is what % of nominal?  | Mean 64 %  |  |
| Toxicity values calculated based on nominal or measured concentrations? | Measured   |  |
| Chemical method documented?   | HPLC   |  |
| Concentration of carrier (if any) in test solutions                     | 0.0150 mL/L, acetone   |  |
| Concentration 1 Nom; Meas (µg/L)  | 9.7; 6.7   | 2 reps, /rep   |
| Concentration 2 Nom; Meas (µg/L)  | 16; 10   |  |
| Concentration 3 Nom; Meas (µg/L)  | 27; 17   |  |
| Concentration 4 Nom; Meas (µg/L)  | 45; 26   |  |
| Concentration 5 Nom; Meas (µg/L)  | 75; 51   |  |
| Control   | Negative: 0; 0<br>Solvent: 0; 0  |  |
| LC <sub>50</sub> (95% CI) (µg/L)  | 24 h: > 51<br>48 h: 38 (33-46)<br>72 h: 30 (25-35)<br>96 h: 25 (21-30) | Method: probit   |
| NOEC (µg/L)   | 6.7  | Method: Not reported<br>p: Not reported<br>MSD: Not reported |
| LOEC (µg/L)   | 10   | Not reported; See table 4                                    |
| MATC  | 8.2  |  |
| % control at NOEC   | 100 % survival   |  |
| % control at LOEC   | 95 % survival  |  |

Notes: Dilution water TOC = 0.5 mg/L

Solubility (S) value for fipronil sulfone (MB 46136) = 160 µg/L, 2S = 320 µg/L.

Reliability points taken off for:

Documentation: Statistical significance (2), Significance level (2), Minimum significant difference (2). Total:  $100 - 6 = 94$

Acceptability: Measured concentrations within 20% nominal (4), Minimum significant difference (1). Total:  $100 - 6 = 95$

**Reliability score:  $\text{mean}(94, 95) = 94.5$**

## Water Toxicity Data Summary

*Lepomis macrochirus*

Fipronil

MB46030

Ward GS. (1991a) M&B 46030: Acute toxicity to bluegill, *Lepomis macrochirus*, under flow-through test conditions. Toxikon Environmental Services, Jupiter, Florida. Laboratory project ID J9005012b. Submitted to Rhone-Poulenc Ag Company, Research Triangle Park, North Carolina. US EPA MRID 42918624. CA DPR 157279.

Relevance

Score: 100

Rating: R

Reliability

Score: 94

Rating: R

Relevance points taken off for: none.

|  | <b>Ward 1991</b>   | <i>L. macrochirus</i> |
|--|--|-----------------------|
| <b>Parameter</b>                             | <b>Value</b>   | <b>Comment</b>        |
| Test method cited                            | Section 72-1 of the Pesticide Assessment Guidelines, Subdivision E (US EPA 1982) |                       |
| Phylum/subphylum                             | Chordata   |                       |
| Class  | Actinopterygii   |                       |
| Order  | Perciformes  |                       |
| Family                                       | Centrarchidae  |                       |
| Genus  | <i>Lepomis</i>   |                       |
| Species                                      | <i>macrochirus</i>   |                       |
| Family native to North America?              | Yes  |                       |
| Age/size at start of test/growth phase       | Juvenile, 17-23 mm, 0.12-0.44 g  |                       |
| Source of organisms                          | Northeastern Biologists, Rhinebeck, New Jersey                                   |                       |
| Have organisms been exposed to contaminants? | No   |                       |
| Animals acclimated and disease-free?         | 2 d prophylactic salt water (5 ‰) then 19 d fresh water                          |                       |
| Animals randomized?                          | Yes  |                       |
| Test vessels randomized?                     | Yes  |                       |
| Test duration                                | 96 h   |                       |
| Data for multiple times?                     | 24, 48, 72, 96 h   |                       |
| Effect 1                                     | Mortality  |                       |
| Control response 1                           | 0 %  |                       |
| Temperature                                  | 21.5 ± 1.6 °C  |                       |
| Test type                                    | Flow through   |                       |

|   | <b>Ward 1991</b>                   | <i>L. macrochirus</i>              |
|---|------------------------------------|------------------------------------|
| <b>Parameter</b>  | <b>Value</b>                       | <b>Comment</b>                     |
| Photoperiod/light intensity   | 16l:8d/325-433 lux                 |                                    |
| Dilution water  | Tap water, carbon-treated, aerated |                                    |
| pH  | 7.1-8.0                            |                                    |
| Hardness  | 56 mg/L CaCO <sub>3</sub>          |                                    |
| Alkalinity  | 20-24 mg/L CaCO <sub>3</sub>       |                                    |
| Conductivity  | 361-372 µmhos/cm                   |                                    |
| Dissolved Oxygen  | ≥7.2 mg/L                          | ≥83 %                              |
| Feeding   | Not fed                            |                                    |
| Purity of test substance  | 100 %                              |                                    |
| Concentrations measured?  | Yes                                |                                    |
| Measured is what % of nominal?  | 94-108 %                           |                                    |
| Toxicity values calculated based on nominal or measured concentrations? | Measured                           |                                    |
| Chemical method documented?   | GC                                 |                                    |
| Concentration of carrier (if any) in test solutions                     | ≤0.1 mL/L dimethylformamide        |                                    |
| Concentration 1 Nom; Meas (µg/L)  | 26; 27.1                           | 1 reps, 20/rep                     |
| Concentration 2 Nom; Meas (µg/L)  | 43; 43.2                           |                                    |
| Concentration 3 Nom; Meas (µg/L)  | 72; 67.4                           |                                    |
| Concentration 4 Nom; Meas (µg/L)  | 120; 134                           |                                    |
| Concentration 5 Nom; Meas (µg/L)  | 200; 217                           |                                    |
| Control   | Negative: 0; 0<br>Solvent: 0; 0    |                                    |
| LC <sub>50</sub> (95% CI) (µg/L)  | 85.2 (74.2-99.0)                   | Method: Probit                     |
| NOEC  | 43.2                               | Method: Not reported<br>p:<br>MSD: |
| % control at NOEC   | 100%                               |                                    |

Notes:

Solubility (S) of fipronil = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Statistical significance (2), Significance level (2), Minimum significant difference (2). Total: 100- 6=94

Acceptability: Temperature variation (3), Adequate replication (2), Minimum significant difference (1). Total: 100-6 =94

**Reliability score: mean(94, 94)=94**

## Water Toxicity Data Summary

*Nectopsyche* sp.  
Fipronil sulfide  
MB45950

Weston DP and Lydy MJ. (2014) Toxicity of the insecticide fipronil and its degradates to benthic macroinvertebrates of urban streams. *Environmental science & technology*, 48(2), 1290-1297.

Relevance  
Score: 90  
Rating: R

Reliability  
Score: 85.5  
Rating: R

Relevance points taken off for: Standard method (10). 100-10=90

|  | <b>Weston 2014</b>  | <i>Nectopsyche</i> sp. |
|--|---|------------------------|
| <b>Parameter</b>                             | <b>Value</b>  | <b>Comment</b>         |
| Test method cited                            | Not reported  |                        |
| Phylum/subphylum                             | Anthropoda  |                        |
| Class  | Insecta   |                        |
| Order  | Trichoptera   |                        |
| Family                                       | Leptoceridae  |                        |
| Genus  | <i>Nectopsyche</i>  |                        |
| Species                                      | sp.   |                        |
| Family native to North America?              | Yes   |                        |
| Age/size at start of test/growth phase       | Not reported  |                        |
| Source of organisms                          | Urban waterbodies with minimal development in Northern California |                        |
| Have organisms been exposed to contaminants? | Not reported  |                        |
| Animals acclimated and disease-free?         | 24 h  |                        |
| Animals randomized?                          | Not reported  |                        |
| Test vessels randomized?                     | Not reported  |                        |
| Test duration                                | 48 h  |                        |
| Data for multiple times?                     | Not reported  |                        |
| Effect 1                                     | Survival  |                        |
| Control response 1                           | 96 %  |                        |
| Effect 2                                     | Immobilization (ability to crawl)                                 |                        |
| Control response 2                           | Not reported  |                        |
| Temperature                                  | 23 °C   |                        |
| Test type                                    | Static  |                        |
| Photoperiod/light intensity                  | 16l:8d; Not reported  |                        |

|   | <b>Weston 2014</b>   | <b><i>Nectopsyche</i> sp.</b>                           |
|---|--|---|
| <b>Parameter</b>  | <b>Value</b>   | <b>Comment</b>  |
| Dilution water  | Milli-Q purified, deionized watermade moderately hard by addition of salts | *According to EPA 821-R-02-012                          |
| pH  | Not reported*  |   |
| Hardness  | Not reported*  |   |
| Alkalinity  | Not reported*  |   |
| Conductivity  | Not reported*  |   |
| Dissolved Oxygen  | Not reported*  |   |
| Feeding   | Not reported   |   |
| Purity of test substance  | 99.0 %   | Not reported but author verified from chemical supplier |
| Concentrations measured?  | Yes  |   |
| Measured is what % of nominal?  | 66-113%  |   |
| Toxicity values calculated based on nominal or measured concentrations? | Measured   |   |
| Chemical method documented?   | GC   |   |
| Concentration of carrier (if any) in test solutions                     | Acetone, <36 µg/L  |   |
| Concentration 1 Nom; Meas (µg/L)  | Not reported; 4-7 concentrations tested; dilution factor of 2              | 3 reps, 4-6/rep   |
| Control   | Negative: 0; 0<br>Solvent: 0; 0  |   |
| LC <sub>50</sub> (95% CI) (µg/L)  | 0.122 (0.0602-0.177)   | Method: Probit  |
| EC <sub>50</sub> (95% CI) (µg/L)  | 0.0285 (0.0187-0.0365)   | Method: Probit  |

Notes: Points were not deducted for lack of water quality parameter reporting because water was prepared according to EPA 821-R-02-012 and measured during study (but not reported).

Solubility value for fipronil sulfide (MB 45950) not available. Solubility (S) of fipronil parent compound (MB 46030) = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Organism life stage/size (5), Nominal concentrations (3), Measured concentrations (3), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100-15 =85

Acceptability: Standard method (5), Organisms randomized (1), Temperature variation (3), Random design (2), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1). Total: 100-14 =86

**Reliability score: mean(85,86)=85.5**

## Water Toxicity Data Summary

*Nectopsyche* sp.

Fipronil

MB46030

Weston DP and Lydy MJ. (2014) Toxicity of the insecticide fipronil and its degradates to benthic macroinvertebrates of urban streams. *Environmental science & technology*, 48(2), 1290-1297.

Relevance

Score: 90

Rating: R

Reliability

Score: 85.5

Rating: R

Relevance points taken off for: Standard method (10). 100-10=90

|  | <b>Weston 2014</b>  | <b><i>Nectopsyche</i> sp.</b> |
|--|---|-------------------------------|
| <b>Parameter</b>                             | <b>Value</b>  | <b>Comment</b>                |
| Test method cited                            | Not reported  |                               |
| Phylum/subphylum                             | Anthropoda  |                               |
| Class  | Insecta   |                               |
| Order  | Trichoptera   |                               |
| Family                                       | Leptoceridae  |                               |
| Genus  | <i>Nectopsyche</i>  |                               |
| Species                                      | sp.   |                               |
| Family native to North America?              | Yes   |                               |
| Age/size at start of test/growth phase       | Not reported  |                               |
| Source of organisms                          | Urban waterbodies with minimal development in Northern California |                               |
| Have organisms been exposed to contaminants? | Not reported  |                               |
| Animals acclimated and disease-free?         | 24 h  |                               |
| Animals randomized?                          | Not reported  |                               |
| Test vessels randomized?                     | Not reported  |                               |
| Test duration                                | 48 h  |                               |
| Data for multiple times?                     | Not reported  |                               |
| Effect 1                                     | Survival  |                               |
| Control response 1                           | 100 %   |                               |
| Effect 2                                     | Immobilization (ability to crawl)                                 |                               |
| Control response 2                           | Not reported  |                               |
| Temperature                                  | 12 °C   |                               |
| Test type                                    | Static  |                               |
| Photoperiod/light intensity                  | 16l:8d; Not reported  |                               |

|   | <b>Weston 2014</b>   | <i>Nectopsyche</i> sp.                                  |
|---|--|---|
| <b>Parameter</b>  | <b>Value</b>   | <b>Comment</b>  |
| Dilution water  | Milli-Q purified, deionized watermade moderately hard by addition of salts | *According to EPA 821-R-02-012                          |
| pH  | Not reported*  |   |
| Hardness  | Not reported*  |   |
| Alkalinity  | Not reported*  |   |
| Conductivity  | Not reported*  |   |
| Dissolved Oxygen  | Not reported*  |   |
| Feeding   | Not reported   |   |
| Purity of test substance  | 99.50 %  | Not reported but author verified from chemical supplier |
| Concentrations measured?  | Yes  |   |
| Measured is what % of nominal?  | 66-113%  |   |
| Toxicity values calculated based on nominal or measured concentrations? | Measured   |   |
| Chemical method documented?   | GC   |   |
| Concentration of carrier (if any) in test solutions                     | Acetone, <36 µg/L  |   |
| Concentration 1 Nom; Meas (µg/L)  | Not reported; 4-7 concentrations tested; dilution factor of 2              | 3 reps, 4-6/rep   |
| Control   | Negative: 0; 0<br>Solvent: 0; 0  |   |
| LC <sub>50</sub> (95% CI) (µg/L)  | >2.947   | Method: Probit  |
| EC <sub>50</sub> (95% CI) (µg/L)  | 0.634 (0.531-0.756)  | Method: Probit  |

Notes: Points were not deducted for lack of water quality parameter reporting because water was prepared according to EPA 821-R-02-012 and measured during study (but not reported).

Solubility (S) of fipronil = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Organism life stage/size (5), Nominal concentrations (3), Measured concentrations (3), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100-15 =85

Acceptability: Standard method (5), Organisms randomized (1), Temperature variation (3), Random design (2), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1). Total: 100-14 =86

**Reliability score: mean(85,86)=85.5**

## Water Toxicity Data Summary

*Nectopsyche* sp.  
Fipronil sulfone  
MB46136

Weston DP and Lydy MJ. (2014) Toxicity of the insecticide fipronil and its degradates to benthic macroinvertebrates of urban streams. *Environmental science & technology*, 48(2), 1290-1297.

Relevance  
Score: 90  
Rating: R

Reliability  
Score: 85.5  
Rating: R

Relevance points taken off for: Standard method (10). 100-10=90

|  | <b>Weston 2014</b>  | <b><i>Nectopsyche</i> sp.</b> |
|--|---|-------------------------------|
| <b>Parameter</b>                             | <b>Value</b>  | <b>Comment</b>                |
| Test method cited                            | Not reported  |                               |
| Phylum/subphylum                             | Anthropoda  |                               |
| Class  | Insecta   |                               |
| Order  | Trichoptera   |                               |
| Family                                       | Leptoceridae  |                               |
| Genus  | <i>Nectopsyche</i>  |                               |
| Species                                      | sp.   |                               |
| Family native to North America?              | Yes   |                               |
| Age/size at start of test/growth phase       | Not reported  |                               |
| Source of organisms                          | Urban waterbodies with minimal development in Northern California |                               |
| Have organisms been exposed to contaminants? | Not reported  |                               |
| Animals acclimated and disease-free?         | 24 h  |                               |
| Animals randomized?                          | Not reported  |                               |
| Test vessels randomized?                     | Not reported  |                               |
| Test duration                                | 48 h  |                               |
| Data for multiple times?                     | Not reported  |                               |
| Effect 1                                     | Survival  |                               |
| Control response 1                           | 96 %  |                               |
| Effect 2                                     | Immobilization (ability to crawl)                                 |                               |
| Control response 2                           | Not reported  |                               |
| Temperature                                  | 23 °C   |                               |
| Test type                                    | Static  |                               |
| Photoperiod/light intensity                  | 16l:8d; Not reported  |                               |

|   | <b>Weston 2014</b>   | <b><i>Nectopsyche</i> sp.</b>                           |
|---|--|---|
| <b>Parameter</b>  | <b>Value</b>   | <b>Comment</b>  |
| Dilution water  | Milli-Q purified, deionized watermade moderately hard by addition of salts | *According to EPA 821-R-02-012                          |
| pH  | Not reported*  |   |
| Hardness  | Not reported*  |   |
| Alkalinity  | Not reported*  |   |
| Conductivity  | Not reported*  |   |
| Dissolved Oxygen  | Not reported*  |   |
| Feeding   | Not reported   |   |
| Purity of test substance  | 99.3 %   | Not reported but author verified from chemical supplier |
| Concentrations measured?  | Yes  |   |
| Measured is what % of nominal?  | 66-113%  |   |
| Toxicity values calculated based on nominal or measured concentrations? | Measured   |   |
| Chemical method documented?   | GC   |   |
| Concentration of carrier (if any) in test solutions                     | Acetone, <36 µg/L  |   |
| Concentration 1 Nom; Meas (µg/L)  | Not reported; 4-7 concentrations tested; dilution factor of 2              | 3 reps, 4-6/rep   |
| Control   | Negative: 0; 0<br>Solvent: 0; 0  |   |
| LC <sub>50</sub> (95% CI) (µg/L)  | 0.0515 (0.0370-0.0691)   | Method: Probit  |
| EC <sub>50</sub> (95% CI) (µg/L)  | 0.0313 (0.0230-0.0401)   | Method: Probit  |

Notes: Points were not deducted for lack of water quality parameter reporting because water was prepared according to EPA 821-R-02-012 and measured during study (but not reported).

Solubility (S) value for fipronil sulfone (MB 46136) = 160 µg/L, 2S = 320 µg/L.

Reliability points taken off for:

Documentation: Organism life stage/size (5), Nominal concentrations (3), Measured concentrations (3), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100-15 =85

Acceptability: Standard method (5), Organisms randomized (1), Temperature variation (3), Random design (2), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1). Total: 100-14 =86

**Reliability score: mean(85,86)=85.5**

## Water Toxicity Data Summary

*Oncorhynchus mykiss*

Fipronil

MB46030

Machado MW. (1992a) The toxicity to rainbow trout (*Oncorhynchus mykiss*) during an early life-stage exposure. Springborn Laboratories, Inc., Wareham, Massachusetts. Laboratory study number 10566.0.391.6209.121. Submitted to Rhone-Poulenc Ag Company, Research Triangle, North Carolina. USEPA MRID 42918627. CA DPR 157287.

Relevance

Score: 100

Rating: R

Reliability

Score: 90

Rating: R

Relevance points taken off for: none.

|  | <b>Machado 1992</b>                            | <b><i>O. mykiss</i></b> |
|--|--|-------------------------|
| <b>Parameter</b>                             | <b>Value</b>                                   | <b>Comment</b>          |
| Test method cited                            | FIFRA Guideline 72-4                           |                         |
| Phylum/subphylum                             | Chordata                                       |                         |
| Class  | Actinopterygii                                 |                         |
| Order  | Salmoniformes                                  |                         |
| Family                                       | Salmonidae                                     |                         |
| Genus  | <i>Oncorhynchus</i>                            |                         |
| Species                                      | <i>mykiss</i>                                  |                         |
| Family native to North America?              | Yes  |                         |
| Age/size at start of test/growth phase       | Fertilized eggs                                |                         |
| Source of organisms                          | Mount Lassen Trout Farm, Red Bluff, California |                         |
| Have organisms been exposed to contaminants? | No   |                         |
| Animals acclimated and disease-free?         | Yes  |                         |
| Animals randomized?                          | Yes  |                         |
| Test vessels randomized?                     | Yes  |                         |
| Test duration                                | 90 d, 60 d post-hatch                          |                         |
| Data for multiple times?                     | Hatch ("0 d"), 60 d post-hatch                 |                         |
| Effect 1                                     | Embryo viability (0 d)                         |                         |
| Control response 1 (mean)                    | 94 %   |                         |
| Effect 2                                     | Survival at hatch (0 d)                        |                         |
| Control response 2 (mean)                    | 98 %   |                         |
| Effect 3                                     | Larval survival (60 d)                         |                         |

|   | <b>Machado 1992</b>                                       | <b><i>O. mykiss</i></b>  |
|---|---|--|
| <b>Parameter</b>  | <b>Value</b>  | <b>Comment</b>   |
| Control response 3 (mean)   | 98  |  |
| Effect 4  | Length (60 d)   |  |
| Control response 4 (mean)   | 60 mm   |  |
| Effect 5  | Wet weight  |  |
| Control response 5 (mean)   | 2.2 g   |  |
| Temperature   | 12 ± 1 °C   |  |
| Test type   | Flow through  |  |
| Photoperiod/light intensity   | 16l:8d/30-50 footcandles                                  |  |
| Dilution water  | Untreated well water                                      |  |
| pH  | 6.8-7.4   |  |
| Hardness  | 24-32 mg/L CaCO <sub>3</sub>                              |  |
| Alkalinity  | 18-25 mg/L CaCO <sub>3</sub>                              |  |
| Conductivity  | 100-150 µmhos/cm  |  |
| Dissolved Oxygen  | 9.6-10 mg/L   | 90-93 %  |
| Feeding   | Live bring shrimp ( <i>Artemia salina</i> ) nauplii 2-3/d |  |
| Purity of test substance  | 96.7 %  |  |
| Concentrations measured?  | Yes   |  |
| Measured is what % of nominal?  | 42-60 %   |  |
| Toxicity values calculated based on nominal or measured concentrations? | Measured  |  |
| Chemical method documented?   | HPLC  |  |
| Concentration of carrier (if any) in test solutions                     | 6.5 µL/L acetone  |  |
| Concentration 1 Nom; Meas (µg/L)  | 6.2; 2.6  | 2 reps, 56 eggs/rep  |
| Concentration 2 Nom; Meas (µg/L)  | 12; 6.6   |  |
| Concentration 3 Nom; Meas (µg/L)  | 25; 15  |  |
| Concentration 4 Nom; Meas (µg/L)  | 50; 26  |  |
| Concentration 5 Nom; Meas (µg/L)  | 100; 60   |  |
| Control   | Negative: 0; 0<br>Solvent: 0; 0                           |  |
| NOEC  | 15  | Method: William's Test<br>p: 0.05<br>MSD:<br>Based on survival |
| LOEC  | 26  | Based on survival  |
| MATC (GeoMean NOEC, LOEC)   | 20  |  |
| % control at NOEC   | Embryo viability (0 d):<br>100%                           | Embryo viability (0 d): 94 (tmt) / 94 (mean controls) =        |

|                   | <b>Machado 1992</b>  | <b><i>O. mykiss</i></b>  |
|-------------------|--|--|
| <b>Parameter</b>  | <b>Value</b>   | <b>Comment</b>   |
|                   | Survival at hatch (0 d): 104%<br>Larval survival (60 d): 95%<br>Length (60 d): 97 %<br>Wet weight (60 d): 95 %                               | 100<br>Survival at hatch (0 d): 100 (tmt) / 96 (mean controls) = 104<br>Larval survival (60 d): 93 (tmt) / 98 (mean controls) = 95<br>Length (60 d): 58 (tmt) / 60 (mean controls) = 97<br>Wet weight (60 d): 2.1 (tmt) / 2.2 (mean controls) = 95   |
| % control at LOEC | Embryo viability (0 d): 103%<br>Survival at hatch (0 d): 100%<br>Larval survival (60 d): 80%<br>Length (60 d): 83%<br>Wet weight (60 d): 77% | Embryo viability (0 d): 97 (tmt) / 94 (mean controls) = 103<br>Survival at hatch (0 d): 98 (tmt) / 98 (mean controls) = 100%<br>Larval survival (60 d): 78 (tmt) / 98 (mean controls) = 80<br>Length (60 d): 50 (tmt) / 60 (mean controls) = 83<br>Wet weight (60 d): 1.7 (tmt) / 2.2 (mean controls) = 77 |

Notes: Dilution water TOC average = 0.66 mg/L

Solubility (S) of fipronil = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Minimum significant difference (2), Point estimates (8). Total: 100- 10=90

Acceptability: Measured concentrations within 20% nominal (4), Adequate replication (2), Minimum significant difference (1), Point estimates (3). Total: 100- 10=90

**Reliability score: mean(90, 90)=90**

## Water Toxicity Data Summary

*Oncorhynchus mykiss*

Fipronil sulfone

MB46136

Bettencourt MJ. (1992b) (M&B 46136)-Acute toxicity to rainbow trout (*Oncorhynchus mykiss*) under flow-through conditions. Springborn Laboratories, Inc., Wareham, Massachusetts. Laboratory study number 10566.0.391.6208.108. Submitted to Rhone-Poulenc Ag Company, Research Triangle, North Carolina. USEPA MRID 42918673. CA DPR 157303.

Relevance

Score: 100

Rating: R

Reliability

Score: 93.5

Rating: R

Relevance points taken off for: none.

|  | <b>Bettencourt 1992</b>                           | <i>O. mykiss</i> |
|--|---|------------------|
| <b>Parameter</b>                             | <b>Value</b>                                      | <b>Comment</b>   |
| Test method cited                            | FIFRA Guideline 72-1                              |                  |
| Phylum/subphylum                             | Chordata  |                  |
| Class  | Actinopterygii                                    |                  |
| Order  | Salmoniformes                                     |                  |
| Family                                       | Salmonidae  |                  |
| Genus  | <i>Oncorhynchus</i>                               |                  |
| Species                                      | <i>mykiss</i>                                     |                  |
| Family native to North America?              | Yes   |                  |
| Age/size at start of test/growth phase       | 0.55 g<br>39 mm                                   |                  |
| Source of organisms                          | Mount Lassen Trout Farm,<br>Red Bluff, California |                  |
| Have organisms been exposed to contaminants? | No  |                  |
| Animals acclimated and disease-free?         | 14 d  |                  |
| Animals randomized?                          | Yes   |                  |
| Test vessels randomized?                     | Yes   |                  |
| Test duration                                | 96 h  |                  |
| Data for multiple times?                     | 24, 48, 72, 96 h                                  |                  |
| Effect 1                                     | Survival  |                  |
| Control response 1 (mean)                    | 100 %   |                  |
| Temperature                                  | 12 ± 1 °C   |                  |
| Test type                                    | Flow through                                      |                  |
| Photoperiod/light intensity                  | 16l:8d  |                  |
| Dilution water                               | Well water  |                  |

|   | <b>Bettencourt 1992</b>  | <b><i>O. mykiss</i></b>  |
|---|--|--|
| <b>Parameter</b>  | <b>Value</b>   | <b>Comment</b>   |
| pH  | 7.0-7.1  |  |
| Hardness  | 30-32 mg/L CaCO <sub>3</sub>   |  |
| Alkalinity  | 22-23 mg/L CaCO <sub>3</sub>   |  |
| Conductivity  | 130-140 µmhos/cm   |  |
| Dissolved Oxygen  | 9-9.7 mg/L   | 83-92 %<br>Not aerated   |
| Feeding   | Not fed  |  |
| Purity of test substance  | 99.2 %   |  |
| Concentrations measured?  | Yes  |  |
| Measured is what % of nominal?  | 78-127 %   |  |
| Toxicity values calculated based on nominal or measured concentrations? | Measured   |  |
| Chemical method documented?   | HPLC   |  |
| Concentration of carrier (if any) in test solutions                     | 0.1 mL/L acetone   |  |
| Concentration 1 Nom; Meas (µg/L)  | 13; 11   | 2 reps, 10/rep   |
| Concentration 2 Nom; Meas (µg/L)  | 22; 18   |  |
| Concentration 3 Nom; Meas (µg/L)  | 36; 29   |  |
| Concentration 4 Nom; Meas (µg/L)  | 60; 47   |  |
| Concentration 5 Nom; Meas (µg/L)  | 79; 100  |  |
| Control   | Negative: 0; 0<br>Solvent: 0; 0  |  |
| LC <sub>50</sub> (95% CI) (µg/L)  | 24 h: 59 (53-66)<br>48 h: 44 (39-50)<br>72 h: 40 (36-44)<br>96 h: 39 (35-43)                 | Method: probit   |
| NOEC (µg/L)   | 18   |  |
| LOEC (µg/L)   | 29   |  |
| MATC  | 23   |  |
| % control at NOEC   | 24 h: 100 % survival<br>48 h: 100 % survival<br>72 h: 100 % survival<br>96 h: 100 % survival |  |
| % control at LOEC   | 24 h: 100 % survival<br>48 h: 95 % survival<br>72 h: 95 % survival<br>96 h: 95 % survival    | 24 h: 100 (tmt) /<br>100 (mean controls)<br>= 100 %<br><br>48 h: 95 (tmt) / 100<br>(mean controls) =<br>95 %<br><br>72 h: 95 (tmt) / 100 |

|                  | <b>Bettencourt 1992</b> | <b><i>O. mykiss</i></b>   |
|------------------|-------------------------|---|
| <b>Parameter</b> | <b>Value</b>            | <b>Comment</b>  |
|                  |                         | (mean controls) = 95 %<br><br>96 h: 95 (tmt) / 100 (mean controls) = 95 % |

Notes: Dilution water TOC average = 0.60 mg/L

Solubility (S) value for fipronil sulfone (MB 46136) = 160 µg/L, 2S = 320 µg/L.

Reliability points taken off for:

Documentation: Statistical significance (2), Significance level (2), Minimum significant difference (2). Total: 100-6 =94

Acceptability: Measured concentrations within 20% nominal (4), Adequate replication (2), Minimum significant difference (1). Total: 100- 7=93

**Reliability score: mean(94, 93)=93.5**

## Water Toxicity Data Summary

*Oncorhynchus mykiss*  
Fipronil desulfinyl  
MB46513

Collins MK. (1993b) MB46513-Acute toxicity to rainbow trout (*Oncorhynchus mykiss*) under static renewal conditions. Springborn Laboratories, Inc., Wareham, Massachusetts. Laboratory study number 10566.0492.6241.103. Submitted to Rhone-Poulenc Ag Company, Research Triangle, North Carolina. USEPA MRID 43279703. CA DPR 157299.

Relevance  
Score: 100  
Rating: R

Reliability  
Score: 93.5  
Rating: R

Relevance points taken off for: none.

|  | <b>Collins 1993</b>                               | <b><i>O. mykiss</i></b> |
|--|---|-------------------------|
| <b>Parameter</b>                             | <b>Value</b>                                      | <b>Comment</b>          |
| Test method cited                            | FIFRA Guideline 72-1                              |                         |
| Phylum/subphylum                             | Chordata  |                         |
| Class  | Actinopterygii                                    |                         |
| Order  | Salmoniformes                                     |                         |
| Family                                       | Salmonidae  |                         |
| Genus  | <i>Oncorhynchus</i>                               |                         |
| Species                                      | <i>mykiss</i>                                     |                         |
| Family native to North America?              | Yes   |                         |
| Age/size at start of test/growth phase       | 0.85 g<br>45 mm                                   |                         |
| Source of organisms                          | Mount Lassen Trout Farm,<br>Red Bluff, California |                         |
| Have organisms been exposed to contaminants? | No  |                         |
| Animals acclimated and disease-free?         | 14 d  |                         |
| Animals randomized?                          | Yes   |                         |
| Test vessels randomized?                     | Yes   |                         |
| Test duration                                | 96 h  |                         |
| Data for multiple times?                     | 24, 48, 72, 96 h                                  |                         |
| Effect 1                                     | Survival  |                         |
| Control response 1 (mean)                    | 100 %   |                         |
| Temperature                                  | 12 ± 1 °C   |                         |
| Test type                                    | Static renewal                                    |                         |
| Photoperiod/light intensity                  | 16l:8d/970 lux                                    |                         |
| Dilution water                               | Reconstituted from                                | ASTM 1980               |

|   | <b>Collins 1993</b>  | <b><i>O. mykiss</i></b>          |
|---|--|----------------------------------|
| <b>Parameter</b>  | <b>Value</b>   | <b>Comment</b>                   |
|   | deionized  |                                  |
| pH  | 7.5  |                                  |
| Hardness  | 36 mg/L CaCO <sub>3</sub>  |                                  |
| Alkalinity  | 24 mg/L CaCO <sub>3</sub>  |                                  |
| Conductivity  | 130 µmhos/cm   |                                  |
| Dissolved Oxygen  | 4.9-9.8 mg/L   | 45-91 %<br>Not aerated           |
| Feeding   | Not fed  |                                  |
| Purity of test substance  | 98.6 %   |                                  |
| Concentrations measured?  | Yes  |                                  |
| Measured is what % of nominal?  | 84-100 %   |                                  |
| Toxicity values calculated based on nominal or measured concentrations? | Measured   |                                  |
| Chemical method documented?   | HPLC   |                                  |
| Concentration of carrier (if any) in test solutions                     | 0.5 mL/L acetone   |                                  |
| Concentration 1 Nom; Meas (µg/L)  | 3.9; 3.3   | 1 reps, 10/rep                   |
| Concentration 2 Nom; Meas (µg/L)  | 6.5; 6.4   |                                  |
| Concentration 3 Nom; Meas (µg/L)  | 11; 11   |                                  |
| Concentration 4 Nom; Meas (µg/L)  | 18; 17   |                                  |
| Concentration 5 Nom; Meas (µg/L)  | 30; 28   |                                  |
| Concentration 6 Nom; Meas (µg/L)  | 50; 42   |                                  |
| Control   | Negative: 0; 0<br>Solvent: 0; 0  |                                  |
| LC <sub>50</sub> (95% CI) (µg/L)  | 24 h: 36 (28-42)<br>48 h: 34 (28-42)<br>72 h: 32 (17-42)<br>96 h: 31 (17-42)                 | Method: Non-linear interpolation |
| NOEC (µg/L)   | 11   |                                  |
| LOEC (µg/L)   | 28   | Not reported; See Table 3        |
| MATC  | 18   |                                  |
| % control at NOEC   | 24 h: 100 % survival<br>48 h: 100 % survival<br>72 h: 100 % survival<br>96 h: 100 % survival |                                  |
| % control at LOEC   | 24 h: 100 % survival<br>48 h: 100 % survival<br>72 h: 80 % survival<br>96 h: 70 % survival   |                                  |

Notes: Dilution water TOC average = 0.60 mg/L

Solubility (S) value for fipronil desulfinyl (MB 46513) = 950 µg/L, 2S = 1900 µg/L.

Reliability points taken off for:

Documentation: Statistical significance (2), Significance level (2), Minimum significant difference (2). Total:  $100 - 6 = 94$

Acceptability: Adequate replication (2), Minimum significant difference (1). Total:  $100 - 3 = 97$

**Reliability score:  $\text{mean}(94, 97) = 95.5$**

## Water Toxicity Data Summary

*Oncorhynchus mykiss*

Fipronil

MB46030

Ward GS. (1991b) M&B 46030: Acute toxicity to rainbow trout, *Oncorhynchus mykiss*, under flow-through test conditions. Toxikon Environmental Services, Jupiter, Florida. Laboratory project ID J9005012a. Submitted to Rhone-Poulenc Ag Company, Research Triangle Park, North Carolina. US EPA MRID 42977902. CA DPR 157280.

Relevance

Score: 100

Rating: R

Reliability

Score: 95.5

Rating: R

Relevance points taken off for: none.

|  | <b>Ward 1991</b>   | <b><i>O. mykiss</i></b> |
|--|--|-------------------------|
| <b>Parameter</b>                             | <b>Value</b>   | <b>Comment</b>          |
| Test method cited                            | Section 72-1 of the Pesticide Assessment Guidelines, Subdivision E (US EPA 1982) |                         |
| Phylum/subphylum                             | Chordata   |                         |
| Class  | Actinopterygii   |                         |
| Order  | Salmoniformes  |                         |
| Family                                       | Salmonidae   |                         |
| Genus  | <i>Oncorhynchus</i>  |                         |
| Species                                      | <i>mykiss</i>  |                         |
| Family native to North America?              | Yes  |                         |
| Age/size at start of test/growth phase       | Juvenile: 36 mm, 0.98 g  |                         |
| Source of organisms                          | Aquatic Research Organisms, Hampton, New Hampshire                               |                         |
| Have organisms been exposed to contaminants? | No   |                         |
| Animals acclimated and disease-free?         | 2 d  |                         |
| Animals randomized?                          | Yes  |                         |
| Test vessels randomized?                     | Yes  |                         |
| Test duration                                | 96 h   |                         |
| Data for multiple times?                     | 24, 48, 72, 96 h   |                         |
| Effect 1                                     | Mortality  |                         |
| Control response 1                           | 0 %  |                         |
| Temperature                                  | 11.55 ± 0.75 °C  |                         |

|   | <b>Ward 1991</b>                       | <i>O. mykiss</i>          |
|---|--|---------------------------|
| <b>Parameter</b>  | <b>Value</b>                           | <b>Comment</b>            |
| Test type   | Flow through                           |                           |
| Photoperiod/light intensity   | 16l:8d/367-508 lux                     |                           |
| Dilution water  | Tap water                              |                           |
| pH  | 6.8-7.7                                |                           |
| Hardness  | 68-84 mg/L CaCO <sub>3</sub>           |                           |
| Alkalinity  | 16-17 mg/L CaCO <sub>3</sub>           |                           |
| Conductivity  | 342-361 µmhos/cm                       |                           |
| Dissolved Oxygen  | 8.3-9.8 mg/L                           | 77-91 %                   |
| Feeding   | Not fed                                |                           |
| Purity of test substance  | 100 %                                  |                           |
| Concentrations measured?  | Yes                                    |                           |
| Measured is what % of nominal?  | 81-95 %                                |                           |
| Toxicity values calculated based on nominal or measured concentrations? | Measured                               |                           |
| Chemical method documented?   | GC                                     |                           |
| Concentration of carrier (if any) in test solutions                     | 10 µL/L, dimethylformamide             |                           |
| Concentration 1 Nom; Meas (µg/L)  | 39; 33.8                               | 1 reps, 10/rep            |
| Concentration 2 Nom; Meas (µg/L)  | 65; 59.1                               |                           |
| Concentration 3 Nom; Meas (µg/L)  | 108; 87.6                              |                           |
| Concentration 4 Nom; Meas (µg/L)  | 180; 160                               |                           |
| Concentration 5 Nom; Meas (µg/L)  | 300; 266                               |                           |
| Control   | Negative: 0; 0<br>Solvent: 0; 0        |                           |
| LC <sub>50</sub> (95% CI) (µg/L)  | 72 h: 248 (160-∞)<br>96 h: 248 (160-∞) | Method: binomial          |
| NOEC (µg/L)   | 33.8                                   | Method:<br>p:<br>MSD:     |
| LOEC (µg/L)   | 160                                    | Not reported; See Table 2 |
| MATC  | 78.0                                   |                           |
| % control at NOEC   | 100 % survival                         |                           |
| % control at LOEC   | 80 % survival                          |                           |

Notes: Solubility (S) of fipronil = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Statistical significance (2), Significance level (2), Minimum significant difference (2). Total: 100- 6=94

Acceptability: Adequate replication (2), Minimum significant difference (1). Total: 100- 3=97

**Reliability score: mean(94,97)=95.5**

## Water Toxicity Data Summary

*Pimephales promelas*

Fipronil

MB 46030

Baird S, Garrison A, Jones J, Avants J, Bringolf R and Black M. (2013) Enantioselective toxicity and bioaccumulation of fipronil in fathead minnows (*Pimephales promelas*) following water and sediment exposures. *Environmental Toxicology and Chemistry*, 32(1), 222-227.

Relevance

Score: 92.5

Rating: R

Reliability

Score: 80

Rating: R

Relevance points taken off for: Control response (7.5).  $100-7.5=92.5$

|  | <b>Baird 2013</b>                             | <b><i>P. promelas</i></b> |
|--|---|---------------------------|
| <b>Parameter</b>                             | <b>Value</b>                                  | <b>Comment</b>            |
| Test method cited                            | US EPA method 1000.0                          |                           |
| Phylum/subphylum                             | Chordata                                      |                           |
| Class  | Actinopterygii                                |                           |
| Order  | Cypriniformes                                 |                           |
| Family                                       | Cyprinidae                                    |                           |
| Genus  | <i>Pimephales</i>                             |                           |
| Species                                      | <i>promelas</i>                               |                           |
| Family native to North America?              | Yes   |                           |
| Age/size at start of test/growth phase       | Larva   |                           |
| Source of organisms                          | US EPA, Region 5 Laboratory, Cincinnati, Ohio |                           |
| Have organisms been exposed to contaminants? | No  |                           |
| Animals acclimated and disease-free?         | Yes   |                           |
| Animals randomized?                          | Not reported                                  |                           |
| Test vessels randomized?                     | Yes   |                           |
| Test duration                                | 7 d   |                           |
| Data for multiple times?                     | No  |                           |
| Effect 1                                     | Survival                                      |                           |
| Control response 1                           | Not reported                                  |                           |
| Effect 2                                     | Dry weight                                    |                           |
| Control response 2                           | Not reported                                  |                           |
| Temperature                                  | 25 ± 1 °C                                     |                           |
| Test type                                    | Static-renewal                                | 80% renewal daily         |
| Photoperiod/light intensity                  | 16l:8d/Not reported                           |                           |

|   | <b>Baird 2013</b>   | <b><i>P. promelas</i></b>      |
|---|---|--------------------------------|
| <b>Parameter</b>  | <b>Value</b>  | <b>Comment</b>                 |
| Dilution water  | Dechlorinated, UV-treated tap water   |                                |
| pH  | 7.3-8.0   |                                |
| Hardness  | Not reported  |                                |
| Alkalinity  | Not reported  |                                |
| Conductivity  | Not reported  |                                |
| Dissolved Oxygen  | >4 mg/L   | >48 %                          |
| Feeding   | <i>Artemia nauplii</i> twice daily  | Not fed final 12 h of exposure |
| Purity of test substance  | 97.8 %  |                                |
| Concentrations measured?  | Yes   |                                |
| Measured is what % of nominal?  | 5 %   |                                |
| Toxicity values calculated based on nominal or measured concentrations? | Nominal   |                                |
| Chemical method documented?   | GCMS  |                                |
| Concentration of carrier (if any) in test solutions                     | Acetone, 0.1% (1 mL/L)  |                                |
| Concentration 1 Nom; Meas (µg/L)  | 50; Not reported  | 4 reps, 10/rep                 |
| Concentration 2 Nom; Meas (µg/L)  | 100; Not reported   |                                |
| Concentration 3 Nom; Meas (µg/L)  | 200; Not reported   |                                |
| Concentration 4 Nom; Meas (µg/L)  | 400; Not reported   |                                |
| Concentration 5 Nom; Meas (µg/L)  | 800; Not reported   |                                |
| Control   | Negative: 0; 0<br>Solvent: 0; 0   |                                |
| LC <sub>50</sub> (95% CI) (µg/L)  | Racemate: 208 (191-224)<br>(+) enantiomer: 227 (201-243)<br>(-) enantiomer: 365 (333-397) | Method: Spearman-Kärber method |

Notes: “(-) enantiomer being less toxic in waterborne exposures than both the racemate and the (+) enantiomer.” Regression plots of growth vs. concentration indicate that “relatively small increases in [enantiomers and racemate] concentrations can result in larger increases in toxicity.”

Solubility (S) of fipronil = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Measured concentrations (3), Exposure Hardness (2), Alkalinity (2), Conductivity (2), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100-13=87

Acceptability: Control response (9), Carrier solvent (4), Hardness (2), Alkalinity (2), Dissolved oxygen (6), Conductivity (1), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1). Total: 100-27 =73

**Reliability score: mean(87,73)=80**

## Water Toxicity Data Summary

*R. subcapitata*  
Fipronil desulfinyl  
MB46513

Hoberg JR (1993a) MB46513-Toxicity to the freshwater green alga, *Selenastrum capricornutum*. Springborn Laboratories, Inc., Wareham, Massachusetts. Laboratory study number 10566.0492.6243.430. Submitted to Rhone-Poulenc Ag Company, Research Triangle, North Carolina. USEPA MRID 43279705.

Relevance  
Score: 100  
Rating: R

Reliability  
Score: 95.5  
Rating: R

Relevance points taken off for: none.

|  | <b>Hoberg 1993</b>               | <b><i>R. subcapitata</i></b>   |
|--|----------------------------------|--|
| <b>Parameter</b>                             | <b>Value</b>                     | <b>Comment</b>   |
| Test method cited                            | FIFRA Guidelines 122-2 and 123-2 |  |
| Phylum/subphylum                             | Chlorophyta                      |  |
| Class  | Chlorophyceae                    |  |
| Order  | Sphaeropleales                   |  |
| Family                                       | Selenastraceae                   |  |
| Genus  | <i>Raphidocelis</i>              |  |
| Species                                      | <i>subcapitata</i>               |  |
| Family native to North America?              | Yes                              |  |
| Age/size at start of test/growth phase       | Not reported                     |  |
| Source of organisms                          | Laboratory cultures              |  |
| Have organisms been exposed to contaminants? | No                               |  |
| Animals acclimated and disease-free?         | Yes                              |  |
| Animals randomized?                          | Not reported                     | Given organism size and presence in growth medium, it is assumed that aliquots are inherently randomly |
| Test vessels randomized?                     | Not reported                     |  |
| Test duration                                | 5 d                              |  |
| Data for multiple times?                     | No                               |  |
| Effect 1                                     | Cell density                     |  |
| Control response 1                           | Mean: $130 \times 10^4$ cells/mL |  |

|   | <b>Hoberg 1993</b>   | <b><i>R. subcapitata</i></b>                              |
|---|--|---|
| <b>Parameter</b>  | <b>Value</b>   | <b>Comment</b>  |
| Temperature   | 24.5 ± 0.5 °C  |   |
| Test type   | Static   |   |
| Photoperiod/light intensity   | Continuous/ 3200-4800 lux  |   |
| Dilution water  | Freshwater growth medium<br>(Marine Biological<br>Laboratory medium) | Made with distilled<br>water                              |
| pH  | 7.5  |   |
| Feeding   | Growth medium  |   |
| Purity of test substance  | 98.6 %   |   |
| Concentrations measured?  | Yes  |   |
| Measured is what % of nominal?  | 70-108 %   |   |
| Toxicity values calculated based on nominal or measured concentrations? | Measured   |   |
| Chemical method documented?   | HPLC   |   |
| Concentration of carrier (if any) in test solutions                     | 0.1 mL/L acetone   |   |
| Concentration 1 Nom; Meas (µg/L)  | 13; 12   | 3 reps, 3 x 10 <sup>3</sup><br>cells/rep                  |
| Concentration 2 Nom; Meas (µg/L)  | 25; 27   |   |
| Concentration 3 Nom; Meas (µg/L)  | 50; 45   |   |
| Concentration 4 Nom; Meas (µg/L)  | 100; 72  |   |
| Concentration 5 Nom; Meas (µg/L)  | 200; 140   |   |
| Concentration 6 Nom; Meas (µg/L)  | 400; 330   |   |
| Control   | Solvent: 0; 0<br>Negative: 0; 0                                      |   |
| EC <sub>50</sub> (95% CI) (µg/L)  | 65 (24-180)  | Method: Linear<br>regression                              |
| NOEC  | <12  | Method: Williams'<br>Test<br>p: 0.05<br>MSD: not reported |
| LOEC  | 12   |   |
| % control at NOEC   | Not calculable   |   |
| % control at LOEC   | 82 %   | 107 (tmt) / 130<br>(mean controls) =<br>82 %              |

Notes:

Solubility (S) value for fipronil desulfinyl (MB 46513) = 950 µg/L, 2S = 1900 µg/L.

Reliability points were not taken off for water quality parameters (hardness, alkalinity, conductivity) because there is no guidance for these parameters in the test guidelines for

algal/plant studies, the growth medium used requires distilled water, and the medium is presumably appropriate for the test species because a specific culture media was used.

Reliability points taken off for:

Documentation: Minimum significant difference (2). Total:  $100-2=98$

Acceptability: Size/age (3), Random design (2), Minimum significant difference (1), % control at NOEC (1). Total:  $100-7=93$

**Reliability score: mean(98, 93)=95.5**

## Water Toxicity Data Summary

*Serratella micheneri*

Fipronil

MB46030

Weston DP and Lydy MJ. (2014) Toxicity of the insecticide fipronil and its degradates to benthic macroinvertebrates of urban streams. *Environmental science & technology*, 48(2), 1290-1297.

Relevance

Score: 90

Rating: R

Reliability

Score: 85.5

Rating: R

Relevance points taken off for: Standard method (10). 100-10=90

|  | <b>Weston 2014</b>  | <i>S. micheneri</i> |
|--|---|---------------------|
| <b>Parameter</b>                             | <b>Value</b>  | <b>Comment</b>      |
| Test method cited                            | Not reported  |                     |
| Phylum/subphylum                             | Anthropoda  |                     |
| Class  | Insecta   |                     |
| Order  | Ephemeroptera   |                     |
| Family                                       | Baetidae  |                     |
| Genus  | <i>Serratella</i>   |                     |
| Species                                      | <i>micheneri</i>  |                     |
| Family native to North America?              | Yes   |                     |
| Age/size at start of test/growth phase       | Not reported  |                     |
| Source of organisms                          | Urban waterbodies with minimal development in Northern California |                     |
| Have organisms been exposed to contaminants? | Not reported  |                     |
| Animals acclimated and disease-free?         | 24 h  |                     |
| Animals randomized?                          | Not reported  |                     |
| Test vessels randomized?                     | Not reported  |                     |
| Test duration                                | 48 h  |                     |
| Data for multiple times?                     | Not reported  |                     |
| Effect 1                                     | Survival  |                     |
| Control response 1                           | 100 %   |                     |
| Effect 2                                     | Immobilization (ability to swim)                                  |                     |
| Control response 2                           | Not reported  |                     |
| Temperature                                  | 23 °C   |                     |
| Test type                                    | Static  |                     |
| Photoperiod/light intensity                  | 16l:8d; Not reported  |                     |

|   | <b>Weston 2014</b>   | <i>S. micheneri</i>                                     |
|---|--|---|
| <b>Parameter</b>  | <b>Value</b>   | <b>Comment</b>  |
| Dilution water  | Milli-Q purified, deionized watermade moderately hard by addition of salts | *According to EPA 821-R-02-012                          |
| pH  | Not reported*  |   |
| Hardness  | Not reported*  |   |
| Alkalinity  | Not reported*  |   |
| Conductivity  | Not reported*  |   |
| Dissolved Oxygen  | Not reported*  |   |
| Feeding   | Not reported   |   |
| Purity of test substance  | 99.50 %  | Not reported but author verified from chemical supplier |
| Concentrations measured?  | Yes  |   |
| Measured is what % of nominal?  | 66-113%  |   |
| Toxicity values calculated based on nominal or measured concentrations? | Measured   |   |
| Chemical method documented?   | GC   |   |
| Concentration of carrier (if any) in test solutions                     | Acetone, <36 µg/L  |   |
| Concentration 1 Nom; Meas (µg/L)  | Not reported; 4-7 concentrations tested; dilution factor of 2              | 3 reps, 4-6/rep   |
| Control   | Negative: 0; 0<br>Solvent: 0; 0  |   |
| LC <sub>50</sub> (95% CI) (µg/L)  | 0.589 (0.478-0.742)  | Method: Probit  |
| EC <sub>50</sub> (95% CI) (µg/L)  | >722   | Method: Probit  |

Notes: Points were not deducted for lack of water quality parameter reporting because water was prepared according to EPA 821-R-02-012 and measured during study (but not reported).

Solubility (S) of fipronil = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Organism life stage/size (5), Nominal concentrations (3), Measured concentrations (3), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100-15 =85

Acceptability: Standard method (5), Organisms randomized (1), Temperature variation (3), Random design (2), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1). Total: 100-14 =86

**Reliability score: mean(85,86)=85.5**

## Water Toxicity Data Summary

*Serratella micheneri*

Fipronil sulfone

MB46136

Weston DP and Lydy MJ. (2014) Toxicity of the insecticide fipronil and its degradates to benthic macroinvertebrates of urban streams. *Environmental science & technology*, 48(2), 1290-1297.

Relevance

Score: 90

Rating: R

Reliability

Score: 85.5

Rating: R

Relevance points taken off for: Standard method (10). 100-10=90

|  | <b>Weston 2014</b>  | <i>S. micheneri</i> |
|--|---|---------------------|
| <b>Parameter</b>                             | <b>Value</b>  | <b>Comment</b>      |
| Test method cited                            | Not reported  |                     |
| Phylum/subphylum                             | Anthropoda  |                     |
| Class  | Insecta   |                     |
| Order  | Ephemeroptera   |                     |
| Family                                       | Baetidae  |                     |
| Genus  | <i>Serratella</i>   |                     |
| Species                                      | <i>micheneri</i>  |                     |
| Family native to North America?              | Yes   |                     |
| Age/size at start of test/growth phase       | Not reported  |                     |
| Source of organisms                          | Urban waterbodies with minimal development in Northern California |                     |
| Have organisms been exposed to contaminants? | Not reported  |                     |
| Animals acclimated and disease-free?         | 24 h  |                     |
| Animals randomized?                          | Not reported  |                     |
| Test vessels randomized?                     | Not reported  |                     |
| Test duration                                | 48 h  |                     |
| Data for multiple times?                     | Not reported  |                     |
| Effect 1                                     | Survival  |                     |
| Control response 1                           | 95 %  |                     |
| Effect 2                                     | Immobilization (ability to swim)                                  |                     |
| Control response 2                           | Not reported  |                     |
| Temperature                                  | 23 °C   |                     |
| Test type                                    | Static  |                     |
| Photoperiod/light intensity                  | 16l:8d; Not reported  |                     |

|   | <b>Weston 2014</b>   | <i>S. micheneri</i>                                     |
|---|--|---|
| <b>Parameter</b>  | <b>Value</b>   | <b>Comment</b>  |
| Dilution water  | Milli-Q purified, deionized watermade moderately hard by addition of salts | *According to EPA 821-R-02-012                          |
| pH  | Not reported*  |   |
| Hardness  | Not reported*  |   |
| Alkalinity  | Not reported*  |   |
| Conductivity  | Not reported*  |   |
| Dissolved Oxygen  | Not reported*  |   |
| Feeding   | Not reported   |   |
| Purity of test substance  | 99.3 %   | Not reported but author verified from chemical supplier |
| Concentrations measured?  | Yes  |   |
| Measured is what % of nominal?  | 66-113%  |   |
| Toxicity values calculated based on nominal or measured concentrations? | Measured   |   |
| Chemical method documented?   | GC   |   |
| Concentration of carrier (if any) in test solutions                     | Acetone, <36 µg/L  |   |
| Concentration 1 Nom; Meas (µg/L)  | Not reported; 4-7 concentrations tested; dilution factor of 2              | 3 reps, 4-6/rep   |
| Control   | Negative: 0; 0<br>Solvent: 0; 0  |   |
| LC <sub>50</sub> (95% CI) (µg/L)  | 0.331 (0.257-0.426)  | Method: Probit  |
| EC <sub>50</sub> (95% CI) (µg/L)  | 0.159 (0.106-0.214)  | Method: Probit  |

Notes: Points were not deducted for lack of water quality parameter reporting because water was prepared according to EPA 821-R-02-012 and measured during study (but not reported).

Solubility (S) value for fipronil sulfone (MB 46136) = 160 µg/L, 2S = 320 µg/L.

Reliability points taken off for:

Documentation: Organism life stage/size (5), Nominal concentrations (3), Measured concentrations (3), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100-15 =85

Acceptability: Standard method (5), Organisms randomized (1), Temperature variation (3), Random design (2), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1). Total: 100-14 =86

**Reliability score: mean(85,86)=85.5**

## Water Toxicity Data Summary

*Simulium vittatum*

Fipronil

MB 46030

Overmyer JP, Mason BN and Armbrust KL. (2005) Acute toxicity of imidacloprid and fipronil to a nontarget aquatic insect, *Simulium vittatum* Zetterstedt cytospecies IS-7. *Bulletin of environmental contamination and toxicology*, 74(5), 872-879.

Relevance

Score: 100

Rating: R

Reliability

Score: 87.5

Rating: R

Relevance points taken off for: none.

|  | <b>Overmyer et al. 2005</b>            | <i>S. vittatum</i> |
|--|--|--------------------|
| <b>Parameter</b>                             | <b>Value</b>                           | <b>Comment</b>     |
| Test method cited                            | Overmyer 2003                          |                    |
| Phylum/subphylum                             | Arthropoda                             |                    |
| Class  | Insecta                                |                    |
| Order  | Diptera                                |                    |
| Family                                       | Simuliidae                             |                    |
| Genus  | <i>Simulium</i>                        |                    |
| Species                                      | <i>vittatum</i>                        |                    |
| Family native to North America?              | Yes                                    |                    |
| Age/size at start of test/growth phase       | 5 <sup>th</sup> instar larvae          |                    |
| Source of organisms                          | University of Georgia, Athens, Georgia |                    |
| Have organisms been exposed to contaminants? | No                                     |                    |
| Animals acclimated and disease-free?         | Yes                                    |                    |
| Animals randomized?                          | Not reported                           |                    |
| Test vessels randomized?                     | Not reported                           |                    |
| Test duration                                | 48 h                                   |                    |
| Data for multiple times?                     | No                                     |                    |
| Effect 1                                     | Survival                               |                    |
| Control response 1                           | >96 %                                  |                    |
| Temperature                                  | 20 ± 1 °C                              |                    |
| Test type                                    | Static                                 |                    |
| Photoperiod/light intensity                  | 16l:8d                                 |                    |
| Dilution water                               | Moderately hard water                  | Weber 1993         |
| pH   | 7.3-7.7                                |                    |

|   | <b>Overmyer et al. 2005</b>             | <i>S. vittatum</i>              |
|---|---|---------------------------------|
| <b>Parameter</b>  | <b>Value</b>                            | <b>Comment</b>                  |
| Hardness  | 92.0 mg/L CaCO <sub>3</sub>             |                                 |
| Alkalinity  | 66.7 mg/L CaCO <sub>3</sub>             |                                 |
| Conductivity  | 273-275 umhos/cm                        |                                 |
| Dissolved Oxygen  | 8.8-8.9 mg/L                            |                                 |
| Feeding   | 5 mL food suspension to<br>140 mL water |                                 |
| Purity of test substance  | >98 %                                   |                                 |
| Concentrations measured?  | Yes                                     |                                 |
| Measured is what % of nominal?  | 56-83 %                                 |                                 |
| Toxicity values calculated based on nominal or measured concentrations? | Measured                                |                                 |
| Chemical method documented?   | Cited reference followed                |                                 |
| Concentration of carrier (if any) in test solutions                     | Acetone, concentration not reported     |                                 |
| Concentration 1 Nom; Meas (µg/L)  | 0.06; 0.05                              | 3 reps, number not reported/rep |
| Concentration 2 Nom; Meas (µg/L)  | 0.13; 0.10                              |                                 |
| Concentration 3 Nom; Meas (µg/L)  | 0.25; 0.14                              |                                 |
| Concentration 4 Nom; Meas (µg/L)  | 0.50; 0.30                              |                                 |
| Concentration 5 Nom; Meas (µg/L)  | 1.00; 0.55                              |                                 |
| Concentration 6 Nom; Meas (µg/L)  | 2.00; 1.19                              |                                 |
| Control   | Negative<br>Solvent                     |                                 |
| LC <sub>50</sub> (95% CI) (µg/L)  | 0.19 (0.16-0.21)                        | Method: probit                  |

Notes:

Solubility (S) of fipronil = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Statistical significance (2), Significance level (2), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100-8 =92

Acceptability: Measured concentrations within 20% nominal (4), Carrier solvent (4), Organisms randomized (1), Feeding (3), Random design (2), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1). Total: 100-17 =83

**Reliability score: mean(92, 83)=87.5**

## Water Toxicity Data Summary

*Taenionema* sp.  
Fipronil sulfone  
MB46136

Weston DP and Lydy MJ. (2014) Toxicity of the insecticide fipronil and its degradates to benthic macroinvertebrates of urban streams. *Environmental science & technology*, 48(2), 1290-1297.

Relevance  
Score: 75  
Rating: L

Reliability  
Score: 80  
Rating: R

Relevance points taken off for: Standard method (10), Toxicity value (15). 100-25=75

|  | <b>Weston 2014</b>  | <i>E. excrucians</i> |
|--|---|----------------------|
| <b>Parameter</b>                             | <b>Value</b>  | <b>Comment</b>       |
| Test method cited                            | Not reported  |                      |
| Phylum/subphylum                             | Anthropoda  |                      |
| Class  | Insecta   |                      |
| Order  | Plecoptera  |                      |
| Family                                       | Taeniopterygidae  |                      |
| Genus  | <i>Taenionema</i>   |                      |
| Species                                      | sp.   |                      |
| Family native to North America?              | Yes   |                      |
| Age/size at start of test/growth phase       | Not reported  |                      |
| Source of organisms                          | Urban waterbodies with minimal development in Northern California |                      |
| Have organisms been exposed to contaminants? | Not reported  |                      |
| Animals acclimated and disease-free?         | 24 h  |                      |
| Animals randomized?                          | Not reported  |                      |
| Test vessels randomized?                     | Not reported  |                      |
| Test duration                                | 96 h  |                      |
| Data for multiple times?                     | Not reported  |                      |
| Effect 1                                     | Survival  |                      |
| Control response 1                           | 100 %   |                      |
| Effect 2                                     | Immobilization (ability to cling)                                 |                      |
| Control response 2                           | Not reported  |                      |
| Temperature                                  | 8 °C  |                      |
| Test type                                    | Static  |                      |
| Photoperiod/light intensity                  | 16l:8d; Not reported  |                      |

|   | <b>Weston 2014</b>   | <b><i>E. excrucians</i></b>                             |
|---|--|---|
| <b>Parameter</b>  | <b>Value</b>   | <b>Comment</b>  |
| Dilution water  | Milli-Q purified, deionized watermade moderately hard by addition of salts | *According to EPA 821-R-02-012                          |
| pH  | Not reported*  |   |
| Hardness  | Not reported*  |   |
| Alkalinity  | Not reported*  |   |
| Conductivity  | Not reported*  |   |
| Dissolved Oxygen  | Not reported*  |   |
| Feeding   | Not reported   |   |
| Purity of test substance  | 99.3 %   | Not reported but author verified from chemical supplier |
| Concentrations measured?  | Yes  |   |
| Measured is what % of nominal?  | 66-113%  |   |
| Toxicity values calculated based on nominal or measured concentrations? | Measured   |   |
| Chemical method documented?   | GC   |   |
| Concentration of carrier (if any) in test solutions                     | Acetone, <36 µg/L  |   |
| Concentration 1 Nom; Meas (µg/L)  | Not reported; 4-7 concentrations tested; dilution factor of 2              | 3 reps, 4-6/rep   |
| Control   | Negative: 0; 0<br>Solvent: 0; 0  |   |
| LC <sub>50</sub> (95% CI) (µg/L)  | >0.261   | Method: Probit  |
| EC <sub>50</sub> (95% CI) (µg/L)  | 0.0959 (0.0621-0.126)  | Method: Probit  |

Notes: Points were not deducted for lack of water quality parameter reporting because water was prepared according to EPA 821-R-02-012 and measured during study (but not reported).

Solubility (S) value for fipronil sulfone (MB 46136) = 160 µg/L, 2S = 320 µg/L.

Reliability points taken off for:

Documentation: Organism life stage/size (5), Nominal concentrations (3), Measured concentrations (3), Minimum significant difference (2), % control at NOEC/LOEC (2), Point estimates (8). Total: 100-23 =77

Acceptability: Standard method (5), Organisms randomized (1), Temperature variation (3), Random design (2), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1), Point estimates (3). Total: 100-17 =83

**Reliability score: mean(77,83)=80**

*Appendix A2 – Aqueous Toxicity Studies rated RL, LR, LL*

## Water Toxicity Data Summary

*Aedes aegypti*

Fipronil

MB 46030

Ali A, Nayar JK and Gu WD. (1998) Toxicity of a phenyl pyrazole insecticide, fipronil, to mosquito and chironomid midge larvae in the laboratory. *Journal of the American Mosquito Control Association*, 14(2), 216-218.

Relevance

Score: 92.5

Rating: R

Reliability

Score: 60.5

Rating: L

Relevance points taken off for: Control response (7.5).  $100-7.5=92.5$

|  | <b>Ali 1998</b>   | <i>A. aegypti</i> |
|--|---|-------------------|
| <b>Parameter</b>                             | <b>Value</b>  | <b>Comment</b>    |
| Test method cited                            | Mulla MS and Khasawinah AM. (1969) Laboratory and field evaluation of larvicides against chironomid midges. <i>Journal of Economic Entomology</i> , 62(1), 37-41. |                   |
| Phylum/subphylum                             | Arthropoda  |                   |
| Class  | Insecta   |                   |
| Order  | Diptera   |                   |
| Family                                       | Culicidae   |                   |
| Genus  | <i>Aedes</i>  |                   |
| Species                                      | <i>aegypti</i>  |                   |
| Family native to North America?              | Yes   |                   |
| Age/size at start of test/growth phase       | 4 <sup>th</sup> instar  |                   |
| Source of organisms                          | Florida Medical Entomology Laboratory, University of Florida, Vero Beach, Florida   |                   |
| Have organisms been exposed to contaminants? | No  |                   |
| Animals acclimated and disease-free?         | Yes   |                   |
| Animals randomized?                          | Not reported  |                   |
| Test vessels randomized?                     | Not reported  |                   |
| Test duration                                | 48 h  |                   |
| Data for multiple times?                     | 24, 48 h  |                   |
| Effect 1                                     | Survival  |                   |

|   | <b>Ali 1998</b>                            | <i>A. aegypti</i>                  |
|---|--|------------------------------------|
| <b>Parameter</b>  | <b>Value</b>                               | <b>Comment</b>                     |
| Control response 1  | Not reported                               |                                    |
| Temperature   | 26 ± 2 °C                                  |                                    |
| Test type   | Static                                     |                                    |
| Photoperiod/light intensity   | 14:10d                                     |                                    |
| Dilution water  | Tap water                                  |                                    |
| pH  | Not reported                               |                                    |
| Hardness  | Not reported                               |                                    |
| Alkalinity  | Not reported                               |                                    |
| Conductivity  | Not reported                               |                                    |
| Dissolved Oxygen  | Not reported                               |                                    |
| Feeding   | 1 mL 1% beef liver plus yeast (1:1) daily  |                                    |
| Purity of test substance  | 97.1 %                                     |                                    |
| Concentrations measured?  | Not reported                               |                                    |
| Measured is what % of nominal?  | Not reported                               |                                    |
| Toxicity values calculated based on nominal or measured concentrations? | Not reported                               |                                    |
| Chemical method documented?   | Not reported                               |                                    |
| Concentration of carrier (if any) in test solutions                     | Acetone, concentration not reported        |                                    |
| Concentration 1 Nom; Meas (µg/L)  | 6-7 concentrations tested but not reported | 3 reps, 20/rep                     |
| Control   | Negative                                   |                                    |
| LC <sub>50</sub> (95% CI) (µg/L)  | 0.00154 (0.00143-0.00165)                  | Method: log-dose-probit regression |

Notes:

Solubility (S) of fipronil = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Analytical method (4), Nominal concentrations (3), Measured concentrations (3), Hardness (2), Alkalinity (2), Dissolved oxygen (4), Conductivity (2), pH (3), Statistical significance (2), Significance level (2), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100-31 =69

Acceptability: Control response (9), Measured concentrations within 20% nominal (4), Concentrations not > 2x solubility (4), Carrier solvent (4), Organisms randomized (1), Feeding (3), Hardness (2), Alkalinity (2), Dissolved oxygen (6), Conductivity (1), pH (2), Random design (2), Dilution factor (2), Hypothesis tests (3), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1). Total: 100-48 =52

**Reliability score: mean(69, 52)=60.5**

## Water Toxicity Data Summary

*Aedes aegypti*

Fipronil

MB46030

Chaton PF, Ravanel P, Meyran JC and Tissut M. (2001) The toxicological effects and bioaccumulation of fipronil in larvae of the mosquito *Aedes aegypti* in aqueous medium. *Pesticide Biochemistry and Physiology*, 69(3), 183-188.

Relevance

Score: 100

Rating: R

Reliability

Score: 60

Rating: L

Relevance points taken off for: none.

|  | <b>Chaton 2001</b>     | <i>A. aegypti</i> |
|--|------------------------|-------------------|
| <b>Parameter</b>                             | <b>Value</b>           | <b>Comment</b>    |
| Test method cited                            | Not reported           |                   |
| Phylum/subphylum                             | Arthropoda             |                   |
| Class  | Insecta                |                   |
| Order  | Diptera                |                   |
| Family                                       | Culicidae              |                   |
| Genus  | <i>Aedes</i>           |                   |
| Species                                      | <i>Aegypti</i>         |                   |
| Family native to North America?              | Yes                    |                   |
| Age/size at start of test/growth phase       | 4 <sup>th</sup> instar |                   |
| Source of organisms                          | Laboratory cultures    |                   |
| Have organisms been exposed to contaminants? | No                     |                   |
| Animals acclimated and disease-free?         | Yes                    |                   |
| Animals randomized?                          | Not reported           |                   |
| Test vessels randomized?                     | Not reported           |                   |
| Test duration                                | 48 h                   |                   |
| Data for multiple times?                     | 24, 48 h               |                   |
| Effect 1                                     | Survival               |                   |
| Control response 1                           | 100 %                  |                   |
| Temperature                                  | Not reported           |                   |
| Test type                                    | Static                 |                   |
| Photoperiod/light intensity                  | Not reported           |                   |
| Dilution water                               | Tap water              |                   |
| Feeding                                      | Not fed                |                   |
| Purity of test substance                     | 99.9 %                 |                   |

|   | <b>Chaton 2001</b>   | <i>A. aegypti</i>  |
|---|--|--------------------|
| <b>Parameter</b>  | <b>Value</b>   | <b>Comment</b>     |
| Concentrations measured?  | Not reported   |                    |
| Measured is what % of nominal?  | Not reported   |                    |
| Toxicity values calculated based on nominal or measured concentrations? | Not reported   |                    |
| Chemical method documented?   | Not reported   |                    |
| Concentration of carrier (if any) in test solutions                     | Ethanol, concentration not reported                                    |                    |
| Concentration 1 Nom; Meas (µg/L)  | Concentrations not reported but graphs indicate range from 0.008-0.043 | 3 reps, 20/rep     |
| Control   | Negative: 0; 0   |                    |
| LC <sub>50</sub> (95% CI) (µg/L)  | 24 h: 0.0108 (24.8 nM)<br>48 h: 0.066 (15.1 nM)                        | Method: Log-probit |

Notes: Values reported in nM; conversion performed using Excel “=(X nM\*437.15\*1000)/10<sup>9</sup>”.

Solubility (S) of fipronil = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Analytical method (4), Nominal concentrations (3), Measured concentrations (3), Hardness (2), Alkalinity (2), Dissolved oxygen (4), Temperature (4), Conductivity (2), pH (3), Photoperiod (3), Statistical significance (2), Significance level (2), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100-38 =62

Acceptability: Standard method (5), Measured concentrations within 20% nominal (4), Carrier solvent (4), Organisms randomized (1), Hardness (2), Alkalinity (2), Dissolved oxygen (6), Temperature (6), Conductivity (1), pH (2), Photoperiod (2), Random design (2), Dilution factor (2), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1). Total: 100-42 =58

**Reliability score: mean(62, 58)=60**

## Water Toxicity Data Summary

*Aedes albopictus*

Fipronil

MB 46030

Ali A, Nayar JK and Gu WD. (1998) Toxicity of a phenyl pyrazole insecticide, fipronil, to mosquito and chironomid midge larvae in the laboratory. *Journal of the American Mosquito Control Association*, 14(2), 216-218.

Relevance

Score: 92.5

Rating: R

Reliability

Score: 60.5

Rating: L

Relevance points taken off for: Control response (7.5).  $100-7.5=92.5$

|  | <b>Ali 1998</b>   | <b><i>A. albopictus</i></b> |
|--|---|-----------------------------|
| <b>Parameter</b>                             | <b>Value</b>  | <b>Comment</b>              |
| Test method cited                            | Mulla MS and Khasawinah AM. (1969) Laboratory and field evaluation of larvicides against chironomid midges. <i>Journal of Economic Entomology</i> , 62(1), 37-41. |                             |
| Phylum/subphylum                             | Arthropoda  |                             |
| Class  | Insecta   |                             |
| Order  | Diptera   |                             |
| Family                                       | Culicidae   |                             |
| Genus  | <i>Aedes</i>  |                             |
| Species                                      | <i>albopictus</i>   |                             |
| Family native to North America?              | Yes   |                             |
| Age/size at start of test/growth phase       | 1 <sup>st</sup> and 4 <sup>th</sup> instar  |                             |
| Source of organisms                          | Florida Medical Entomology Laboratory, University of Florida, Vero Beach, Florida   |                             |
| Have organisms been exposed to contaminants? | No  |                             |
| Animals acclimated and disease-free?         | Yes   |                             |
| Animals randomized?                          | Not reported  |                             |
| Test vessels randomized?                     | Not reported  |                             |
| Test duration                                | 48 h  |                             |
| Data for multiple times?                     | 24, 48 h  |                             |
| Effect 1                                     | Survival  |                             |

|   | <b>Ali 1998</b>   | <b><i>A. albopictus</i></b>        |
|---|---|------------------------------------|
| <b>Parameter</b>  | <b>Value</b>  | <b>Comment</b>                     |
| Control response 1  | Not reported  |                                    |
| Temperature   | 26 ± 2 °C   |                                    |
| Test type   | Static  |                                    |
| Photoperiod/light intensity   | 14:10d  |                                    |
| Dilution water  | Tap water   |                                    |
| pH  | Not reported  |                                    |
| Hardness  | Not reported  |                                    |
| Alkalinity  | Not reported  |                                    |
| Conductivity  | Not reported  |                                    |
| Dissolved Oxygen  | Not reported  |                                    |
| Feeding   | 1 mL 1% beef liver plus yeast (1:1) daily   |                                    |
| Purity of test substance  | 97.1 %  |                                    |
| Concentrations measured?  | Not reported  |                                    |
| Measured is what % of nominal?  | Not reported  |                                    |
| Toxicity values calculated based on nominal or measured concentrations? | Not reported  |                                    |
| Chemical method documented?   | Not reported  |                                    |
| Concentration of carrier (if any) in test solutions                     | Acetone, concentration not reported   |                                    |
| Concentration 1 Nom; Meas (µg/L)  | 6-7 concentrations tested but not reported  | 3 reps, 20/rep                     |
| Control   | Negative  |                                    |
| LC <sub>50</sub> (95% CI) (µg/L)  | 1 <sup>st</sup> instar:<br>48 h: 0.0081 (0.0071-0.0090)<br><br>4 <sup>th</sup> instar:<br>48 h: 0.023 (0.015-0.032) | Method: log-dose-probit regression |

Notes: Solubility (S) of fipronil = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Analytical method (4), Nominal concentrations (3), Measured concentrations (3), Hardness (2), Alkalinity (2), Dissolved oxygen (4), Conductivity (2), pH (3), Statistical significance (2), Significance level (2), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100-31 =69

Acceptability: Control response (9), Measured concentrations within 20% nominal (4), Concentrations not > 2x solubility (4), Carrier solvent (4), Organisms randomized (1), Feeding (3), Hardness (2), Alkalinity (2), Dissolved oxygen (6), Conductivity (1), pH (2), Random design (2), Dilution factor (2), Hypothesis tests (3), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1). Total: 100-48 =52

**Reliability score: mean(69, 52)=60.5**

## Water Toxicity Data Summary

*Americamysis bahia*

Fipronil

MB46030

Machado MW. (1994) Fipronil-Chronic toxicity to mysids (*Mysidopsis bahia*) under flow-through conditions. Springborn Laboratories, Inc., Wareham, Massachusetts. Laboratory study number 10566.1294.6353.530. Submitted to Rhone-Poulenc Ag Company, Research Triangle, North Carolina. USEPA MRID 43681201.

Relevance

Score: 85

Rating: L

Reliability

Score: 88

Rating: R

Relevance points taken off for: Freshwater (15). 100-15=85

|  | <b>Machado 1995</b>            | <b><i>A. bahia</i></b>                |
|--|--------------------------------|---------------------------------------|
| <b>Parameter</b>                             | <b>Value</b>                   | <b>Comment</b>                        |
| Test method cited                            | FIFRA Guideline 72-4           |                                       |
| Phylum/subphylum                             | Arthropoda/Crustacea           |                                       |
| Class  | Malacostraca                   |                                       |
| Order  | Mysida                         |                                       |
| Family                                       | Mysidae                        |                                       |
| Genus  | <i>Americamysis</i>            |                                       |
| Species                                      | <i>bahia</i>                   |                                       |
| Family native to North America?              | Yes                            |                                       |
| Age/size at start of test/growth phase       | <24 h                          |                                       |
| Source of organisms                          | Laboratory cultures            |                                       |
| Have organisms been exposed to contaminants? | No                             |                                       |
| Animals acclimated and disease-free?         | Yes                            |                                       |
| Animals randomized?                          | Yes                            |                                       |
| Test vessels randomized?                     | Yes                            |                                       |
| Test duration                                | 28 d                           |                                       |
| Data for multiple times?                     | No                             |                                       |
| Effect 1                                     | Survival                       |                                       |
| Control response 1 (mean)                    | F <sub>0</sub> : 85 %          |                                       |
| Effect 2                                     | Reproductive success           | Offspring/female/<br>reproductive day |
| Control response 2 (mean)                    | F <sub>0</sub> : 0.35          |                                       |
| Effect 3                                     | Growth (length and dry weight) |                                       |

|   | <b>Machado 1995</b>  | <b><i>A. bahia</i></b>   |
|---|--|--|
| <b>Parameter</b>  | <b>Value</b>   | <b>Comment</b>   |
| Control response 3 (mean)   | F <sub>0</sub> male length: 7.3 mm<br>F <sub>0</sub> female length: 7.0 mm<br><br>F <sub>0</sub> male weight: 0.81 mg<br>F <sub>0</sub> female weight: 0.93 mg |  |
| Temperature   | 24.5 ± 0.5 °C  |  |
| Test type   | Flow through   |  |
| Photoperiod/light intensity   | 16l:8d/20-75 footcandles   |  |
| Dilution water  | Artificial seawater made with soft freshwater using commercial mix (hw-MARINEMIX)  | 26-27 ‰  |
| pH  | 8.2-8.3  |  |
| Hardness  | 20-40 mg/L CaCO <sub>3</sub>   |  |
| Dissolved Oxygen  | 7.2-8.7 mg/L   | 86-104 %   |
| Feeding   | Brine shrimp nauplii ( <i>Artemia salina</i> ) 2/d, Selco 1/d  |  |
| Purity of test substance  | 97.7 %   | Radiochemical purity   |
| Concentrations measured?  | Yes  |  |
| Measured is what % of nominal?  | 80-114 %   |  |
| Toxicity values calculated based on nominal or measured concentrations? | Measured   |  |
| Chemical method documented?   | HPLC-RAM   |  |
| Concentration of carrier (if any) in test solutions                     | Acetone, 520 mL/L  | Very high; see page 12: "52 mL of acetone [diluted] with distilled water to volume in a 100-mL volumetric flask" |
| Concentration 1 Nom; Meas (µg/L)  | 0.0044; 0.0050   | 2 reps, 30/rep   |
| Concentration 2 Nom; Meas (µg/L)  | 0.0088; 0.0077   |  |
| Concentration 3 Nom; Meas (µg/L)  | 0.018; 0.015   |  |
| Concentration 4 Nom; Meas (µg/L)  | 0.035; 0.028   |  |
| Concentration 5 Nom; Meas (µg/L)  | 0.070; 0.057   |  |
| Control   | Negative: 0; 0<br>Solvent: 0; 0  |  |
| NOEC  | 0.0077   | Method: Williams' Test<br>p: 0.05  |

|                           | <b>Machado 1995</b>              | <b><i>A. bahia</i></b>   |
|---------------------------|----------------------------------|--|
| <b>Parameter</b>          | <b>Value</b>                     | <b>Comment</b>   |
|                           |                                  | MSD: Not reported<br>Based on male body length                     |
| LOEC                      | 0.015                            |  |
| MATC (GeoMean NOEC, LOEC) | 0.011                            |  |
| % control at NOEC         | F <sub>0</sub> male length: 99 % | F <sub>0</sub> male length: 7.2 (tmt) / 7.3 (mean controls) = 99 % |
| % control at LOEC         | F <sub>0</sub> male length: 95 % | F <sub>0</sub> male length: 6.9 (tmt) / 7.3 (mean controls) = 95 % |

Notes: Dilution water TOC average = 0.49 mg/L

Solubility (S) of fipronil = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Alkalinity (2), Minimum significant difference (2), Point estimates (8). Total: 100- 12=88

Acceptability: Carrier solvent (4), Alkalinity (2), Adequate replication (2), Minimum significant difference (1), Point estimates (3). Total: 100-12 =88

**Reliability score: mean(88, 88)=88**

## Water Toxicity Data Summary

*Americamysis bahia*

Fipronil

MB46030

Machado MW. (1994) MB 46030-Acute toxicity to mysids (*Mysidopsis bahia*) under static conditions. Springborn Laboratories, Inc., Wareham, Massachusetts. Laboratory study number 10566.0394.6340.510. Submitted to Rhone-Poulenc Ag Company, Research Triangle, North Carolina. USEPA MRID 43279701. CA DPR 157286.

Relevance

Score: 85

Rating: L

Reliability

Score: 88.5

Rating: R

Relevance points taken off for: Freshwater (15). 100-15=85

|  | <b>Machado 1994</b>                        | <b><i>A. bahia</i></b> |
|--|--|------------------------|
| <b>Parameter</b>                             | <b>Value</b>                               | <b>Comment</b>         |
| Test method cited                            | FIFRA Guideline 72-3                       |                        |
| Phylum/subphylum                             | Arthropoda/Crustacea                       |                        |
| Class  | Malacostraca                               |                        |
| Order  | Mysida                                     |                        |
| Family                                       | Mysidae                                    |                        |
| Genus  | <i>Americamysis</i>                        |                        |
| Species                                      | <i>bahia</i>                               |                        |
| Family native to North America?              | Yes  |                        |
| Age/size at start of test/growth phase       | <24 h                                      |                        |
| Source of organisms                          | Aquatic Biosystems, Fort Collins, Colorado |                        |
| Have organisms been exposed to contaminants? | No   |                        |
| Animals acclimated and disease-free?         | Yes  |                        |
| Animals randomized?                          | Yes  |                        |
| Test vessels randomized?                     | Yes  |                        |
| Test duration                                | 96 h                                       |                        |
| Data for multiple times?                     | 24, 48, 72, 96 h                           |                        |
| Effect 1                                     | Mortality                                  |                        |
| Control response 1 (mean)                    | 2.5 %                                      |                        |
| Temperature                                  | 25.5 ± 0.5 °C                              |                        |
| Test type                                    | Static                                     |                        |
| Photoperiod/light intensity                  | 16l:8d/60 footcandles                      |                        |
| Dilution water                               | Filtered seawater                          | 30-32 ‰                |

|   | <b>Machado 1994</b>  | <b><i>A. bahia</i></b>   |
|---|--|--|
| <b>Parameter</b>  | <b>Value</b>   | <b>Comment</b>   |
| pH  | 7.7-7.8  |  |
| Dissolved Oxygen  | 5.1-6.9 mg/L   | 62-84 %<br>Not aerated   |
| Feeding   | Brine shrimp nauplii<br>( <i>Artemia salina</i> ) 1/d  |  |
| Purity of test substance  | 96.1 %   |  |
| Concentrations measured?  | Yes  |  |
| Measured is what % of nominal?  | 82-102 %   |  |
| Toxicity values calculated based on nominal or measured concentrations? | Measured   |  |
| Chemical method documented?   | HPLC   |  |
| Concentration of carrier (if any) in test solutions                     | 0.10 mL/L acetone  |  |
| Concentration 1 Nom; Meas (µg/L)  | 0.061; 0.062   | 2 reps, 20/rep   |
| Concentration 2 Nom; Meas (µg/L)  | 0.100; 0.097   |  |
| Concentration 3 Nom; Meas (µg/L)  | 0.170; 0.140   |  |
| Concentration 4 Nom; Meas (µg/L)  | 0.280; 0.240   |  |
| Concentration 5 Nom; Meas (µg/L)  | 0.470; 0.390   |  |
| Control   | Negative: 0; 0<br>Solvent: 0; 0  |  |
| LC <sub>50</sub> (95% CI) (µg/L)  | 24 h: >0.390<br>48 h: 0.170 (0.140-0.240) <sup>a</sup><br>72 h: 0.170 (0.140-0.240) <sup>a</sup><br>96 h: 0.140 (0.120-0.160) <sup>b</sup> | Method: <sup>a</sup> Nonlinear interpolation and binomial probability; <sup>b</sup> probit |
| NOEC  | 0.062  | Method: Not reported<br>p: Not reported<br>MSD: Not reported                               |
| LOEC  | 0.097  | Not reported; See Table 4  |
| MATC (GeoMean NOEC, LOEC)   | 0.078  |  |
| % control at NOEC   | 100 % survival   |  |
| % control at LOEC   | 90 % survival  |  |

Notes: Dilution water TOC average = <2.0 mg/L

Solubility (S) of fipronil = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Hardness (2), Alkalinity (2), Conductivity (2), Statistics method (5), Statistical significance (2), Significance level (2), Minimum significant difference (2). Total: 100- 15=85

Acceptability: Hardness (2), Alkalinity (2), Conductivity (1), Replicates (2), Minimum significant difference (1). Total:  $100 - 8 = 92$

**Reliability score: mean(85,92)=88.5**

## Water Toxicity Data Summary

*Americamysis bahia*

Fipronil sulfide

MB45950

Putt AE. (2000a) [<sup>14</sup>C]MB 45950-Acute toxicity to mysids (*Mysidopsis bahia*) under static acute conditions. Springborn Laboratories, Inc., Wareham, Massachusetts. Laboratory study number 10566.6547. Submitted to Rhone-Poulenc Ag Company, Research Triangle, North Carolina. USEPA MRID 45156302.

Relevance

Score: 85

Rating: L

Reliability

Score: 84

Rating: R

Relevance points taken off for: Freshwater (15). 100-15=85

|  | <b>Putt 2000c</b>         | <b><i>A. bahia</i></b> |
|--|---------------------------|------------------------|
| <b>Parameter</b>                             | <b>Value</b>              | <b>Comment</b>         |
| Test method cited                            | FIFRA 72-3                |                        |
| Phylum/subphylum                             | Arthropoda/Crustacea      |                        |
| Class  | Malacostraca              |                        |
| Order  | Mysida                    |                        |
| Family                                       | Mysidae                   |                        |
| Genus  | <i>Americamysis</i>       |                        |
| Species                                      | <i>bahia</i>              |                        |
| Family native to North America?              | Yes                       |                        |
| Age/size at start of test/growth phase       | Not reported              |                        |
| Source of organisms                          | Laboratory culture        |                        |
| Have organisms been exposed to contaminants? | No                        |                        |
| Animals acclimated and disease-free?         | 14 d                      |                        |
| Animals randomized?                          | Yes                       |                        |
| Test vessels randomized?                     | Yes                       |                        |
| Test duration                                | 96 h                      |                        |
| Data for multiple times?                     | 24, 48, 72, 96 h          |                        |
| Effect 1                                     | Survival                  |                        |
| Control response 1                           | 100 %                     |                        |
| Temperature                                  | 24.5 ± 0.5 °C             |                        |
| Test type                                    | Static                    |                        |
| Photoperiod/light intensity                  | 16l:8d/80-100 footcandles |                        |
| Dilution water                               | Filtered natural seawater | 32 ‰ salinity          |
| pH   | 7.9-8.1                   |                        |

|   | <b>Putt 2000c</b>                                  | <b>A. bahia</b>  |
|---|--|--|
| <b>Parameter</b>  | <b>Value</b>                                       | <b>Comment</b>   |
| Dissolved Oxygen  | 3.1-6.6 mg/L                                       | 45-97 %  |
| Feeding   | Brine shrimp nauplii ( <i>Artemia salina</i> ) 1/d |  |
| Purity of test substance  | 98.8 %   |  |
| Concentrations measured?  | Yes  |  |
| Measured is what % of nominal?  | 87-98 %  |  |
| Toxicity values calculated based on nominal or measured concentrations? | Measured   |  |
| Chemical method documented?   | HPLC   |  |
| Concentration of carrier (if any) in test solutions                     | Acetone, 0.100 mL/L                                |  |
| Concentration 1 Nom; Meas (µg/L)  | 0.0310; 0.0330                                     | 2 reps, 10/rep   |
| Concentration 2 Nom; Meas (µg/L)  | 0.0630; 0.0670                                     |  |
| Concentration 3 Nom; Meas (µg/L)  | 0.1300; 0.1200                                     |  |
| Concentration 4 Nom; Meas (µg/L)  | 0.2500; 0.2500                                     |  |
| Concentration 5 Nom; Meas (µg/L)  | 0.5000; 0.4900                                     |  |
| Control   | Negative: 0; 0<br>Solvent: 0; 0                    |  |
| LC <sub>50</sub> (95% CI) (µg/L)  | 0.077 (0.030-0.120)                                | Method: Nonlinear interpolation                              |
| NOEC  | 0.033  | Method: Not reported<br>p: Not reported<br>MSD: Not reported |
| LOEC  | 96 h: 0.067  | Not reported; See Table 4                                    |
| MATC (GeoMean NOEC, LOEC)   | 0.047  |  |
| % control at NOEC   | 100 %  | 100 (tmt) / 100 (mean controls) = 100 %                      |
| % control at LOEC   | 70 %   | 70 (tmt) / 100 (mean controls) = 70 %                        |

Notes:

Solubility value for fipronil sulfide (MB 45950) not available. Solubility (S) of fipronil parent compound (MB 46030) = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Hardness (2), Alkalinity (2), Conductivity (2), Statistical significance (2), Significance level (2), Minimum significant difference (2). Total: 100- 12=88

Acceptability: Organism age/size (3), Feeding (3), Hardness (2), Alkalinity (2), Dissolved oxygen (6), Conductivity (1), Adequate replication (2), Minimum significant difference (1).  
Total: 100-20 =80

**Reliability score: mean(88, 80)=84**

## Water Toxicity Data Summary

*Americamysis bahia*

Fipronil sulfone

MB46136

Lima W. (2000) [<sup>14</sup>C]MB 46136-Life-cycle toxicity test with mysids (*Mysidopsis bahia*). Springborn Laboratories, Inc., Wareham, Massachusetts. Laboratory study number 13726.6116. Submitted to Aventis CropScience, Research Triangle, North Carolina. USEPA MRID 45259203.

Relevance

Score: 85

Rating: L

Reliability

Score: 81.5

Rating: R

Relevance points taken off for: Freshwater (15). 100-15=85

|  | <b>Lima 2000</b>               | <b><i>A. bahia</i></b>                |
|--|--------------------------------|---------------------------------------|
| <b>Parameter</b>                             | <b>Value</b>                   | <b>Comment</b>                        |
| Test method cited                            | FIFRA Guideline 72-4           |                                       |
| Phylum/subphylum                             | Arthropoda/Crustacea           |                                       |
| Class  | Malacostraca                   |                                       |
| Order  | Mysida                         |                                       |
| Family                                       | Mysidae                        |                                       |
| Genus  | <i>Americamysis</i>            |                                       |
| Species                                      | <i>bahia</i>                   |                                       |
| Family native to North America?              | Yes                            |                                       |
| Age/size at start of test/growth phase       | <24 h                          |                                       |
| Source of organisms                          | Laboratory cultures            |                                       |
| Have organisms been exposed to contaminants? | No                             |                                       |
| Animals acclimated and disease-free?         | Yes                            |                                       |
| Animals randomized?                          | Yes                            |                                       |
| Test vessels randomized?                     | Not reported                   |                                       |
| Test duration                                | 28 d                           |                                       |
| Data for multiple times?                     | No                             |                                       |
| Effect 1                                     | Survival                       |                                       |
| Control response 1 (mean)                    | F <sub>0</sub> : 77 %          |                                       |
| Effect 2                                     | Reproductive success           | Offspring/female/<br>reproductive day |
| Control response 2 (mean)                    | F <sub>0</sub> : 1.62          |                                       |
| Effect 3                                     | Growth (length and dry weight) |                                       |

|   | <b>Lima 2000</b>   | <b>A. bahia</b>  |
|---|--|--|
| <b>Parameter</b>  | <b>Value</b>   | <b>Comment</b>   |
| Control response 3 (mean)   | F <sub>0</sub> male length: 7.6 mm<br>F <sub>0</sub> female length: 7.7 mm<br><br>F <sub>0</sub> male weight: 0.91 mg<br>F <sub>0</sub> female weight: 1.23 mg |  |
| Temperature   | 27 ± 1 °C  |  |
| Test type   | Flow through   |  |
| Photoperiod/light intensity   | 16l:8d/60-90 footcandles   |  |
| Dilution water  | Artificial seawater made with soft freshwater using commercial mix (hw-MARINEMIX)  | 26-28 ‰ salinity   |
| pH  | 8.1-8.3  |  |
| Conductivity  | 35,000-36,000 µS/cm  |  |
| Dissolved Oxygen  | 6.7 mg/L   | 72 %   |
| Feeding   | Brine shrimp nauplii ( <i>Artemia salina</i> ) 2/d, Selco 1/d  |  |
| Purity of test substance  | 97.7 %   |  |
| Concentrations measured?  | Yes  |  |
| Measured is what % of nominal?  | 70-84 %  |  |
| Toxicity values calculated based on nominal or measured concentrations? | Measured   |  |
| Chemical method documented?   | HPLC-RAM   |  |
| Concentration of carrier (if any) in test solutions                     | Not reported   |  |
| Concentration 1 Nom; Meas (µg/L)  | 0.0031; 0.0026   | 2 reps, 30/rep   |
| Concentration 2 Nom; Meas (µg/L)  | 0.0062; 0.0051   |  |
| Concentration 3 Nom; Meas (µg/L)  | 0.012; 0.0093  |  |
| Concentration 4 Nom; Meas (µg/L)  | 0.025; 0.019   |  |
| Concentration 5 Nom; Meas (µg/L)  | 0.050; 0.035   |  |
| Control   | Negative: 0; 0   |  |
| NOEC  | 0.0051   | Method: Williams' Test<br>p: 0.05<br>MSD: Not reported<br>Based dry weight |
| LOEC  | 0.0093   |  |
| MATC (GeoMean NOEC, LOEC)   | 0.0069   |  |
| % control at NOEC   | F <sub>0</sub> male length: 100 %<br>F <sub>0</sub> female length: 100 %   | F <sub>0</sub> male length: 7.6 (tmt) / 7.6 (mean                          |

|                   | <b>Lima 2000</b>   | <b>A. bahia</b>   |
|-------------------|--|---|
| <b>Parameter</b>  | <b>Value</b>   | <b>Comment</b>  |
|                   |  | controls) = 100 %<br><br>F <sub>0</sub> female length:<br>7.8 (tmt) / 7.8<br>(mean controls) =<br>100 %   |
| % control at LOEC | F <sub>0</sub> male length: 97 %<br>F <sub>0</sub> female length: 99 % | F <sub>0</sub> male length: 7.4<br>(tmt) / 7.6 (mean<br>controls) = 97 %<br><br>F <sub>0</sub> female length:<br>7.7 (tmt) / 7.8<br>(mean controls) =<br>99 % |

Notes: Dilution water TOC average = 0.72-0.60 mg/L

Solubility (S) value for fipronil sulfone (MB 46136) = 160 µg/L, 2S = 320 µg/L.

Reliability points taken off for:

Documentation: Hardness (2), Alkalinity (2), Statistical significance (2), Significance level (2), Minimum significant difference (2), Point estimates (8). Total: 100-18 =82

Acceptability: Measured concentrations within 20% nominal (4), Hardness (2), Alkalinity (2), Temperature tolerance (3), Random design (2), Adequate replication (2), Minimum significant difference (1), Point estimates (3). Total: 100-19 =81

**Reliability score: mean(82, 81)=81.5**

## Water Toxicity Data Summary

*Americamysis bahia*

Fipronil sulfone

MB46136

Putt AE. (2000b) [<sup>14</sup>C]MB 46136-Acute toxicity to mysids (*Mysidopsis bahia*) under static acute conditions. Springborn Laboratories, Inc., Wareham, Massachusetts. Laboratory study number 10566.6545. Submitted to Rhone-Poulenc Ag Company, Research Triangle, North Carolina. USEPA MRID 45156301.

Relevance

Score: 85

Rating: L

Reliability

Score: 87

Rating: R

Relevance points taken off for: Freshwater (15). 100-15=85

|  | <b>Putt 2000b</b>   | <b><i>A. bahia</i></b> |
|--|---|------------------------|
| <b>Parameter</b>                             | <b>Value</b>  | <b>Comment</b>         |
| Test method cited                            | FIFRA 72-3, Ecological Effects Test Guidelines OPPTS 850.1035 |                        |
| Phylum/subphylum                             | Arthropoda/Crustacea  |                        |
| Class  | Malacostraca  |                        |
| Order  | Mysida  |                        |
| Family                                       | Mysidae   |                        |
| Genus  | <i>Americamysis</i>   |                        |
| Species                                      | <i>bahia</i>  |                        |
| Family native to North America?              | Yes   |                        |
| Age/size at start of test/growth phase       | Not reported  |                        |
| Source of organisms                          | Laboratory culture  |                        |
| Have organisms been exposed to contaminants? | No  |                        |
| Animals acclimated and disease-free?         | 14 d  |                        |
| Animals randomized?                          | Yes   |                        |
| Test vessels randomized?                     | Yes   |                        |
| Test duration                                | 96 h  |                        |
| Data for multiple times?                     | 24, 48, 72, 96 h  |                        |
| Effect 1                                     | Survival  |                        |
| Control response 1                           | 100 %   |                        |
| Temperature                                  | 24.5 ± 0.5 °C   |                        |
| Test type                                    | Static  |                        |
| Photoperiod/light intensity                  | 16l:8d/80-100 footcandles                                     |                        |

|   | <b>Putt 2000b</b>                                     | <b><i>A. bahia</i></b>                                       |
|---|---|--|
| <b>Parameter</b>  | <b>Value</b>  | <b>Comment</b>   |
| Dilution water  | Filtered natural seawater                             | 32 ‰ salinity  |
| pH  | 7.9-8.1   |  |
| Dissolved Oxygen  | 4.4-6.6 mg/L  | 64-97 %  |
| Feeding   | Brine shrimp nauplii<br>( <i>Artemia salina</i> ) 1/d |  |
| Purity of test substance  | 99.7 %  |  |
| Concentrations measured?  | Yes   |  |
| Measured is what % of nominal?  | 87-98 %   |  |
| Toxicity values calculated based on nominal or measured concentrations? | Measured  |  |
| Chemical method documented?   | HPLC  |  |
| Concentration of carrier (if any) in test solutions                     | Acetone, 0.100 mL/L                                   |  |
| Concentration 1 Nom; Meas (µg/L)  | 0.0310; 0.0310  | 2 reps, 10/rep   |
| Concentration 2 Nom; Meas (µg/L)  | 0.0630; 0.0580  |  |
| Concentration 3 Nom; Meas (µg/L)  | 0.1300; 0.1200  |  |
| Concentration 4 Nom; Meas (µg/L)  | 0.2500; 0.2400  |  |
| Concentration 5 Nom; Meas (µg/L)  | 0.5000; 0.4300  |  |
| Control   | Negative: 0; 0<br>Solvent: 0; 0                       |  |
| LC <sub>50</sub> (95% CI) (µg/L)  | 0.056 (0.031-0.120)                                   | Method: Nonlinear interpolation                              |
| NOEC  | 0.031   | Method: Not reported<br>p: Not reported<br>MSD: Not reported |
| LOEC  | 96 h: 0.058   | Not reported; See Table 4                                    |
| MATC (GeoMean NOEC, LOEC)   | 0.042   |  |
| % control at NOEC   | 100 %   | 100 (tmt) / 100 (mean controls) = 100 %                      |
| % control at LOEC   | 45 %  | 45 (tmt) / 100 (mean controls) = 45 %                        |

Notes:

Solubility (S) value for fipronil sulfone (MB 46136) = 160 µg/L, 2S = 320 µg/L.

Reliability points taken off for:

Documentation: Hardness (2), Alkalinity (2), Conductivity (2), Statistical significance (2), Significance level (2), Minimum significant difference (2). Total:  $100 - 12 = 88$

Acceptability: Organism age/size (3), Feeding (3), Hardness (2), Alkalinity (2), Conductivity (1), Adequate replication (2), Minimum significant difference (1). Total:  $100 - 14 = 86$

**Reliability score: mean(88, 86)=87**

## Water Toxicity Data Summary

*Americamysis bahia*  
Fipronil desulfinyl  
MB46030

Putt AE. (2000c) [<sup>14</sup>C]MB 46513-Acute toxicity to mysids (*Mysidopsis bahia*) under static acute conditions. Springborn Laboratories, Inc., Wareham, Massachusetts. Laboratory study number 10566.6549. Submitted to Rhone-Poulenc Ag Company, Research Triangle, North Carolina. USEPA MRID 45120001.

Relevance  
Score: 85  
Rating: L

Reliability  
Score: 87  
Rating: R

Relevance points taken off for: Freshwater (15). 100-15=85

|  | <b>Putt 2000a</b>   | <b><i>A. bahia</i></b> |
|--|---|------------------------|
| <b>Parameter</b>                             | <b>Value</b>  | <b>Comment</b>         |
| Test method cited                            | FIFRA 72-3, Ecological Effects Test Guidelines OPPTS 850.1035 |                        |
| Phylum/subphylum                             | Arthropoda/Crustacea  |                        |
| Class  | Malacostraca  |                        |
| Order  | Mysida  |                        |
| Family                                       | Mysidae   |                        |
| Genus  | <i>Americamysis</i>   |                        |
| Species                                      | <i>bahia</i>  |                        |
| Family native to North America?              | Yes   |                        |
| Age/size at start of test/growth phase       | Not reported  |                        |
| Source of organisms                          | Laboratory culture  |                        |
| Have organisms been exposed to contaminants? | No  |                        |
| Animals acclimated and disease-free?         | 14 d  |                        |
| Animals randomized?                          | Yes   |                        |
| Test vessels randomized?                     | Yes   |                        |
| Test duration                                | 96 h  |                        |
| Data for multiple times?                     | 24, 48, 72, 96 h  |                        |
| Effect 1                                     | Survival  |                        |
| Control response 1                           | 100 %   |                        |
| Temperature                                  | 24.5 ± 0.5 °C   |                        |
| Test type                                    | Static  |                        |
| Photoperiod/light intensity                  | 16l:8d/80-100 footcandles                                     |                        |

|   | <b>Putt 2000a</b>                                     | <b>A. bahia</b>  |
|---|---|--|
| <b>Parameter</b>  | <b>Value</b>  | <b>Comment</b>   |
| Dilution water  | Filtered natural seawater                             | 32 ‰ salinity  |
| pH  | 7.9-8.1   |  |
| Dissolved Oxygen  | 2.7-6.5 mg/L  | 37-95 %  |
| Feeding   | Brine shrimp nauplii<br>( <i>Artemia salina</i> ) 1/d |  |
| Purity of test substance  | 97.8 %  |  |
| Concentrations measured?  | Yes   |  |
| Measured is what % of nominal?  | 100-110 %   |  |
| Toxicity values calculated based on nominal or measured concentrations? | Measured  |  |
| Chemical method documented?   | HPLC  |  |
| Concentration of carrier (if any) in test solutions                     | Acetone, 0.100 mL/L                                   |  |
| Concentration 1 Nom; Meas (µg/L)  | 0.0310; 0.0340  | 2 reps, 10/rep   |
| Concentration 2 Nom; Meas (µg/L)  | 0.0630; 0.0660  |  |
| Concentration 3 Nom; Meas (µg/L)  | 0.1300; 0.1300  |  |
| Concentration 4 Nom; Meas (µg/L)  | 0.2500; 0.2600  |  |
| Concentration 5 Nom; Meas (µg/L)  | 0.5000; 0.5200  |  |
| Control   | Negative: 0; 0<br>Solvent: 0; 0                       |  |
| LC <sub>50</sub> (95% CI) (µg/L)  | 0.1500 (0.0660-0.2500)                                | Method: Nonlinear interpolation                              |
| NOEC  | 0.0660  | Method: Not reported<br>p: Not reported<br>MSD: Not reported |
| LOEC  | 0.1300  | Not reported; See Table 4                                    |
| MATC (GeoMean NOEC, LOEC)   | 0.0926  |  |
| % control at NOEC   | 65 %  | 65 (tmt) / 100<br>(mean controls) =<br>65 %                  |
| % control at LOEC   | 100 %   | 100 (tmt) / 100<br>(mean controls) =<br>100 %                |

Notes:

Solubility (S) value for fipronil desulfinyl (MB 46513) = 950 µg/L, 2S = 1900 µg/L.

Reliability points taken off for:

Documentation: Hardness (2), Alkalinity (2), Conductivity (2), Statistical significance (2), Significance level (2), Minimum significant difference (2). Total:  $100 - 12 = 88$

Acceptability: Organism age/size (3), Feeding (3), Hardness (2), Alkalinity (2), Conductivity (1), Adequate replication (2), Minimum significant difference (1). Total:  $100 - 14 = 86$

**Reliability score:  $\text{mean}(88, 86) = 87$**

## Water Toxicity Data Summary

*Anopheles quadrimaculatus*

Fipronil

MB 46030

Ali A, Nayar JK and Gu WD. (1998) Toxicity of a phenyl pyrazole insecticide, fipronil, to mosquito and chironomid midge larvae in the laboratory. *Journal of the American Mosquito Control Association*, 14(2), 216-218.

Relevance

Score: 92.5

Rating: R

Reliability

Score: 60.5

Rating: L

Relevance points taken off for: Control response (7.5).  $100-7.5=92.5$

|  | <b>Ali 1998</b>   | <i>A. quadrimaculatus</i> |
|--|---|---------------------------|
| <b>Parameter</b>                             | <b>Value</b>  | <b>Comment</b>            |
| Test method cited                            | Mulla MS and Khasawinah AM. (1969) Laboratory and field evaluation of larvicides against chironomid midges. <i>Journal of Economic Entomology</i> , 62(1), 37-41. |                           |
| Phylum/subphylum                             | Arthropoda  |                           |
| Class  | Insecta   |                           |
| Order  | Diptera   |                           |
| Family                                       | Culicidae   |                           |
| Genus  | <i>Anopheles</i>  |                           |
| Species                                      | <i>quadrimaculatus</i>  |                           |
| Family native to North America?              | Yes   |                           |
| Age/size at start of test/growth phase       | 4 <sup>th</sup> instar  |                           |
| Source of organisms                          | Florida Medical Entomology Laboratory, University of Florida, Vero Beach, Florida   |                           |
| Have organisms been exposed to contaminants? | No  |                           |
| Animals acclimated and disease-free?         | Yes   |                           |
| Animals randomized?                          | Not reported  |                           |
| Test vessels randomized?                     | Not reported  |                           |
| Test duration                                | 48 h  |                           |
| Data for multiple times?                     | 24, 48 h  |                           |
| Effect 1                                     | Survival  |                           |

|   | <b>Ali 1998</b>                            | <i>A. quadrimaculatus</i>          |
|---|--|------------------------------------|
| <b>Parameter</b>  | <b>Value</b>                               | <b>Comment</b>                     |
| Control response 1  | Not reported                               |                                    |
| Temperature   | 26 ± 2 °C                                  |                                    |
| Test type   | Static                                     |                                    |
| Photoperiod/light intensity   | 14:10d                                     |                                    |
| Dilution water  | Tap water                                  |                                    |
| pH  | Not reported                               |                                    |
| Hardness  | Not reported                               |                                    |
| Alkalinity  | Not reported                               |                                    |
| Conductivity  | Not reported                               |                                    |
| Dissolved Oxygen  | Not reported                               |                                    |
| Feeding   | 1 mL 1% beef liver plus yeast (1:1) daily  |                                    |
| Purity of test substance  | 97.1 %                                     |                                    |
| Concentrations measured?  | Not reported                               |                                    |
| Measured is what % of nominal?  | Not reported                               |                                    |
| Toxicity values calculated based on nominal or measured concentrations? | Not reported                               |                                    |
| Chemical method documented?   | Not reported                               |                                    |
| Concentration of carrier (if any) in test solutions                     | Acetone, concentration not reported        |                                    |
| Concentration 1 Nom; Meas (µg/L)  | 6-7 concentrations tested but not reported | 3 reps, 20/rep                     |
| Control   | Negative                                   |                                    |
| LC <sub>50</sub> (95% CI) (µg/L)  | 48 h: 0.00043 (0.00009-0.00081)            | Method: log-dose-probit regression |

Notes:

Solubility (S) of fipronil = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Analytical method (4), Nominal concentrations (3), Measured concentrations (3), Hardness (2), Alkalinity (2), Dissolved oxygen (4), Conductivity (2), pH (3), Statistical significance (2), Significance level (2), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100-31 =69

Acceptability: Control response (9), Measured concentrations within 20% nominal (4), Concentrations not > 2x solubility (4), Carrier solvent (4), Organisms randomized (1), Feeding (3), Hardness (2), Alkalinity (2), Dissolved oxygen (6), Conductivity (1), pH (2), Random design (2), Dilution factor (2), Hypothesis tests (3), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1). Total: 100-48 =52

**Reliability score: mean(69, 52)=60.5**

## Water Toxicity Data Summary

*Aedes taeniorhynchus*

Fipronil

MB 46030

Ali A, Nayar JK and Gu WD. (1998) Toxicity of a phenyl pyrazole insecticide, fipronil, to mosquito and chironomid midge larvae in the laboratory. *Journal of the American Mosquito Control Association*, 14(2), 216-218.

Relevance

Score: 92.5

Rating: R

Reliability

Score: 60.5

Rating: L

Relevance points taken off for: Control response (7.5).  $100-7.5=92.5$

|  | <b>Ali 1998</b>   | <i>A. taeniorhynchus</i> |
|--|---|--------------------------|
| <b>Parameter</b>                             | <b>Value</b>  | <b>Comment</b>           |
| Test method cited                            | Mulla MS and Khasawinah AM. (1969) Laboratory and field evaluation of larvicides against chironomid midges. <i>Journal of Economic Entomology</i> , 62(1), 37-41. |                          |
| Phylum/subphylum                             | Arthropoda  |                          |
| Class  | Insecta   |                          |
| Order  | Diptera   |                          |
| Family                                       | Culicidae   |                          |
| Genus  | <i>Aedes</i>  |                          |
| Species                                      | <i>taeniorhynchus</i>   |                          |
| Family native to North America?              | Yes   |                          |
| Age/size at start of test/growth phase       | 4 <sup>th</sup> instar  |                          |
| Source of organisms                          | Florida Medical Entomology Laboratory, University of Florida, Vero Beach, Florida   |                          |
| Have organisms been exposed to contaminants? | No  |                          |
| Animals acclimated and disease-free?         | Yes   |                          |
| Animals randomized?                          | Not reported  |                          |
| Test vessels randomized?                     | Not reported  |                          |
| Test duration                                | 48 h  |                          |
| Data for multiple times?                     | 24, 48 h  |                          |
| Effect 1                                     | Survival  |                          |

|   | <b>Ali 1998</b>   | <b><i>A. taeniorhynchus</i></b>    |
|---|---|------------------------------------|
| <b>Parameter</b>  | <b>Value</b>  | <b>Comment</b>                     |
| Control response 1  | Not reported  |                                    |
| Temperature   | 26 ± 2 °C   |                                    |
| Test type   | Static  |                                    |
| Photoperiod/light intensity   | 14:10d  |                                    |
| Dilution water  | Tap water   |                                    |
| pH  | Not reported  |                                    |
| Hardness  | Not reported  |                                    |
| Alkalinity  | Not reported  |                                    |
| Conductivity  | Not reported  |                                    |
| Dissolved Oxygen  | Not reported  |                                    |
| Feeding   | 1 mL 1% beef liver plus yeast (1:1) daily                         |                                    |
| Purity of test substance  | 97.1 %  |                                    |
| Concentrations measured?  | Not reported  |                                    |
| Measured is what % of nominal?  | Not reported  |                                    |
| Toxicity values calculated based on nominal or measured concentrations? | Not reported  |                                    |
| Chemical method documented?   | Not reported  |                                    |
| Concentration of carrier (if any) in test solutions                     | Acetone, concentration not reported                               |                                    |
| Concentration 1 Nom; Meas (µg/L)  | 6-7 concentrations tested but not reported                        | 3 reps, 20/rep                     |
| Control   | Negative  |                                    |
| LC <sub>50</sub> (95% CI) (µg/L)  | 24 h: 0.0014 (0.00119-0.00163)<br>48 h: 0.00043 (0.00034-0.00050) | Method: log-dose-probit regression |

Notes:

Solubility (S) of fipronil = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Analytical method (4), Nominal concentrations (3), Measured concentrations (3), Hardness (2), Alkalinity (2), Dissolved oxygen (4), Conductivity (2), pH (3), Statistical significance (2), Significance level (2), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100-31 =69

Acceptability: Control response (9), Measured concentrations within 20% nominal (4), Concentrations not > 2x solubility (4), Carrier solvent (4), Organisms randomized (1), Feeding (3), Hardness (2), Alkalinity (2), Dissolved oxygen (6), Conductivity (1), pH (2), Random design (2), Dilution factor (2), Hypothesis tests (3), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1). Total: 100-48 =52

**Reliability score: mean(69, 52)=60.5**

## Water Toxicity Data Summary

*Cheumatopsyche brevilineata*

Fipronil sulfide

MB 45950

Iwafune T, Yokoyama A, Nagai T and Horio T. (2011) Evaluation of the risk of mixtures of paddy insecticides and their transformation products to aquatic organisms in the Sakura River, Japan. *Environmental Toxicology and Chemistry*, 30(8), 1834-1842.

Relevance

Score: 82.5

Rating: L

Reliability

Score: 69

Rating: L

Relevance points taken off for: Standard method (10), Control described (7.5). 100-17.5= 82.5

|  | <b>Iwafune 2011</b>                  | <b><i>C. brevilineata</i></b> |
|--|--------------------------------------|-------------------------------|
| <b>Parameter</b>                             | <b>Value</b>                         | <b>Comment</b>                |
| Test method cited                            | Not reported                         |                               |
| Phylum/subphylum                             | Arthropoda                           |                               |
| Class  | Insecta                              |                               |
| Order  | Trichoptera                          |                               |
| Family                                       | Hydropsychoidea                      |                               |
| Genus  | <i>Cheumatopsyche</i>                |                               |
| Species                                      | <i>brevilineata</i>                  |                               |
| Family native to North America?              | Yes                                  |                               |
| Age/size at start of test/growth phase       | 1 <sup>st</sup> instar               |                               |
| Source of organisms                          | Not reported                         |                               |
| Have organisms been exposed to contaminants? | Not reported                         |                               |
| Animals acclimated and disease-free?         | Not reported                         |                               |
| Animals randomized?                          | Not reported                         |                               |
| Test vessels randomized?                     | Not reported                         |                               |
| Test duration                                | 48 h                                 |                               |
| Data for multiple times?                     | No                                   |                               |
| Effect 1                                     | Immobilization                       |                               |
| Control response 1                           | ≤5 %                                 |                               |
| Temperature                                  | 20.9 ± 0.6 °C                        |                               |
| Test type                                    | Static                               |                               |
| Photoperiod/light intensity                  | Not reported                         |                               |
| Dilution water                               | Dechlorinated and filtered tap water |                               |
| pH   | 8                                    |                               |
| Hardness                                     | 70 mg/L CaCO <sub>3</sub>            |                               |

|   | <b>Iwafune 2011</b>  | <i>C. brevilineata</i>    |
|---|--|---------------------------|
| <b>Parameter</b>  | <b>Value</b>   | <b>Comment</b>            |
| Alkalinity  | Not reported   |                           |
| Conductivity  | Not reported   |                           |
| Dissolved Oxygen  | Not reported   |                           |
| Feeding   | Not reported   |                           |
| Purity of test substance  | >98 %  |                           |
| Concentrations measured?  | Yes  |                           |
| Measured is what % of nominal?  | 84.1-105.3 %   |                           |
| Toxicity values calculated based on nominal or measured concentrations? | Measured   |                           |
| Chemical method documented?   | LC-MS/MS   |                           |
| Concentration of carrier (if any) in test solutions                     | Acetone or acetonitrile, ≤1 mL/L   |                           |
| Concentration 1 Nom; Meas (µg/L)  | Concentrations not reported but nominal range 0.0201-0.150 across 7 treatments | Reps not reported, 20/rep |
| Control   | Not reported   |                           |
| EC <sub>50</sub> (95% CI) (µg/L)  | 0.052 (0.042-0.059)  | Method: probit            |

Notes:

Solubility value for fipronil sulfide (MB 45950) not available. Solubility (S) of fipronil parent compound (MB 46030) = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Organism source (5), Nominal concentrations (3), Measured concentrations (3), Alkalinity (2), Dissolved oxygen (4), Conductivity (2), Photoperiod (3), Statistical significance (2), Significance level (2), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100-30 =70

Acceptability: Standard method (5), No prior contamination (4), Organisms randomized (1), Feeding (3), Acclimation (1), Alkalinity (2), Dissolved oxygen (6), Conductivity (1), Photoperiod (2), Random design (2), Statistical method (2), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1). Total: 100-32 =68

**Reliability score: mean(70, 68)=69**

## Water Toxicity Data Summary

*Cheumatopsyche brevilineata*

Fipronil

MB 46030

Iwafune T, Yokoyama A, Nagai T and Horio T. (2011) Evaluation of the risk of mixtures of paddy insecticides and their transformation products to aquatic organisms in the Sakura River, Japan. *Environmental Toxicology and Chemistry*, 30(8), 1834-1842.

Relevance

Score: 82.5

Rating: L

Reliability

Score: 69

Rating: L

Relevance points taken off for:

|  | <b>Iwafune 2011</b>                  | <i>C. brevilineata</i> |
|--|--------------------------------------|------------------------|
| <b>Parameter</b>                             | <b>Value</b>                         | <b>Comment</b>         |
| Test method cited                            | Not reported                         |                        |
| Phylum/subphylum                             | Arthropoda                           |                        |
| Class  | Insecta                              |                        |
| Order  | Trichoptera                          |                        |
| Family                                       | Hydropsychoidea                      |                        |
| Genus  | <i>Cheumatopsyche</i>                |                        |
| Species                                      | <i>brevilineata</i>                  |                        |
| Family native to North America?              | Yes                                  |                        |
| Age/size at start of test/growth phase       | 1 <sup>st</sup> instar               |                        |
| Source of organisms                          | Not reported                         |                        |
| Have organisms been exposed to contaminants? | Not reported                         |                        |
| Animals acclimated and disease-free?         | Not reported                         |                        |
| Animals randomized?                          | Not reported                         |                        |
| Test vessels randomized?                     | Not reported                         |                        |
| Test duration                                | 48 h                                 |                        |
| Data for multiple times?                     | No                                   |                        |
| Effect 1                                     | Immobilization                       |                        |
| Control response 1                           | ≤5 %                                 |                        |
| Temperature                                  | 20.9 ± 0.6 °C                        |                        |
| Test type                                    | Static                               |                        |
| Photoperiod/light intensity                  | Not reported                         |                        |
| Dilution water                               | Dechlorinated and filtered tap water |                        |
| pH   | 8                                    |                        |
| Hardness                                     | 70 mg/L CaCO <sub>3</sub>            |                        |

|   | <b>Iwafune 2011</b>   | <i>C. brevilineata</i>    |
|---|---|---------------------------|
| <b>Parameter</b>  | <b>Value</b>  | <b>Comment</b>            |
| Alkalinity  | Not reported  |                           |
| Conductivity  | Not reported  |                           |
| Dissolved Oxygen  | Not reported  |                           |
| Feeding   | Not reported  |                           |
| Purity of test substance  | >98 %   |                           |
| Concentrations measured?  | Yes   |                           |
| Measured is what % of nominal?  | 84.1-105.3 %  |                           |
| Toxicity values calculated based on nominal or measured concentrations? | Measured  |                           |
| Chemical method documented?   | LC-MS/MS  |                           |
| Concentration of carrier (if any) in test solutions                     | Acetone or acetonitrile, ≤1 mL/L  |                           |
| Concentration 1 Nom; Meas (µg/L)  | Concentrations not reported but nominal range: 0.0954–0.286 across 7 treatments | Reps not reported, 20/rep |
| Control   | Not reported  |                           |
| EC <sub>50</sub> (95% CI) (µg/L)  | 0.133 (0.112-0.148)   | Method: probit            |

Notes:

Solubility (S) of fipronil = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Organism source (5), Nominal concentrations (3), Measured concentrations (3), Alkalinity (2), Dissolved oxygen (4), Conductivity (2), Photoperiod (3), Statistical significance (2), Significance level (2), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100-30 =70

Acceptability: Standard method (5), No prior contamination (4), Organisms randomized (1), Feeding (3), Acclimation (1), Alkalinity (2), Dissolved oxygen (6), Conductivity (1), Photoperiod (2), Random design (2), Statistical method (2), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1). Total: 100-32 =68

**Reliability score: mean(70, 68)=69**

## Water Toxicity Data Summary

*Cheumatopsyche brevilineata*

Fipronil sulfone

MB 46136

Iwafune T, Yokoyama A, Nagai T and Horio T. (2011) Evaluation of the risk of mixtures of paddy insecticides and their transformation products to aquatic organisms in the Sakura River, Japan. *Environmental Toxicology and Chemistry*, 30(8), 1834-1842.

Relevance

Score: 82.5

Rating: L

Reliability

Score: 69

Rating: L

Relevance points taken off for: Standard method (10), Control described (7.5). 100-17.5= 82.5

|  | <b>Iwafune 2011</b>                  | <b><i>C. brevilineata</i></b> |
|--|--------------------------------------|-------------------------------|
| <b>Parameter</b>                             | <b>Value</b>                         | <b>Comment</b>                |
| Test method cited                            | Not reported                         |                               |
| Phylum/subphylum                             | Arthropoda                           |                               |
| Class  | Insecta                              |                               |
| Order  | Trichoptera                          |                               |
| Family                                       | Hydropsychoidea                      |                               |
| Genus  | <i>Cheumatopsyche</i>                |                               |
| Species                                      | <i>brevilineata</i>                  |                               |
| Family native to North America?              | Yes                                  |                               |
| Age/size at start of test/growth phase       | 1 <sup>st</sup> instar               |                               |
| Source of organisms                          | Not reported                         |                               |
| Have organisms been exposed to contaminants? | Not reported                         |                               |
| Animals acclimated and disease-free?         | Not reported                         |                               |
| Animals randomized?                          | Not reported                         |                               |
| Test vessels randomized?                     | Not reported                         |                               |
| Test duration                                | 48 h                                 |                               |
| Data for multiple times?                     | No                                   |                               |
| Effect 1                                     | Immobilization                       |                               |
| Control response 1                           | ≤5 %                                 |                               |
| Temperature                                  | 20.9 ± 0.6 °C                        |                               |
| Test type                                    | Static                               |                               |
| Photoperiod/light intensity                  | Not reported                         |                               |
| Dilution water                               | Dechlorinated and filtered tap water |                               |
| pH   | 8                                    |                               |

|   | <b>Iwafune 2011</b>  | <i>C. brevilineata</i>    |
|---|--|---------------------------|
| <b>Parameter</b>  | <b>Value</b>   | <b>Comment</b>            |
| Hardness  | 70 mg/L CaCO <sub>3</sub>  |                           |
| Alkalinity  | Not reported   |                           |
| Conductivity  | Not reported   |                           |
| Dissolved Oxygen  | Not reported   |                           |
| Feeding   | Not reported   |                           |
| Purity of test substance  | >98 %  |                           |
| Concentrations measured?  | Yes  |                           |
| Measured is what % of nominal?  | 84.1-105.3 %   |                           |
| Toxicity values calculated based on nominal or measured concentrations? | Measured   |                           |
| Chemical method documented?   | LC-MS/MS   |                           |
| Concentration of carrier (if any) in test solutions                     | Acetone or acetonitrile, ≤1 mL/L   |                           |
| Concentration 1 Nom; Meas (µg/L)  | Concentrations not reported but nominal range 0.0200-0.178 across 8 treatments | Reps not reported, 20/rep |
| Control   | Not reported   |                           |
| EC <sub>50</sub> (95% CI) (µg/L)  | 0.066 (0.054-0.078)  | Method: probit            |

Notes:

Solubility (S) value for fipronil sulfone (MB 46136) = 160 µg/L, 2S = 320 µg/L.

Reliability points taken off for:

Documentation: Organism source (5), Nominal concentrations (3), Measured concentrations (3), Alkalinity (2), Dissolved oxygen (4), Conductivity (2), Photoperiod (3), Statistical significance (2), Significance level (2), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100-30 =70

Acceptability: Standard method (5), No prior contamination (4), Organisms randomized (1), Feeding (3), Acclimation (1), Alkalinity (2), Dissolved oxygen (6), Conductivity (1), Photoperiod (2), Random design (2), Statistical method (2), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1). Total: 100-32 =68

**Reliability score: mean(70, 68)=69**

## Water Toxicity Data Summary

*Cheumatopsyche brevilineata*

Fipronil desulfinyl

MB 46513

Iwafune T, Yokoyama A, Nagai T and Horio T. (2011) Evaluation of the risk of mixtures of paddy insecticides and their transformation products to aquatic organisms in the Sakura River, Japan. *Environmental Toxicology and Chemistry*, 30(8), 1834-1842.

Relevance

Score: 82.5

Rating: L

Reliability

Score: 69

Rating: L

Relevance points taken off for: Standard method (10), Control described (7.5). 100-17.5= 82.5

| <b>Parameter</b>                             | <b>Iwafune 2011<br/>Value</b>        | <b><i>C. brevilineata</i><br/>Comment</b> |
|--|--------------------------------------|---|
| Test method cited                            | Not reported                         |   |
| Phylum/subphylum                             | Arthropoda                           |   |
| Class  | Insecta                              |   |
| Order  | Trichoptera                          |   |
| Family                                       | Hydropsychoidea                      |   |
| Genus  | <i>Cheumatopsyche</i>                |   |
| Species                                      | <i>brevilineata</i>                  |   |
| Family native to North America?              | Yes                                  |   |
| Age/size at start of test/growth phase       | 1 <sup>st</sup> instar               |   |
| Source of organisms                          | Not reported                         |   |
| Have organisms been exposed to contaminants? | Not reported                         |   |
| Animals acclimated and disease-free?         | Not reported                         |   |
| Animals randomized?                          | Not reported                         |   |
| Test vessels randomized?                     | Not reported                         |   |
| Test duration                                | 48 h                                 |   |
| Data for multiple times?                     | No                                   |   |
| Effect 1                                     | Immobilization                       |   |
| Control response 1                           | ≤5 %                                 |   |
| Temperature                                  | 20.9 ± 0.6 °C                        |   |
| Test type                                    | Static                               |   |
| Photoperiod/light intensity                  | Not reported                         |   |
| Dilution water                               | Dechlorinated and filtered tap water |   |
| pH   | 8                                    |   |
| Hardness                                     | 70 mg/L CaCO <sub>3</sub>            |   |

|   | <b>Iwafune 2011</b>   | <i>C. brevilineata</i>    |
|---|---|---------------------------|
| <b>Parameter</b>  | <b>Value</b>  | <b>Comment</b>            |
| Alkalinity  | Not reported  |                           |
| Conductivity  | Not reported  |                           |
| Dissolved Oxygen  | Not reported  |                           |
| Feeding   | Not reported  |                           |
| Purity of test substance  | >98 %   |                           |
| Concentrations measured?  | Yes   |                           |
| Measured is what % of nominal?  | 84.1-105.3 %  |                           |
| Toxicity values calculated based on nominal or measured concentrations? | Measured  |                           |
| Chemical method documented?   | LC-MS/MS  |                           |
| Concentration of carrier (if any) in test solutions                     | Acetone or acetonitrile, ≤1 mL/L  |                           |
| Concentration 1 Nom; Meas (µg/L)  | Concentrations not reported but nominal range: 0.0400-0.420 across 7 treatments | Reps not reported, 20/rep |
| Control   | Not reported  |                           |
| EC <sub>50</sub> (95% CI) (µg/L)  | 0.177 (0.054-0.078)   | Method: probit            |

Notes:

Solubility (S) value for fipronil desulfinyl (MB 46513) = 950 µg/L, 2S = 1900 µg/L.

Reliability points taken off for:

Documentation: Organism source (5), Nominal concentrations (3), Measured concentrations (3), Alkalinity (2), Dissolved oxygen (4), Conductivity (2), Photoperiod (3), Statistical significance (2), Significance level (2), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100-30 =70

Acceptability: Standard method (5), No prior contamination (4), Organisms randomized (1), Feeding (3), Acclimation (1), Alkalinity (2), Dissolved oxygen (6), Conductivity (1), Photoperiod (2), Random design (2), Statistical method (2), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1). Total: 100-32 =68

**Reliability score: mean(70, 68)=69**

## Water Toxicity Data Summary

*Cheumatopsyche brevilineata*

Fipronil carboxamide

RPA 200766

Iwafune T, Yokoyama A, Nagai T and Horio T. (2011) Evaluation of the risk of mixtures of paddy insecticides and their transformation products to aquatic organisms in the Sakura River, Japan. *Environmental Toxicology and Chemistry*, 30(8), 1834-1842.

Relevance

Score: 82.5

Rating: L

Reliability

Score: 69

Rating: L

Relevance points taken off for: Standard method (10), Control described (7.5). 100-17.5= 82.5

|  | <b>Iwafune 2011</b>                  | <b><i>C. brevilineata</i></b> |
|--|--------------------------------------|-------------------------------|
| <b>Parameter</b>                             | <b>Value</b>                         | <b>Comment</b>                |
| Test method cited                            | Not reported                         |                               |
| Phylum/subphylum                             | Arthropoda                           |                               |
| Class  | Insecta                              |                               |
| Order  | Trichoptera                          |                               |
| Family                                       | Hydropsychoidea                      |                               |
| Genus  | <i>Cheumatopsyche</i>                |                               |
| Species                                      | <i>brevilineata</i>                  |                               |
| Family native to North America?              | Yes                                  |                               |
| Age/size at start of test/growth phase       | 1 <sup>st</sup> instar               |                               |
| Source of organisms                          | Not reported                         |                               |
| Have organisms been exposed to contaminants? | Not reported                         |                               |
| Animals acclimated and disease-free?         | Not reported                         |                               |
| Animals randomized?                          | Not reported                         |                               |
| Test vessels randomized?                     | Not reported                         |                               |
| Test duration                                | 48 h                                 |                               |
| Data for multiple times?                     | No                                   |                               |
| Effect 1                                     | Immobilization                       |                               |
| Control response 1                           | ≤5 %                                 |                               |
| Temperature                                  | 20.9 ± 0.6 °C                        |                               |
| Test type                                    | Static                               |                               |
| Photoperiod/light intensity                  | Not reported                         |                               |
| Dilution water                               | Dechlorinated and filtered tap water |                               |
| pH   | 8                                    |                               |
| Hardness                                     | 70 mg/L CaCO <sub>3</sub>            |                               |

|   | <b>Iwafune 2011</b>   | <i>C. brevilineata</i>    |
|---|---|---------------------------|
| <b>Parameter</b>  | <b>Value</b>  | <b>Comment</b>            |
| Alkalinity  | Not reported  |                           |
| Conductivity  | Not reported  |                           |
| Dissolved Oxygen  | Not reported  |                           |
| Feeding   | Not reported  |                           |
| Purity of test substance  | >98 %   |                           |
| Concentrations measured?  | Yes   |                           |
| Measured is what % of nominal?  | 84.1-105.3 %  |                           |
| Toxicity values calculated based on nominal or measured concentrations? | Measured  |                           |
| Chemical method documented?   | LC-MS/MS  |                           |
| Concentration of carrier (if any) in test solutions                     | Acetone or acetonitrile, ≤1 mL/L  |                           |
| Concentration 1 Nom; Meas (µg/L)  | Concentrations not reported but nominal range: 04.80-10.0 across 5 treatments | Reps not reported, 20/rep |
| Control   | Not reported  |                           |
| EC <sub>50</sub> (95% CI) (µg/L)  | 4.95 (3.23-26.0)  | Method: probit            |

Notes:

Solubility value for this fipronil metabolite (RPA 200766) not available. Solubility (S) of fipronil parent compound (MB 46030) = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Organism source (5), Nominal concentrations (3), Measured concentrations (3), Alkalinity (2), Dissolved oxygen (4), Conductivity (2), Photoperiod (3), Statistical significance (2), Significance level (2), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100-30 =70

Acceptability: Standard method (5), No prior contamination (4), Organisms randomized (1), Feeding (3), Acclimation (1), Alkalinity (2), Dissolved oxygen (6), Conductivity (1), Photoperiod (2), Random design (2), Statistical method (2), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1). Total: 100-32 =68

**Reliability score: mean(70, 68)=69**

## Water Toxicity Data Summary

*Cheumatopsyche brevilineata*

Fipronil

MB 46030

Yokoyama A, Ohtsu K, Iwafune T, Nagai T, Ishihara S, Kobara Y, Horio T and Endo S. (2009) A useful new insecticide bioassay using first-instar larvae of a net-spinning caddisfly, *Cheumatopsyche brevilineata* (Trichoptera: Hydropsychidae). *Journal of Pesticide Science*, 34(1), 13-20.

Relevance

Score: 82.5

Rating: L

Reliability

Score: 66

Rating: L

Relevance points taken off for: Standard method (10), Control response (7.5). 100-7.5=82.5

|  | <b>Yokoyama 2009</b>                            | <b><i>C. brevilineata</i></b> |
|--|---|-------------------------------|
| <b>Parameter</b>                             | <b>Value</b>                                    | <b>Comment</b>                |
| Test method cited                            | Not reported                                    |                               |
| Phylum/subphylum                             | Arthropoda                                      |                               |
| Class  | Insecta   |                               |
| Order  | Trichoptera                                     |                               |
| Family                                       | Hydropsychidae                                  |                               |
| Genus  | <i>Cheumatopsyche</i>                           |                               |
| Species                                      | <i>brevilineata</i>                             |                               |
| Family native to North America?              | Yes   |                               |
| Age/size at start of test/growth phase       | 5 <sup>th</sup> instar                          |                               |
| Source of organisms                          | Collected from Miyakawa River, Yokohama, Japan, |                               |
| Have organisms been exposed to contaminants? | Not reported                                    |                               |
| Animals acclimated and disease-free?         | Yes   |                               |
| Animals randomized?                          | Not reported                                    |                               |
| Test vessels randomized?                     | Not reported                                    |                               |
| Test duration                                | 48 h  |                               |
| Data for multiple times?                     | No  |                               |
| Effect 1                                     | Survival  |                               |
| Control response 1                           | 95 %  |                               |
| Temperature                                  | 20 ± °C   |                               |
| Test type                                    | Static  |                               |
| Photoperiod/light intensity                  | 18l:6d  |                               |
| Dilution water                               | Dechlorinated tap water                         |                               |
| pH   | 7   |                               |

|   | <b>Yokoyama 2009</b>                                    | <i>C. brevilineata</i> |
|---|---|------------------------|
| <b>Parameter</b>  | <b>Value</b>  | <b>Comment</b>         |
| Hardness  | 70 mg/L CaCO <sub>3</sub>                               |                        |
| Alkalinity  | Not reported  |                        |
| Conductivity  | Not reported  |                        |
| Dissolved Oxygen  | Measured but not reported                               | Not aerated            |
| Feeding   | Not fed   |                        |
| Purity of test substance  | Analytical grade  |                        |
| Concentrations measured?  | No  |                        |
| Measured is what % of nominal?  | Not reported  |                        |
| Toxicity values calculated based on nominal or measured concentrations? | Nominal   |                        |
| Chemical method documented?   | Not reported  |                        |
| Concentration of carrier (if any) in test solutions                     | <0.1 %, carrier not reported                            |                        |
| Concentration 1 Nom; Meas (µg/L)  | 5-10 concentrations tested; concentrations not reported | 20 reps, 1/rep         |
| Control   | Solvent   |                        |
| LC <sub>50</sub> (95% CI) (µg/L)  | 0.153 (0.142–0.164)                                     | Method: Probit         |

Notes:

Solubility (S) of fipronil = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Control type (8), Nominal concentrations (3), Measured concentrations (3), Alkalinity (2), Dissolved oxygen (4), Conductivity (2), Statistical significance (2), Significance level (2), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100- 30=70

Acceptability: Appropriate control (6), Measured concentrations within 20% nominal (4), Concentrations not > 2x solubility (4), No prior contamination (4), Organisms randomized (1), Alkalinity (2), Dissolved oxygen (6), Temperature variation (3), Conductivity (1), Random design (2), Dilution factor (2), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1). Total: 100-38 =62

**Reliability score: mean(70, 62)=66**

## Water Toxicity Data Summary

*Chironomus crassicaudatus*

Fipronil

MB 46030

Ali A, Nayar JK and Gu WD. (1998) Toxicity of a phenyl pyrazole insecticide, fipronil, to mosquito and chironomid midge larvae in the laboratory. *Journal of the American Mosquito Control Association*, 14(2), 216-218.

Relevance

Score: 92.5

Rating: R

Reliability

Score: 60.5

Rating: L

Relevance points taken off for: Control response (7.5).  $100-7.5=92.5$

|  | <b>Ali 1998</b>   | <i>C. crassicaudatus</i> |
|--|---|--------------------------|
| <b>Parameter</b>                             | <b>Value</b>  | <b>Comment</b>           |
| Test method cited                            | Mulla MS and Khasawinah AM. (1969) Laboratory and field evaluation of larvicides against chironomid midges. <i>Journal of Economic Entomology</i> , 62(1), 37-41. |                          |
| Phylum/subphylum                             | Arthropoda  |                          |
| Class  | Insecta   |                          |
| Order  | Diptera   |                          |
| Family                                       | Culicidae   |                          |
| Genus  | <i>Chironomus</i>   |                          |
| Species                                      | <i>crassicaudatus</i>   |                          |
| Family native to North America?              | Yes   |                          |
| Age/size at start of test/growth phase       | 4 <sup>th</sup> instar  |                          |
| Source of organisms                          | Collected from Lake Jessup, central Florida   |                          |
| Have organisms been exposed to contaminants? | No  |                          |
| Animals acclimated and disease-free?         | Yes   |                          |
| Animals randomized?                          | Not reported  |                          |
| Test vessels randomized?                     | Not reported  |                          |
| Test duration                                | 48 h  |                          |
| Data for multiple times?                     | 48 h  |                          |
| Effect 1                                     | Survival  |                          |
| Control response 1                           | Not reported  |                          |
| Temperature                                  | 26 ± 2 °C   |                          |

|   | <b>Ali 1998</b>                            | <i>C. crassicaudatus</i>           |
|---|--|------------------------------------|
| <b>Parameter</b>  | <b>Value</b>                               | <b>Comment</b>                     |
| Test type   | Static                                     |                                    |
| Photoperiod/light intensity   | 14:10d                                     |                                    |
| Dilution water  | Tap water                                  |                                    |
| pH  | Not reported                               |                                    |
| Hardness  | Not reported                               |                                    |
| Alkalinity  | Not reported                               |                                    |
| Conductivity  | Not reported                               |                                    |
| Dissolved Oxygen  | Not reported                               |                                    |
| Feeding   | 1 mL 1% beef liver plus yeast (1:1) daily  |                                    |
| Purity of test substance  | 97.1 %                                     |                                    |
| Concentrations measured?  | Not reported                               |                                    |
| Measured is what % of nominal?  | Not reported                               |                                    |
| Toxicity values calculated based on nominal or measured concentrations? | Not reported                               |                                    |
| Chemical method documented?   | Not reported                               |                                    |
| Concentration of carrier (if any) in test solutions                     | Acetone, concentration not reported        |                                    |
| Concentration 1 Nom; Meas (µg/L)  | 6-7 concentrations tested but not reported | 3 reps, 20/rep                     |
| Control   | Negative                                   |                                    |
| LC <sub>50</sub> (95% CI) (µg/L)  | 0.00042 (0.00032-0.00052)                  | Method: log-dose-probit regression |

Notes:

Solubility (S) of fipronil = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Analytical method (4), Nominal concentrations (3), Measured concentrations (3), Hardness (2), Alkalinity (2), Dissolved oxygen (4), Conductivity (2), pH (3), Statistical significance (2), Significance level (2), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100-31 =69

Acceptability: Control response (9), Measured concentrations within 20% nominal (4), Concentrations not > 2x solubility (4), Carrier solvent (4), Organisms randomized (1), Feeding (3), Hardness (2), Alkalinity (2), Dissolved oxygen (6), Conductivity (1), pH (2), Random design (2), Dilution factor (2), Hypothesis tests (3), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1). Total: 100-48 =52

**Reliability score: mean(69, 52)=60.5**

## Water Toxicity Data Summary

*Ceriodaphnia dubia*

Fipronil

MB 46030

Konwick BJ, Fisk AT, Garrison AW, Avants JK and Black MC. (2005) Acute enantioselective toxicity of fipronil and its desulfinyl photoproduct to *Ceriodaphnia dubia*. *Environmental Toxicology and Chemistry*, 24(9), 2350-2355.

Relevance

Score: 100

Rating: R

Reliability

Score: 70.5

Rating: L

Relevance points taken off for: none.

|  | <b>Konwick et al. 2005</b>  | <i>C. dubia</i> |
|--|---|-----------------|
| <b>Parameter</b>                             | <b>Value</b>  | <b>Comment</b>  |
| Test method cited                            | EPA Methods for measuring the acute toxicity of effluents and receiving water to freshwater and marine organisms EPA/600/4-90/027F. |                 |
| Phylum/subphylum                             | Arthropoda  |                 |
| Class  | Branchiopoda  |                 |
| Order  | Cladocera   |                 |
| Family                                       | Daphniidae  |                 |
| Genus  | <i>Ceriodaphnia</i>   |                 |
| Species                                      | <i>dubia</i>  |                 |
| Family native to North America?              | Yes   |                 |
| Age/size at start of test/growth phase       | <24 h   |                 |
| Source of organisms                          | USEPA laboratory cultures, Region IV Ecological Services Laboratory, Athens, Georgia  |                 |
| Have organisms been exposed to contaminants? | No  |                 |
| Animals acclimated and disease-free?         | Yes   |                 |
| Animals randomized?                          | Not reported  |                 |
| Test vessels randomized?                     | Not reported  |                 |
| Test duration                                | 48 h  |                 |
| Data for multiple times?                     | No  |                 |
| Effect 1                                     | Immobilization  |                 |

|   | <b>Konwick et al. 2005</b>  | <b><i>C. dubia</i></b>             |
|---|---|------------------------------------|
| <b>Parameter</b>  | <b>Value</b>  | <b>Comment</b>                     |
| Control response 1  | <10 %   |                                    |
| Temperature   | 25 ± 1 °C   |                                    |
| Test type   | Static  |                                    |
| Photoperiod/light intensity   | Two series: 16l:8d, no photoperiod  |                                    |
| Dilution water  | Moderately hard water   | 20% Perrier in Milli-Q water (v:v) |
| pH  | 8.3-8.46  |                                    |
| Dissolved Oxygen  | 7.80-8.38 mg/L  |                                    |
| Feeding   | Not fed   |                                    |
| Purity of test substance  | Racemate: 98 %<br>(+): 97.3 %<br>(-): 98.1 %                                      |                                    |
| Concentrations measured?  | Yes   |                                    |
| Measured is what % of nominal?  | Not reported  |                                    |
| Toxicity values calculated based on nominal or measured concentrations? | See notes   |                                    |
| Chemical method documented?   | GCMS  |                                    |
| Concentration of carrier (if any) in test solutions                     | Acetone, 0.1 mL/L   |                                    |
| Concentration 1 Meas (µg/L)   | Racemate: 4.7<br>(+): 4.1<br>(-): 4.5   | 3 reps, 15/rep<br>*See notes       |
| Concentration 2 Nom (µg/L)  | Racemate: 9.3<br>(+): 8.1<br>(-): 9.0   |                                    |
| Concentration 3 Nom (µg/L)  | Racemate: 18.6<br>(+): 16.2<br>(-): 17.8  |                                    |
| Concentration 4 Nom (µg/L)  | Racemate: 37.2<br>(+): 32.4<br>(-): 35.7  |                                    |
| Concentration 5 Meas (µg/L)   | Racemate: 74.4<br>(+): 64.8<br>(-): 71.9  |                                    |
| Control   | Negative: 0; 0<br>Solvent: 0; 0   |                                    |
| LC <sub>50</sub> (SE) (µg/L)  | Light:<br>Racemate: 17.9 ± 2.7<br>(+): 11.3 ± 2.0<br>(-): 35.4 ± 2.6<br><br>Dark: | Method: Trimmed Spearman-Kärber    |

|                  | <b>Konwick et al. 2005</b>  | <i>C. dubia</i> |
|------------------|---|-----------------|
| <b>Parameter</b> | <b>Value</b>  | <b>Comment</b>  |
|                  | Racemate: $17.5 \pm 0.7$<br>(+): $9.4 \pm 0.7$<br>(-): $28.4 \pm 2.4$ |                 |

Notes: Only highest/lowest concentrations measured. Other nominal concentrations calculated by adjustment using standard deviations of those measured.

Solubility (S) of fipronil = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Measured concentrations (3), Hardness (2), Alkalinity (2), Dissolved oxygen (4), Conductivity (2), pH (3), Photoperiod (3), Statistics method (5), Statistical significance (2), Significance level (2), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100-32 =68

Acceptability: Measured concentrations within 20% nominal (4), Organisms randomized (1), Hardness (2), Alkalinity (2), Dissolved oxygen (6), Conductivity (1), Random design (2), Adequate replication (2), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1). Total: 100-23 =77

**Reliability score: mean(68, 73)=70.5**

## Water Toxicity Data Summary

*Ceriodaphnia dubia*  
Fipronil desulfinyl  
MB 46513

Konwick BJ, Fisk AT, Garrison AW, Avants JK and Black MC. (2005) Acute enantioselective toxicity of fipronil and its desulfinyl photoproduct to *Ceriodaphnia dubia*. *Environmental Toxicology and Chemistry*, 24(9), 2350-2355.

Relevance  
Score: 100  
Rating: R

Reliability  
Score: 70.5  
Rating: L

Relevance points taken off for: none.

|  | <b>Konwick et al. 2005</b>  | <i>C. dubia</i> |
|--|---|-----------------|
| <b>Parameter</b>                             | <b>Value</b>  | <b>Comment</b>  |
| Test method cited                            | EPA Methods for measuring the acute toxicity of effluents and receiving water to freshwater and marine organisms EPA/600/4-90/027F. |                 |
| Phylum/subphylum                             | Arthropoda  |                 |
| Class  | Branchiopoda  |                 |
| Order  | Cladocera   |                 |
| Family                                       | Daphniidae  |                 |
| Genus  | <i>Ceriodaphnia</i>   |                 |
| Species                                      | <i>dubia</i>  |                 |
| Family native to North America?              | Yes   |                 |
| Age/size at start of test/growth phase       | <24 h   |                 |
| Source of organisms                          | USEPA laboratory cultures, Region IV Ecological Services Laboratory, Athens, Georgia  |                 |
| Have organisms been exposed to contaminants? | No  |                 |
| Animals acclimated and disease-free?         | Yes   |                 |
| Animals randomized?                          | Not reported  |                 |
| Test vessels randomized?                     | Not reported  |                 |
| Test duration                                | 48 h  |                 |
| Data for multiple times?                     | No  |                 |
| Effect 1                                     | Immobilization  |                 |

|   | <b>Konwick et al. 2005</b>      | <b><i>C. dubia</i></b>             |
|---|---------------------------------|------------------------------------|
| <b>Parameter</b>  | <b>Value</b>                    | <b>Comment</b>                     |
| Control response 1  | <10 %                           |                                    |
| Temperature   | 25 ± 1 °C                       |                                    |
| Test type   | Static                          |                                    |
| Photoperiod/light intensity   | 16l:8d                          |                                    |
| Dilution water  | Moderately hard water           | 20% Perrier in Milli-Q water (v:v) |
| pH  | 8.3-8.46                        |                                    |
| Dissolved Oxygen  | 7.80-8.38 mg/L                  |                                    |
| Feeding   | Not fed                         |                                    |
| Purity of test substance  | 97.8 %                          |                                    |
| Concentrations measured?  | Yes                             |                                    |
| Measured is what % of nominal?  | Not reported                    |                                    |
| Toxicity values calculated based on nominal or measured concentrations? | See notes                       |                                    |
| Chemical method documented?   | GCMS                            |                                    |
| Concentration of carrier (if any) in test solutions                     | Acetone, 0.1 mL/L               |                                    |
| Concentration 1 Meas (µg/L)   | 213                             | 3 reps, 15/rep<br>*See notes       |
| Concentration 2 Nom (µg/L)  | 251                             |                                    |
| Concentration 3 Nom (µg/L)  | 290                             |                                    |
| Concentration 4 Nom (µg/L)  | 329                             |                                    |
| Concentration 5 Meas (µg/L)   | 367                             |                                    |
| Control   | Negative: 0; 0<br>Solvent: 0; 0 |                                    |
| LC <sub>50</sub> ± SE (µg/L)  | 355 ± 9.3                       | Method: Trimmed Spearman-Kärber    |

Notes: Only highest/lowest concentrations measured. Other nominal concentrations calculated by adjustment using standard deviations of those measured. Solubility (S) value for fipronil desulfinyl (MB 46513) = 950 µg/L, 2S = 1900 µg/L.

Reliability points taken off for:

Documentation: Measured concentrations (3), Hardness (2), Alkalinity (2), Dissolved oxygen (4), Conductivity (2), pH (3), Photoperiod (3), Statistics method (5), Statistical significance (2), Significance level (2), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100-32 =68

Acceptability: Measured concentrations within 20% nominal (4), Organisms randomized (1), Hardness (2), Alkalinity (2), Dissolved oxygen (6), Conductivity (1), Random design (2), Adequate replication (2), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1). Total: 100-23 =77

**Reliability score: mean(68, 73)=70.5**

## Water Toxicity Data Summary

*Corbicula fluminea*

Fipronil

MB 46030

Putt AE. (2003b) Fipronil-Acute toxicity to clams (*Corbicula fluminea*) under static-renewal conditions. Springborn Smithers Laboratories, Wareham, Massachusetts. Laboratory study number 986.6161. Submitted to BASF Corporation, Research Triangle Park, North Carolina. USEPA MRID 46329904.

Relevance

Score: 85

Rating: L

Reliability

Score: 93.5

Rating: R

Relevance points taken off for: Toxicity value (15). 100-15=85

|  | <b>Putt 2003</b>                                    | <i>C. fluminea</i> |
|--|---|--------------------|
| <b>Parameter</b>                             | <b>Value</b>  | <b>Comment</b>     |
| Test method cited                            | ASTM Guideline E-729                                |                    |
| Phylum/subphylum                             | Mollusca  |                    |
| Class  | Bivalvia  |                    |
| Order  | Veneroida   |                    |
| Family                                       | Cyrenidae   |                    |
| Genus  | <i>Corbicula</i>                                    |                    |
| Species                                      | <i>fluminea</i>                                     |                    |
| Family native to North America?              | Yes   |                    |
| Age/size at start of test/growth phase       | 12.4 mm shell width<br>0.09319 g soft tissue weight |                    |
| Source of organisms                          | Osage Cat Fisheries, Osage Beach, Missouri          |                    |
| Have organisms been exposed to contaminants? | No  |                    |
| Animals acclimated and disease-free?         | 2 w   |                    |
| Animals randomized?                          | Yes   |                    |
| Test vessels randomized?                     | Yes   |                    |
| Test duration                                | 96 h  |                    |
| Data for multiple times?                     | 24, 48, 72, 96 h                                    |                    |
| Effect 1                                     | Survival  |                    |
| Control response 1                           | 100 %   |                    |
| Temperature                                  | 20 ± 1 °C   |                    |
| Test type                                    | Static renewal                                      |                    |
| Photoperiod/light intensity                  | 16l:8d/690-900 lux                                  |                    |
| Dilution water                               | Well water  |                    |
| pH   | 7.6   |                    |

|   | <b>Putt 2003</b>                | <i>C. fluminea</i>   |
|---|---------------------------------|--|
| <b>Parameter</b>  | <b>Value</b>                    | <b>Comment</b>   |
| Hardness  | 36 mg/L CaCO <sub>3</sub>       |  |
| Alkalinity  | 28 mg/L CaCO <sub>3</sub>       |  |
| Conductivity  | 120 µmhos/cm                    |  |
| Dissolved Oxygen  | 8.5-9.2 mg/L                    | 94-101 %   |
| Feeding   | Not fed                         |  |
| Purity of test substance  | 99.7 %                          |  |
| Concentrations measured?  | Yes                             |  |
| Measured is what % of nominal?  | 92-105 %                        |  |
| Toxicity values calculated based on nominal or measured concentrations? | Measured                        |  |
| Chemical method documented?   | GC/ECD                          |  |
| Concentration of carrier (if any) in test solutions                     | Acetone, 0.10 mL/L              |  |
| Concentration 1 Nom; Meas (µg/L)  | 260; 250                        | 2 reps, 10/rep   |
| Concentration 2 Nom; Meas (µg/L)  | 430; 450                        |  |
| Concentration 3 Nom; Meas (µg/L)  | 720; 690                        |  |
| Concentration 4 Nom; Meas (µg/L)  | 1200; 1100                      |  |
| Concentration 5 Nom; Meas (µg/L)  | 2000; 2000                      |  |
| Control   | Negative: 0; 0<br>Solvent: 0; 0 |  |
| LC <sub>50</sub> (95% CI) (µg/L)  | >2000                           | Method:<br>Empirically estimated   |
| NOEC  | 2000                            | Method:<br>Empirically estimated<br>p: Not reported<br>MSD: Not reported |
| % control at NOEC   | 100 %                           |  |

Notes:

Solubility (S) of fipronil = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Statistical significance (2), Significance level (2), Minimum significant difference (2). Total: 100-6 =94

Acceptability: Adequate replication (2), Minimum significant difference (1), % control at NOEC (1), Point estimates (3). Total: 100-7 =93

**Reliability score: mean(94, 93)=93.5**

## Water Toxicity Data Summary

*Cricotopus lebetis*

Fipronil

MB 46030

Stratman KN, Wilson PC, Overholt WA, Cuda JP and Netherland MD. (2013) Toxicity of fipronil to the midge, *Cricotopus lebetis* Sublette. *Journal of Toxicology and Environmental Health, Part A*, 76(12), 716-722.

Relevance

Score: 75

Rating: L

Reliability

Score: 61.5

Rating: L

Relevance points taken off for: Standard method (10), Controls (15). 100-25=75

|  | <b>Stratman 2013</b>                              | <b><i>C. lebetis</i></b> |
|--|---|--------------------------|
| <b>Parameter</b>                             | <b>Value</b>                                      | <b>Comment</b>           |
| Test method cited                            | Not reported                                      |                          |
| Phylum/subphylum                             | Arthropoda  |                          |
| Class  | Insecta   |                          |
| Order  | Diptera   |                          |
| Family                                       | Chironomidae                                      |                          |
| Genus  | <i>Cricotopus</i>                                 |                          |
| Species                                      | <i>lebetis</i>                                    |                          |
| Family native to North America?              | Yes   |                          |
| Age/size at start of test/growth phase       | 8 d   |                          |
| Source of organisms                          | Collected from Lake Rowell, Bradford Co., Florida |                          |
| Have organisms been exposed to contaminants? | Not reported                                      |                          |
| Animals acclimated and disease-free?         | Yes   |                          |
| Animals randomized?                          | Not reported                                      |                          |
| Test vessels randomized?                     | Not reported                                      |                          |
| Test duration                                | 96 h  |                          |
| Data for multiple times?                     | 24, 48, 72, 96 h                                  |                          |
| Effect 1                                     | Survival  |                          |
| Control response 1                           | Not reported                                      |                          |
| Temperature                                  | 25 °C   |                          |
| Test type                                    | Static  |                          |
| Photoperiod/light intensity                  | 14l:10d   |                          |
| Dilution water                               | Well water  |                          |
| pH   | 7.9   |                          |

|   | <b>Stratman 2013</b>   | <i>C. lebetis</i> |
|---|--|-------------------|
| <b>Parameter</b>  | <b>Value</b>   | <b>Comment</b>    |
| Hardness  | 146 mg/L CaCO <sub>3</sub>   |                   |
| Alkalinity  | 290 mg/L CaCO <sub>3</sub>   |                   |
| Conductivity  | 0.885 umhos/cm   |                   |
| Dissolved Oxygen  | Not reported   |                   |
| Feeding   | Hydrilla tube  |                   |
| Purity of test substance  | 99 %   |                   |
| Concentrations measured?  | Yes  |                   |
| Measured is what % of nominal?  | Not reported   |                   |
| Toxicity values calculated based on nominal or measured concentrations? | Not reported   |                   |
| Chemical method documented?   | GC-ECD   |                   |
| Concentration of carrier (if any) in test solutions                     | Not reported   |                   |
| Concentration 1 Nom; Meas (µg/L)  | 0.5; Not reported  | 5 reps, 1/rep     |
| Concentration 2 Nom; Meas (µg/L)  | 2; Not reported  |                   |
| Concentration 3 Nom; Meas (µg/L)  | 5; Not reported  |                   |
| Concentration 4 Nom; Meas (µg/L)  | 10; Not reported   |                   |
| Concentration 5 Nom; Meas (µg/L)  | 15; Not reported   |                   |
| Concentration 6 Nom; Meas (µg/L)  | 20; Not reported   |                   |
| Control   | Not reported   |                   |
| LC <sub>50</sub> (95% CI) (µg/L)  | 24 h: 7.26 (4.92–10.89)<br>48 h: 2.61 (1.78–3.55)<br>72 h: 1.78 (1.18–2.47)<br>96 h: 1.06 (0.6–1.57) | Method: Probit    |

Notes: Solubility (S) of fipronil = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Control type (8), Organism source (5), Measured concentrations (3), Dissolved oxygen (4), Statistical significance (2), Significance level (2), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100-28 =72

Acceptability: Standard method (5), Appropriate control (6), Control response (9), Measured concentrations within 20% nominal (4), Carrier solvent (4), No prior contamination (4), Organisms randomized (1), Adequate organisms per rep (2), Dissolved oxygen (6), Temperature variation (3), Random design (2), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1). Total: 100-49 =51

**Reliability score: mean(72, 51)=61.5**

## Water Toxicity Data Summary

*Chironomus crassicaudatus*

Fipronil

MB 46030

Ali A, Nayar JK and Gu WD. (1998) Toxicity of a phenyl pyrazole insecticide, fipronil, to mosquito and chironomid midge larvae in the laboratory. *Journal of the American Mosquito Control Association*, 14(2), 216-218.

Relevance

Score: 92.5

Rating: R

Reliability

Score: 60.5

Rating: L

Relevance points taken off for: Control response (7.5).  $100-7.5=92.5$

|  | <b>Ali 1998</b>   | <b><i>C. crassicaudatus</i></b> |
|--|---|---------------------------------|
| <b>Parameter</b>                             | <b>Value</b>  | <b>Comment</b>                  |
| Test method cited                            | Mulla MS and Khasawinah AM. (1969) Laboratory and field evaluation of larvicides against chironomid midges. <i>Journal of Economic Entomology</i> , 62(1), 37-41. |                                 |
| Phylum/subphylum                             | Arthropoda  |                                 |
| Class  | Insecta   |                                 |
| Order  | Diptera   |                                 |
| Family                                       | Culicidae   |                                 |
| Genus  | <i>Chironomus</i>   |                                 |
| Species                                      | <i>crassicaudatus</i>   |                                 |
| Family native to North America?              | Yes   |                                 |
| Age/size at start of test/growth phase       | 4 <sup>th</sup> instar  |                                 |
| Source of organisms                          | Collected from Lake Jessup, central Florida   |                                 |
| Have organisms been exposed to contaminants? | No  |                                 |
| Animals acclimated and disease-free?         | Yes   |                                 |
| Animals randomized?                          | Not reported  |                                 |
| Test vessels randomized?                     | Not reported  |                                 |
| Test duration                                | 48 h  |                                 |
| Data for multiple times?                     | 24, 48 h  |                                 |
| Effect 1                                     | Survival  |                                 |
| Control response 1                           | Not reported  |                                 |
| Temperature                                  | 26 ± 2 °C   |                                 |

|   | <b>Ali 1998</b>   | <i>C. crassicaudatus</i>           |
|---|---|------------------------------------|
| <b>Parameter</b>  | <b>Value</b>  | <b>Comment</b>                     |
| Test type   | Static  |                                    |
| Photoperiod/light intensity   | 14:10d  |                                    |
| Dilution water  | Tap water   |                                    |
| pH  | Not reported  |                                    |
| Hardness  | Not reported  |                                    |
| Alkalinity  | Not reported  |                                    |
| Conductivity  | Not reported  |                                    |
| Dissolved Oxygen  | Not reported  |                                    |
| Feeding   | 1 mL 1% beef liver plus yeast (1:1) daily   |                                    |
| Purity of test substance  | 97.1 %  |                                    |
| Concentrations measured?  | Not reported  |                                    |
| Measured is what % of nominal?  | Not reported  |                                    |
| Toxicity values calculated based on nominal or measured concentrations? | Not reported  |                                    |
| Chemical method documented?   | Not reported  |                                    |
| Concentration of carrier (if any) in test solutions                     | Acetone, concentration not reported   |                                    |
| Concentration 1 Nom; Meas (µg/L)  | 6-7 concentrations tested but not reported  | 3 reps, 20/rep                     |
| Control   | Negative  |                                    |
| LC <sub>50</sub> (95% CI) (µg/L)  | 1 <sup>st</sup> instar:<br>48 h: 0.0046 (0.00004-0.0087)<br><br>4 <sup>th</sup> instar:<br>48 h: 0.0073 (0.0069-0.0077) | Method: log-dose-probit regression |

Notes: Solubility (S) of fipronil = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Analytical method (4), Nominal concentrations (3), Measured concentrations (3), Hardness (2), Alkalinity (2), Dissolved oxygen (4), Conductivity (2), pH (3), Statistical significance (2), Significance level (2), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100-31 =69

Acceptability: Control response (9), Measured concentrations within 20% nominal (4), Concentrations not > 2x solubility (4), Carrier solvent (4), Organisms randomized (1), Feeding (3), Hardness (2), Alkalinity (2), Dissolved oxygen (6), Conductivity (1), pH (2), Random design (2), Dilution factor (2), Hypothesis tests (3), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1). Total: 100-48 =52

**Reliability score: mean(69, 52)=60.5**

## Water Toxicity Data Summary

*Cyprinodon variegatus*

Fipronil

MB 46030

Dionne E. (2000) Fipronil technical-Chronic toxicity to the sheepshead minnow (*Cyprinodon variegatus*) during a full life-cycle exposure. Springborn Laboratories, Inc., Wareham, Massachusetts. Laboratory study number 10566.6580. Submitted to Aventis CropScience, Research Triangle, North Carolina. USEPA MRID 45265101.

Relevance

Score: 85

Rating: L

Reliability

Score: 83.5

Rating: R

Relevance points taken off for: Freshwater (15). 100-15=85

|  | <b>Dionne 2000</b>   | <b><i>C. variegatus</i></b> |
|--|--|-----------------------------|
| <b>Parameter</b>                             | <b>Value</b>   | <b>Comment</b>              |
| Test method cited                            | FIFRA 72-5   |                             |
| Phylum/subphylum                             | Chordata   |                             |
| Class  | Actinopterygii   |                             |
| Order  | Cyprinodontiformes   |                             |
| Family                                       | Cyprinodontidae  |                             |
| Genus  | <i>Cyprinodon</i>  |                             |
| Species                                      | <i>variegatus</i>  |                             |
| Family native to North America?              | Yes  |                             |
| Age/size at start of test/growth phase       | <26 h embryos  |                             |
| Source of organisms                          | Aquatic Biosystems, Fort Collins, Colorado                         |                             |
| Have organisms been exposed to contaminants? | No   |                             |
| Animals acclimated and disease-free?         | Yes  |                             |
| Animals randomized?                          | Not reported   |                             |
| Test vessels randomized?                     | Not reported   |                             |
| Test duration                                | F <sub>0</sub> : 110 d<br>F <sub>1</sub> : 28 d post-hatch         |                             |
| Data for multiple times?                     | F <sub>0</sub> : 28, 59, 110 d<br>F <sub>1</sub> : 28 d post-hatch |                             |
| Effect 1                                     | F <sub>0</sub> embryo hatch  |                             |
| Control response 1                           | 75 %   |                             |
| Effect 2                                     | F <sub>0</sub> 28 d survival                                       |                             |
| Control response 2                           | 97 %   |                             |
| Effect 3                                     | F <sub>0</sub> 28 d length   |                             |

|                                     | <b>Dionne 2000</b>  | <i>C. variegatus</i> |
|-------------------------------------|---|----------------------|
| <b>Parameter</b>                    | <b>Value</b>  | <b>Comment</b>       |
| Control response 3                  | 29 mm   |                      |
| Effect 4                            | F <sub>0</sub> 28 d wet weight  |                      |
| Control response 4                  | 414 mg  |                      |
| Effect 5                            | F <sub>0</sub> 59 d survival  |                      |
| Control response 5                  | 100 %   |                      |
| Effect 6                            | F <sub>0</sub> 59 d length  |                      |
| Control response 6                  | 40.4 mm   |                      |
| Effect 7                            | F <sub>0</sub> 110 d survival   |                      |
| Control response 7                  | 84 %  |                      |
| Effect 8                            | F <sub>0</sub> 110 d length   |                      |
| Control response 8                  | Male: 48.2 mm<br>Female: 43.0 mm  |                      |
| Effect 9                            | F <sub>0</sub> 110 d wet weight   |                      |
| Control response 9                  | Male: 2.06 g<br>Female: 1.35 g  |                      |
| Effect 10                           | Eggs/female/day   |                      |
| Control response 10                 | 24  |                      |
| Effect 11                           | F <sub>1</sub> hatching success   |                      |
| Control response 11                 | 86 %  |                      |
| Effect 12                           | F <sub>1</sub> 28 d survival  |                      |
| Control response 12                 | 98 %  |                      |
| Effect 13                           | F <sub>1</sub> length   |                      |
| Control response 13                 | 25.7 mm   |                      |
| Effect 13                           | F <sub>1</sub> weight   |                      |
| Control response 13                 | 275 mg  |                      |
| Temperature                         | 28 ± 1 °C   |                      |
| Test type                           | Flow through  |                      |
| Photoperiod/light intensity         | 16l:8d/70-120 footcandles   |                      |
| Dilution water                      | Filtered natural seawater,<br>Cape Cod Canal, Bourne,<br>Massachusetts  | 32-33 ‰              |
| pH                                  | 7.6-8.2   |                      |
| Dissolved Oxygen                    | 5.7 mg/L  | 72 %                 |
| Feeding                             | Post hatch larvae: live brine<br>shrimp nauplii ( <i>Artemia<br/>salina</i> ) 3/d<br>Juvenile/adult (>14 d old):<br>Zeigler Prime Flakes and<br>frozen brine shrimp 2/d |                      |
| Purity of test substance            | 98 %  |                      |
| Concentrations measured?            | Yes   |                      |
| Measured is what % of nominal?      | 91-106 %  |                      |
| Toxicity values calculated based on | Measured  |                      |

|   | <b>Dionne 2000</b>                 | <b><i>C. variegatus</i></b>  |
|---|------------------------------------|--|
| <b>Parameter</b>                                    | <b>Value</b>                       | <b>Comment</b>   |
| nominal or measured concentrations?                 |                                    |  |
| Chemical method documented?                         | GC                                 |  |
| Concentration of carrier (if any) in test solutions | Not reported                       |  |
| Concentration 1 Nom; Meas (µg/L)                    | 0.81; 0.85                         | 2 reps, 50 embryos/rep   |
| Concentration 2 Nom; Meas (µg/L)                    | 1.6; 1.7                           |  |
| Concentration 3 Nom; Meas (µg/L)                    | 3.3; 3.0                           |  |
| Concentration 4 Nom; Meas (µg/L)                    | 6.5; 6.0                           |  |
| Concentration 5 Nom; Meas (µg/L)                    | 13; 13                             |  |
| Control   | Negative: 0; 0                     |  |
| NOEC  | 6.0                                | Method: Williams' Test and binomial probability<br>p: 0.05<br>MSD: Not reported<br><br>Based on F <sub>0</sub> female length |
| LOEC  | 13                                 | Based on F <sub>0</sub> and F <sub>1</sub> length; F <sub>0</sub> fecundity; F <sub>1</sub> hatching success                 |
| MATC (GeoMean NOEC, LOEC)                           | 8.8                                |  |
| % control at NOEC                                   | F <sub>0</sub> female length: 93 % | F <sub>0</sub> female length: 40.2 (tmt) / 43 (mean controls) = 93 %   |
| % control at LOEC                                   | F <sub>0</sub> female length: 86 % | F <sub>0</sub> female length: 37.0 (tmt) / 43 (mean controls) = 86 %   |

Notes: Dilution water TOC <1.0 mg/L

Solubility (S) of fipronil = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Hardness (2), Alkalinity (2), Conductivity (2), Minimum significant difference (2), Point estimates (8). Total: 100-16 =84

Acceptability: Organisms randomized (1), Hardness (2), Alkalinity (2), Temperature tolerance (3), Conductivity (1), Random design (2), Adequate replication (2), Minimum significant difference (1), Point estimates (3). Total: 100-17 =83

**Reliability score: mean(84, 83)=83.5**

## Water Toxicity Data Summary

*Cyprinodon variegatus*

Fipronil

MB 46030

Sousa JV. (1998a) Fipronil technical-Early life-stage toxicity test with sheepshead minnow (*Cyprinodon variegatus*). Springborn Laboratories, Inc., Wareham, Massachusetts. Laboratory study number 10566.0796.6402.520. Submitted to Rhone-Poulenc Ag Company, Research Triangle, North Carolina. USEPA MRID 44605501. CA DPR 169427.

Relevance

Score: 85

Rating: L

Reliability

Score: 87

Rating: R

Relevance points taken off for: Freshwater (15). 100-15=85

|  | <b>Sousa 1998</b>                          | <b><i>C. variegatus</i></b> |
|--|--|-----------------------------|
| <b>Parameter</b>                             | <b>Value</b>                               | <b>Comment</b>              |
| Test method cited                            | FIFRA 72-4                                 |                             |
| Phylum/subphylum                             | Chordata                                   |                             |
| Class  | Actinopterygii                             |                             |
| Order  | Cyprinodontiformes                         |                             |
| Family                                       | Cyprinodontidae                            |                             |
| Genus  | <i>Cyprinodon</i>                          |                             |
| Species                                      | <i>variegatus</i>                          |                             |
| Family native to North America?              | Yes  |                             |
| Age/size at start of test/growth phase       | <30 h embryos                              |                             |
| Source of organisms                          | Aquatic Biosystems, Fort Collins, Colorado |                             |
| Have organisms been exposed to contaminants? | No   |                             |
| Animals acclimated and disease-free?         | Yes  |                             |
| Animals randomized?                          | Yes  |                             |
| Test vessels randomized?                     | Yes  |                             |
| Test duration                                | 34 d (28 d post-hatch)                     |                             |
| Data for multiple times?                     | No   |                             |
| Effect 1                                     | Survival                                   |                             |
| Control response 1                           | 78 %                                       |                             |
| Effect 2                                     | Length                                     |                             |
| Control response 2                           | 26.6 mm                                    |                             |
| Effect 3                                     | Weight                                     |                             |
| Control response 3                           | Wet: 0.34 g<br>Dry: 0.095 g                |                             |

|   | <b>Sousa 1998</b>  | <b><i>C. variegatus</i></b>   |
|---|--|---|
| <b>Parameter</b>  | <b>Value</b>   | <b>Comment</b>  |
| Temperature   | 25 ± 1 °C  |   |
| Test type   | Flow through   |   |
| Photoperiod/light intensity   | 16l:8d/60-90 footcandles   |   |
| Dilution water  | Filtered natural seawater,<br>Cape Cod Canal, Bourne,<br>Massachusetts     | 31-32 ‰   |
| pH  | 7.8-8.0  |   |
| Dissolved Oxygen  | 6.5-7.1 mg/L   | 79-86 ‰   |
| Feeding   | Post hatch: live brine<br>shrimp nauplii ( <i>Artemia<br/>salina</i> ) 3/d |   |
| Purity of test substance  | 97.08 ‰  |   |
| Concentrations measured?  | Yes  |   |
| Measured is what ‰ of nominal?  | 87-100 ‰   |   |
| Toxicity values calculated based on<br>nominal or measured<br>concentrations? | Measured   |   |
| Chemical method documented?   | HPLC   |   |
| Concentration of carrier (if any) in<br>test solutions                        | Acetone, 18µL/L  |   |
| Concentration 1 Nom; Meas (µg/L)  | 1.6; 1.6   | 2 reps, 30/rep  |
| Concentration 2 Nom; Meas (µg/L)  | 3.1; 2.7   |   |
| Concentration 3 Nom; Meas (µg/L)  | 6.3; 5.7   |   |
| Concentration 4 Nom; Meas (µg/L)  | 13; 10   |   |
| Concentration 5 Nom; Meas (µg/L)  | 25; 22   |   |
| Control   | Negative: 0; 0<br>Solvent: 0; 0  |   |
| NOEC  | <1.6   | Method: Williams' Test<br>p: Not reported<br>MSD: Not reported<br>Based on larval wet<br>and dry weight |
| LOEC  | 1.6  |   |
| MATC (GeoMean NOEC, LOEC)   | Not calculable   |   |
| % control at NOEC   | Not calculable   |   |
| % control at LOEC   | Larval wet weight: 85 ‰<br><br>Larval dry weight: 86 ‰                     | Larval wet weight:<br>0.29 (tmt) / 0.34<br>b(mean controls) =<br>85 ‰<br><br>Larval dry weight:         |

|                  | <b>Sousa 1998</b> | <i>C. variegatus</i>                             |
|------------------|-------------------|--|
| <b>Parameter</b> | <b>Value</b>      | <b>Comment</b>                                   |
|                  |                   | 0.082 (tmt) / 0.095<br>(mean controls) =<br>86 % |

Notes: Dilution water TOC 2.0 mg/L

Solubility (S) of fipronil = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Hardness (2), Alkalinity (2), Conductivity (2), Statistical significance (2), Significance level (2), Minimum significant difference (2), Point estimates (8). Total: 100-16=84

Acceptability: Hardness (2), Alkalinity (2), Conductivity (1), Adequate replication (2), Minimum significant difference (1), % control at NOEC (1), Point estimates (3). Total: 100-10 =90

**Reliability score: mean(84, 90)=87**

## Water Toxicity Data Summary

*Cyprinodon variegatus*

Fipronil

MB 46030

Sousa JV. (1998b) Fipronil technical-Early life-stage toxicity test with sheepshead minnow (*Cyprinodon variegatus*). Springborn Laboratories, Inc., Wareham, Massachusetts. Laboratory study number 10566.0797.6438.520. Submitted to Rhone-Poulenc Ag Company, Research Triangle, North Carolina. USEPA MRID 44605502. CA DPR 169428.

Relevance

Score: 85

Rating: L

Reliability

Score: 87

Rating: R

Relevance points taken off for: Freshwater (15). 100-15=85

|  | <b>Sousa 1998</b>                          | <b><i>C. variegatus</i></b> |
|--|--|-----------------------------|
| <b>Parameter</b>                             | <b>Value</b>                               | <b>Comment</b>              |
| Test method cited                            | FIFRA 72-4                                 |                             |
| Phylum/subphylum                             | Chordata                                   |                             |
| Class  | Actinopterygii                             |                             |
| Order  | Cyprinodontiformes                         |                             |
| Family                                       | Cyprinodontidae                            |                             |
| Genus  | <i>Cyprinodon</i>                          |                             |
| Species                                      | <i>variegatus</i>                          |                             |
| Family native to North America?              | Yes  |                             |
| Age/size at start of test/growth phase       | <23 h embryos                              |                             |
| Source of organisms                          | Aquatic Biosystems, Fort Collins, Colorado |                             |
| Have organisms been exposed to contaminants? | No   |                             |
| Animals acclimated and disease-free?         | Yes  |                             |
| Animals randomized?                          | Yes  |                             |
| Test vessels randomized?                     | Yes  |                             |
| Test duration                                | 35 d                                       |                             |
| Data for multiple times?                     | No   |                             |
| Effect 1                                     | Survival                                   |                             |
| Control response 1                           | 91 %                                       |                             |
| Effect 2                                     | Length                                     |                             |
| Control response 2                           | 23.5 mm                                    |                             |
| Effect 3                                     | Weight                                     |                             |
| Control response 3                           | Wet: 0.22 g<br>Dry: 0.065 g                |                             |

|   | <b>Sousa 1998</b>  | <i>C. variegatus</i>  |
|---|--|---|
| <b>Parameter</b>  | <b>Value</b>   | <b>Comment</b>  |
| Temperature   | 25 ± 1 °C  |   |
| Test type   | Flow through   |   |
| Photoperiod/light intensity   | 16l:8d/36-100 footcandles  |   |
| Dilution water  | Filtered natural seawater,<br>Cape Cod Canal, Bourne,<br>Massachusetts     | 31-33 ‰   |
| pH  | 7.8-8.0  | 67-85 %   |
| Dissolved Oxygen  | 5.5-7.0 mg/L   |   |
| Feeding   | Post hatch: live brine<br>shrimp nauplii ( <i>Artemia<br/>salina</i> ) 3/d |   |
| Purity of test substance  | 97.08 %  |   |
| Concentrations measured?  | Yes  |   |
| Measured is what % of nominal?  | 97-126 %   |   |
| Toxicity values calculated based on<br>nominal or measured<br>concentrations? | Measured   |   |
| Chemical method documented?   | HPLC   |   |
| Concentration of carrier (if any) in<br>test solutions                        | Dimethylformamide,<br>18µL/L   |   |
| Concentration 1 Nom; Meas (µg/L)  | 0.19; 0.24   | 2 reps, 80/rep  |
| Concentration 2 Nom; Meas (µg/L)  | 0.38; 0.41   |   |
| Concentration 3 Nom; Meas (µg/L)  | 0.75; 0.74   |   |
| Concentration 4 Nom; Meas (µg/L)  | 1.5; 1.5   |   |
| Concentration 5 Nom; Meas (µg/L)  | 3.0; 2.9   |   |
| Control   | Negative: 0; 0<br>Solvent: 0; 0  |   |
| NOEC  | 2.9  | Method: Williams' Test<br>p: Not reported<br>MSD: Not reported  |
| LOEC  | >2.9   |   |
| MATC (GeoMean NOEC, LOEC)   | Not calculable   |   |
| % control at NOEC   | Larval wet weight: 95 %<br><br>Larval dry weight: 94 %                     | Larval wet weight:<br>0.21 (tmt) / 0.22<br>b(mean controls) =<br>95 %<br><br>Larval dry weight:<br>0.061 (tmt) / 0.065<br>(mean controls) =<br>94 % |

Notes:

Solubility (S) of fipronil = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Hardness (2), Alkalinity (2), Conductivity (2), Statistical significance (2), Significance level (2), Minimum significant difference (2), Point estimates (8). Total: 100-16=84

Acceptability: Hardness (2), Alkalinity (2), Conductivity (1), Adequate replication (2), Minimum significant difference (1), % control at NOEC (1), Point estimates (3). Total: 100-10 =90

**Reliability score: mean(84, 90)=87**

## Water Toxicity Data Summary

*Cyprinodon variegatus*

Fipronil

MB 46030

Machado MW. (1993) MB 46030-Acute toxicity to sheepshead minnow (*Cyprinodon variegatus*) under flow-through conditions. Springborn Laboratories, Inc., Wareham, Massachusetts. Laboratory study number 10566.0393.6267.505. Submitted to Rhone-Poulenc Ag Company, Research Triangle, North Carolina. USEPA MRID 43291702. CA DPR 157284.

Relevance

Score: 85

Rating: L

Reliability

Score: 92.5

Rating: R

Relevance points taken off for: Freshwater (15). 100-15=85

|  | <b>Machado 1993</b>                                | <b><i>C. variegatus</i></b> |
|--|--|-----------------------------|
| <b>Parameter</b>                             | <b>Value</b>                                       | <b>Comment</b>              |
| Test method cited                            | FIFRA 72-3   |                             |
| Phylum/subphylum                             | Chordata   |                             |
| Class  | Actinopterygii                                     |                             |
| Order  | Cyprinodontiformes                                 |                             |
| Family                                       | Cyprinodontidae                                    |                             |
| Genus  | <i>Cyprinodon</i>                                  |                             |
| Species                                      | <i>variegatus</i>                                  |                             |
| Family native to North America?              | Yes  |                             |
| Age/size at start of test/growth phase       | 0.29 g<br>26 mm                                    |                             |
| Source of organisms                          | Aquatic Biosystems, Fort Collins, Colorado         |                             |
| Have organisms been exposed to contaminants? | No   |                             |
| Animals acclimated and disease-free?         | 14 d   |                             |
| Animals randomized?                          | Yes  |                             |
| Test vessels randomized?                     | Yes  |                             |
| Test duration                                | 96 h   |                             |
| Data for multiple times?                     | 24, 48, 72, 96 h                                   |                             |
| Effect 1                                     | Survival   |                             |
| Control response 1                           | 100 %  |                             |
| Temperature                                  | 22 ± 1 °C  |                             |
| Test type                                    | Flow through                                       |                             |
| Photoperiod/light intensity                  | 16l:8d/30-40 footcandles                           |                             |
| Dilution water                               | Filtered natural seawater, Cape Cod Canal, Bourne, | 31-32 ‰                     |

|   | <b>Machado 1993</b>  | <b><i>C. variegatus</i></b>  |
|---|--|--|
| <b>Parameter</b>  | <b>Value</b>   | <b>Comment</b>   |
|   | Massachusetts  |  |
| pH  | 7.9-8.0  |  |
| Dissolved Oxygen  | 6.9-7.3mg/L  | 79-83 %  |
| Feeding   | Not fed  |  |
| Purity of test substance  | 96.1 %   |  |
| Concentrations measured?  | Yes  |  |
| Measured is what % of nominal?  | 76-113 %   |  |
| Toxicity values calculated based on nominal or measured concentrations? | Measured   |  |
| Chemical method documented?   | HPLC   |  |
| Concentration of carrier (if any) in test solutions                     | Acetone, 0.075 mL/L  |  |
| Concentration 1 Nom; Meas (µg/L)  | 97; 110  | 2 reps, 20/rep   |
| Concentration 2 Nom; Meas (µg/L)  | 160; 150   |  |
| Concentration 3 Nom; Meas (µg/L)  | 270; 240   |  |
| Concentration 4 Nom; Meas (µg/L)  | 450; 340   |  |
| Concentration 5 Nom; Meas (µg/L)  | 750; 660   |  |
| Control   | Negative: 0; 0<br>Solvent: 0; 0  |  |
| LC <sub>50</sub> (95% CI) (µg/L)  | 24 h: 300 (240-340) <sup>a</sup><br>48 h: 180 (150-200) <sup>b</sup><br>72 h: 180 (150-200) <sup>b</sup><br>96 h: 130 (110-150) <sup>a</sup> | Method: Non-linear interpolation <sup>a</sup> ; probit <sup>b</sup>                |
| NOEC  | <110   | Method: Not reported<br>p: Not reported<br>MSD: Not reported                       |
| LOEC  | 24 h: 340<br>48 h: 150<br>72 h: 150<br>96 h: 150   |  |
| MATC (GeoMean NOEC, LOEC)   | Not calculable   |  |
| % control at NOEC   | Not calculable   |  |
| % control at LOEC   | 24 h: 15 %<br>48 h: 50 %<br>72 h: 50 %<br>96 h: 0%   | 24 h: 15/100 = 15%<br>48 h: 50/100 = 50%<br>72 h: 50/100 = 50%<br>96 h: 0/100 = 0% |

Notes: Dilution water TOC 2.0 mg/L

Solubility (S) of fipronil = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Hardness (2), Alkalinity (2), Conductivity (2), Statistical significance (2), Significance level (2), Minimum significant difference (2). Total:  $100 - 12 = 88$

Acceptability: Hardness (2), Alkalinity (2), Conductivity (1), Minimum significant difference (1), % control at NOEC (1). Total:  $100 - 7 = 93$

**Reliability score: mean(88, 93)=90.5**

## Water Toxicity Data Summary

*Daphnia magna*  
Fipronil sulfide  
MB 45950

Iwafune T, Yokoyama A, Nagai T and Horio T. (2011) Evaluation of the risk of mixtures of paddy insecticides and their transformation products to aquatic organisms in the Sakura River, Japan. *Environmental Toxicology and Chemistry*, 30(8), 1834-1842.

Relevance  
Score: 82.5  
Rating: L

Reliability  
Score: 69  
Rating: L

Relevance points taken off for: Standard method (10), Control described (7.5). 100-17.5= 82.5

|  | <b>Iwafune 2011</b>                  | <b><i>D. magna</i></b> |
|--|--------------------------------------|------------------------|
| <b>Parameter</b>                             | <b>Value</b>                         | <b>Comment</b>         |
| Test method cited                            | Not reported                         |                        |
| Phylum/subphylum                             | Arthropoda/Crustacea                 |                        |
| Class  | Branchiopoda                         |                        |
| Order  | Cladocera                            |                        |
| Family                                       | Daphniidae                           |                        |
| Genus  | <i>Daphnia</i>                       |                        |
| Species                                      | <i>magna</i>                         |                        |
| Family native to North America?              | Yes                                  |                        |
| Age/size at start of test/growth phase       | 1 <sup>st</sup> instar               |                        |
| Source of organisms                          | Not reported                         |                        |
| Have organisms been exposed to contaminants? | Not reported                         |                        |
| Animals acclimated and disease-free?         | Not reported                         |                        |
| Animals randomized?                          | Not reported                         |                        |
| Test vessels randomized?                     | Not reported                         |                        |
| Test duration                                | 48 h                                 |                        |
| Data for multiple times?                     | No                                   |                        |
| Effect 1                                     | Immobilization                       |                        |
| Control response 1                           | ≤5 %                                 |                        |
| Temperature                                  | 20.9 ± 0.6 °C                        |                        |
| Test type                                    | Static                               |                        |
| Photoperiod/light intensity                  | Not reported                         |                        |
| Dilution water                               | Dechlorinated and filtered tap water |                        |
| pH   | 8                                    |                        |
| Hardness                                     | 70 mg/L CaCO <sub>3</sub>            |                        |

|   | <b>Iwafune 2011</b>  | <b><i>D. magna</i></b>    |
|---|--|---------------------------|
| <b>Parameter</b>  | <b>Value</b>   | <b>Comment</b>            |
| Alkalinity  | Not reported   |                           |
| Conductivity  | Not reported   |                           |
| Dissolved Oxygen  | Not reported   |                           |
| Feeding   | Not reported   |                           |
| Purity of test substance  | >98 %  |                           |
| Concentrations measured?  | Yes  |                           |
| Measured is what % of nominal?  | 84.1-105.3 %   |                           |
| Toxicity values calculated based on nominal or measured concentrations? | Measured   |                           |
| Chemical method documented?   | LC-MS/MS   |                           |
| Concentration of carrier (if any) in test solutions                     | Acetone or acetonitrile, ≤1 mL/L   |                           |
| Concentration 1 Nom; Meas (µg/L)  | Concentrations not reported but nominal range 12.0-154 across 7 treatments | Reps not reported, 20/rep |
| Control   | Not reported   |                           |
| EC <sub>50</sub> (95% CI) (µg/L)  | 28.0 (22.6-33.8)   | Method: probit            |

Notes:

Solubility value for fipronil sulfide (MB 45950) not available. Solubility (S) of fipronil parent compound (MB 46030) = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Organism source (5), Nominal concentrations (3), Measured concentrations (3), Alkalinity (2), Dissolved oxygen (4), Conductivity (2), Photoperiod (3), Statistical significance (2), Significance level (2), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100-30 =70

Acceptability: Standard method (5), No prior contamination (4), Organisms randomized (1), Feeding (3), Acclimation (1), Alkalinity (2), Dissolved oxygen (6), Conductivity (1), Photoperiod (2), Random design (2), Statistical method (2), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1). Total: 100-32 =68

**Reliability score: mean(70, 68)=69**

## Water Toxicity Data Summary

*Daphnia magna*

Fipronil

MB 46030

Iwafune T, Yokoyama A, Nagai T and Horio T. (2011) Evaluation of the risk of mixtures of paddy insecticides and their transformation products to aquatic organisms in the Sakura River, Japan. *Environmental Toxicology and Chemistry*, 30(8), 1834-1842.

Relevance

Score: 82.5

Rating: L

Reliability

Score: 69

Rating: L

Relevance points taken off for: Standard method (10), Control described (7.5). 100-17.5= 82.5

| <b>Parameter</b>                             | <b>Iwafune 2011<br/>Value</b>        | <b><i>D. magna</i><br/>Comment</b> |
|--|--------------------------------------|------------------------------------|
| Test method cited                            | Not reported                         |                                    |
| Phylum/subphylum                             | Arthropoda/Crustacea                 |                                    |
| Class  | Branchiopoda                         |                                    |
| Order  | Cladocera                            |                                    |
| Family                                       | Daphniidae                           |                                    |
| Genus  | <i>Daphnia</i>                       |                                    |
| Species                                      | <i>magna</i>                         |                                    |
| Family native to North America?              | Yes                                  |                                    |
| Age/size at start of test/growth phase       | 1 <sup>st</sup> instar               |                                    |
| Source of organisms                          | Not reported                         |                                    |
| Have organisms been exposed to contaminants? | Not reported                         |                                    |
| Animals acclimated and disease-free?         | Not reported                         |                                    |
| Animals randomized?                          | Not reported                         |                                    |
| Test vessels randomized?                     | Not reported                         |                                    |
| Test duration                                | 48 h                                 |                                    |
| Data for multiple times?                     | No                                   |                                    |
| Effect 1                                     | Immobilization                       |                                    |
| Control response 1                           | ≤5 %                                 |                                    |
| Temperature                                  | 20.9 ± 0.6 °C                        |                                    |
| Test type                                    | Static                               |                                    |
| Photoperiod/light intensity                  | Not reported                         |                                    |
| Dilution water                               | Dechlorinated and filtered tap water |                                    |
| pH   | 8                                    |                                    |
| Hardness                                     | 70 mg/L CaCO <sub>3</sub>            |                                    |

|   | <b>Iwafune 2011</b>  | <b><i>D. magna</i></b>    |
|---|--|---------------------------|
| <b>Parameter</b>  | <b>Value</b>   | <b>Comment</b>            |
| Alkalinity  | Not reported   |                           |
| Conductivity  | Not reported   |                           |
| Dissolved Oxygen  | Not reported   |                           |
| Feeding   | Not reported   |                           |
| Purity of test substance  | >98 %  |                           |
| Concentrations measured?  | Yes  |                           |
| Measured is what % of nominal?  | 84.1-105.3 %   |                           |
| Toxicity values calculated based on nominal or measured concentrations? | Measured   |                           |
| Chemical method documented?   | LC-MS/MS   |                           |
| Concentration of carrier (if any) in test solutions                     | Acetone or acetonitrile, ≤1 mL/L   |                           |
| Concentration 1 Nom; Meas (µg/L)  | Concentrations not reported but nominal range 12.0-126 across 6 treatments | Reps not reported, 20/rep |
| Control   | Not reported   |                           |
| EC <sub>50</sub> (95% CI) (µg/L)  | 42.9 (35.9-51.7)   | Method: probit            |

Notes:

Solubility (S) of fipronil = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Organism source (5), Nominal concentrations (3), Measured concentrations (3), Alkalinity (2), Dissolved oxygen (4), Conductivity (2), Photoperiod (3), Statistical significance (2), Significance level (2), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100-30 =70

Acceptability: Standard method (5), No prior contamination (4), Organisms randomized (1), Feeding (3), Acclimation (1), Alkalinity (2), Dissolved oxygen (6), Conductivity (1), Photoperiod (2), Random design (2), Statistical method (2), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1). Total: 100-32 =68

**Reliability score: mean(70, 68)=69**

## Water Toxicity Data Summary

*Daphnia magna*  
Fipronil sulfone  
MB 46136

Iwafune T, Yokoyama A, Nagai T and Horio T. (2011) Evaluation of the risk of mixtures of paddy insecticides and their transformation products to aquatic organisms in the Sakura River, Japan. *Environmental Toxicology and Chemistry*, 30(8), 1834-1842.

Relevance  
Score: 82.5  
Rating: L

Reliability  
Score: 69  
Rating: L

Relevance points taken off for: Standard method (10), Control described (7.5). 100-17.5= 82.5

|  | <b>Iwafune 2011</b>                  | <b><i>D. magna</i></b> |
|--|--------------------------------------|------------------------|
| <b>Parameter</b>                             | <b>Value</b>                         | <b>Comment</b>         |
| Test method cited                            | Not reported                         |                        |
| Phylum/subphylum                             | Arthropoda/Crustacea                 |                        |
| Class  | Branchiopoda                         |                        |
| Order  | Cladocera                            |                        |
| Family                                       | Daphniidae                           |                        |
| Genus  | <i>Daphnia</i>                       |                        |
| Species                                      | <i>magna</i>                         |                        |
| Family native to North America?              | Yes                                  |                        |
| Age/size at start of test/growth phase       | 1 <sup>st</sup> instar               |                        |
| Source of organisms                          | Not reported                         |                        |
| Have organisms been exposed to contaminants? | Not reported                         |                        |
| Animals acclimated and disease-free?         | Not reported                         |                        |
| Animals randomized?                          | Not reported                         |                        |
| Test vessels randomized?                     | Not reported                         |                        |
| Test duration                                | 48 h                                 |                        |
| Data for multiple times?                     | No                                   |                        |
| Effect 1                                     | Immobilization                       |                        |
| Control response 1                           | ≤5 %                                 |                        |
| Temperature                                  | 20.9 ± 0.6 °C                        |                        |
| Test type                                    | Static                               |                        |
| Photoperiod/light intensity                  | Not reported                         |                        |
| Dilution water                               | Dechlorinated and filtered tap water |                        |
| pH   | 8                                    |                        |

|   | <b>Iwafune 2011</b>   | <b><i>D. magna</i></b>    |
|---|---|---------------------------|
| <b>Parameter</b>  | <b>Value</b>  | <b>Comment</b>            |
| Hardness  | 70 mg/L CaCO <sub>3</sub>   |                           |
| Alkalinity  | Not reported  |                           |
| Conductivity  | Not reported  |                           |
| Dissolved Oxygen  | Not reported  |                           |
| Feeding   | Not reported  |                           |
| Purity of test substance  | >98 %   |                           |
| Concentrations measured?  | Yes   |                           |
| Measured is what % of nominal?  | 84.1-105.3 %  |                           |
| Toxicity values calculated based on nominal or measured concentrations? | Measured  |                           |
| Chemical method documented?   | LC-MS/MS  |                           |
| Concentration of carrier (if any) in test solutions                     | Acetone or acetonitrile, ≤1 mL/L  |                           |
| Concentration 1 Nom; Meas (µg/L)  | Concentrations not reported but nominal range 1.60-18.2 across 7 treatments | Reps not reported, 20/rep |
| Control   | Not reported  |                           |
| EC <sub>50</sub> (95% CI) (µg/L)  | 5.17 (2.45-3.28)  | Method: probit            |

Notes:

Solubility (S) value for fipronil sulfone (MB 46136) = 160 µg/L, 2S = 320 µg/L.

Reliability points taken off for:

Documentation: Organism source (5), Nominal concentrations (3), Measured concentrations (3), Alkalinity (2), Dissolved oxygen (4), Conductivity (2), Photoperiod (3), Statistical significance (2), Significance level (2), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100-30 =70

Acceptability: Standard method (5), No prior contamination (4), Organisms randomized (1), Feeding (3), Acclimation (1), Alkalinity (2), Dissolved oxygen (6), Conductivity (1), Photoperiod (2), Random design (2), Statistical method (2), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1). Total: 100-32 =68

**Reliability score: mean(70, 68)=69**

## Water Toxicity Data Summary

*Dunaliella tertiolecta*

Fipronil

MB 46030

Overmyer JP, Rouse DR, Avants JK, Garrison AW, DeLorenzo ME, Chung KW, Key PB, Wilson WA and Black MC. (2007) Toxicity of fipronil and its enantiomers to marine and freshwater non-targets. *Journal of Environmental Science and Health Part B*, 42(5), 471-480.

Relevance

Score: 85

Rating: L

Reliability

Score: 91.5

Rating: R

Relevance points taken off for: Freshwater (15). 100-15=85

|  | <b>Overmyer et al. 2007</b>   | <b><i>D. tertiolecta</i></b>   |
|--|---|--|
| <b>Parameter</b>                             | <b>Value</b>  | <b>Comment</b>   |
| Test method cited                            | American Society for Testing and Materials (ASTM). Annual Book of ASTM Standards; ASTM: West Conshohocken, PA, 1996; Vol. 11.05, 575–586. |  |
| Phylum/subphylum                             | Chlorophyta   |  |
| Class  | Chlorophyceae   |  |
| Order  | Chlamydomonadales   |  |
| Family                                       | Dunaliellaceae  |  |
| Genus  | <i>Dunaliella</i>   |  |
| Species                                      | <i>tertiolecta</i>  |  |
| Family native to North America?              | Yes   |  |
| Age/size at start of test/growth phase       | Log phase growth  |  |
| Source of organisms                          | University of Texas Culture Collection  |  |
| Have organisms been exposed to contaminants? | No  |  |
| Animals acclimated and disease-free?         | 48 h  |  |
| Animals randomized?                          | Not reported  | Given organism size and presence in growth medium, it is assumed that aliquots are inherently randomly |

|   | <b>Overmyer et al. 2007</b>                 | <b><i>D. tertiolecta</i></b>                    |
|---|---|---|
| <b>Parameter</b>  | <b>Value</b>                                | <b>Comment</b>                                  |
| Test vessels randomized?  | Yes   |   |
| Test duration   | 96 h  |   |
| Data for multiple times?  | No  |   |
| Effect 1  | Cell density                                |   |
| Control response 1  | Not reported                                |   |
| Temperature   | 25 °C                                       |   |
| Test type   | Static                                      |   |
| Photoperiod/light intensity   | 16l:8d/86 µE/m-2s-1                         |   |
| Dilution water  | Guillard's F/2 marine medium                | 20 ‰ salinity                                   |
| pH  | Measured but not reported                   |   |
| Hardness  | Not reported                                |   |
| Alkalinity  | Not reported                                |   |
| Conductivity  | Measured but not reported                   |   |
| Dissolved Oxygen  | Measured but not reported                   |   |
| Feeding   | Not fed                                     |   |
| Purity of test substance  | 98 %  |   |
| Concentrations measured?  | Yes   |   |
| Measured is what % of nominal?  | Racemate: 92-106 %                          | Only highest and lowest concentrations measured |
| Toxicity values calculated based on nominal or measured concentrations? | Nominal                                     |   |
| Chemical method documented?   | GCMS  |   |
| Concentration of carrier (if any) in test solutions                     | Acetone, 0.1%                               |   |
| Concentration 1 Nom; Meas (µg/L)  | Racemate: 63;<br>(+): 100;<br>(-): 100;     | 3 reps, 1,250,000 cells/rep                     |
| Concentration 2 Nom; Meas (µg/L)  | Racemate: 125;<br>(+): 200;<br>(-): 200;    |   |
| Concentration 3 Nom; Meas (µg/L)  | Racemate: 250;<br>(+): 400;<br>(-): 400;    |   |
| Concentration 4 Nom; Meas (µg/L)  | Racemate: 500;<br>(+): 800;<br>(-): 800;    |   |
| Concentration 5 Nom; Meas (µg/L)  | Racemate: 1000;<br>(+): 1600;<br>(-): 1600; |   |

|                                  | <b>Overmyer et al. 2007</b>                | <i>D. tertiolecta</i>           |
|----------------------------------|--|---------------------------------|
| <b>Parameter</b>                 | <b>Value</b>                               | <b>Comment</b>                  |
| Control                          | All: Negative<br>All: Solvent              |                                 |
| EC <sub>50</sub> (95% CI) (µg/L) | Racemate: 631.20 (no CI limits determined) | Method: Trimmed Spearman-Kärber |
| NOEC (µg/L)                      | 250  |                                 |
| LOEC (µg/L)                      | 500  |                                 |
| MATC                             | 354  |                                 |

Notes: Raw data not available so % control at NOEC/LOEC not calculable.

Reliability points were not taken off for water quality parameters (hardness, alkalinity, conductivity) because there is no guidance for these parameters in the test guidelines for algal/plant studies and the medium is presumably appropriate for the test species because a specific culture media was used.

Solubility (S) of fipronil = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Measured concentrations (3), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100-7 =93

Acceptability: Measured concentrations (4), Temperature variation (3), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1). Total: 100-10 =90

**Reliability score: mean(93, 90)=91.5**

## Water Toxicity Data Summary

*Elliptio complanata*

Fipronil

MB 46030

Bringolf RB, Cope WG, Eads CB, Lazaro PR, Barnhart MC and Shea D. (2007) Acute and chronic toxicity of technical-grade pesticides to glochidia and juveniles of freshwater mussels (unionidae). *Environmental Toxicology and Chemistry*, 26(10), 2086-2093.

Relevance

Score: 85

Rating: L

Reliability

Score: 79

Rating: R

Relevance points taken off for: Point estimates (10). 100-15=85

| <b>Parameter</b>                             | <b>Bringolf 2007<br/>Value</b>  | <b><i>E. complanata</i><br/>Comment</b> |
|--|---|---|
| Test method cited                            | ASTM 2006: Standard guide for conducting laboratory toxicity tests with freshwater mussels. E2455-06. |   |
| Phylum/subphylum                             | Mollusca  |   |
| Class  | Bivalvia  |   |
| Order  | Unionoida   |   |
| Family                                       | Unionidae   |   |
| Genus  | <i>Elliptio</i>   |   |
| Species                                      | <i>complanata</i>   |   |
| Family native to North America?              | Yes   |   |
| Age/size at start of test/growth phase       | Glochidia   |   |
| Source of organisms                          | Brooding adult females collected from rural Richland Creek, Wake County, Missouri                     |   |
| Have organisms been exposed to contaminants? | Not reported  |   |
| Animals acclimated and disease-free?         | Yes   |   |
| Animals randomized?                          | Not reported  |   |
| Test vessels randomized?                     | Not reported  |   |
| Test duration                                | 96 h  |   |
| Data for multiple times?                     | 48, 96 h  |   |
| Effect 1                                     | Survival  |   |
| Control response 1                           | >90 %   |   |

|   | <b>Bringolf 2007</b>                           | <i>E. complanata</i>            |
|---|--|---------------------------------|
| <b>Parameter</b>  | <b>Value</b>                                   | <b>Comment</b>                  |
| Temperature   | 21 ± 1 °C                                      |                                 |
| Test type   | Static renewal                                 | Renewed at 48 h                 |
| Photoperiod/light intensity   | Not reported                                   |                                 |
| Dilution water  | Reconstituted hard water                       | ASTM 2006                       |
| pH  | 8.32-8.61                                      |                                 |
| Hardness  | 170-192 mg/L CaCO <sub>3</sub>                 |                                 |
| Alkalinity  | 116-130 mg/L CaCO <sub>3</sub>                 |                                 |
| Conductivity  | 523-625 µmhos/cm                               |                                 |
| Dissolved Oxygen  | >80 %  |                                 |
| Feeding   | Not reported                                   |                                 |
| Purity of test substance  | 99.7 %   |                                 |
| Concentrations measured?  | Yes  |                                 |
| Measured is what % of nominal?  | Mean: 64.6 %                                   |                                 |
| Toxicity values calculated based on nominal or measured concentrations? | Measured                                       |                                 |
| Chemical method documented?   | GC   |                                 |
| Concentration of carrier (if any) in test solutions                     | Acetone, concentration not reported            |                                 |
| Concentration 1 Nom; Meas (µg/L)  | 5-6 concentrations tested; values not reported | 3 reps, 150-200 glochidia/rep   |
| Control   | Negative Solvent                               |                                 |
| EC <sub>50</sub> (95% CI) (µg/L)  | Glochidia:<br>24 h: >2000                      | Method: Trimmed Spearman-Kärber |

Notes:

Solubility (S) of fipronil = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Nominal concentrations (3), Measured concentrations (3), Photoperiod (3), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100-13 =87

Acceptability: Measured concentrations (4), Concentrations not > 2x solubility (4), Carrier solvent (4), No prior contamination (4), Organisms randomized (1), Photoperiod (2), Random design (2), Dilution factor (2), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1), Point estimates (3). Total: 100-29 =71

**Reliability score: mean(87, 71)=79**

## Water Toxicity Data Summary

*Ephemerella excrucians*

Fipronil

MB46030

Weston DP and Lydy MJ. (2014) Toxicity of the insecticide fipronil and its degradates to benthic macroinvertebrates of urban streams. *Environmental science & technology*, 48(2), 1290-1297.

Relevance

Score: 75

Rating: L

Reliability

Score: 80

Rating: R

Relevance points taken off for: Standard method (10), Toxicity value (15). 100-25=75

|  | <b>Weston 2014</b>  | <i>E. excrucians</i> |
|--|---|----------------------|
| <b>Parameter</b>                             | <b>Value</b>  | <b>Comment</b>       |
| Test method cited                            | Not reported  |                      |
| Phylum/subphylum                             | Anthropoda  |                      |
| Class  | Insecta   |                      |
| Order  | Ephemeroptera   |                      |
| Family                                       | Ephemerellidae  |                      |
| Genus  | <i>Ephemerella</i>  |                      |
| Species                                      | <i>excrucians</i>   |                      |
| Family native to North America?              | Yes   |                      |
| Age/size at start of test/growth phase       | Not reported  |                      |
| Source of organisms                          | Urban waterbodies with minimal development in Northern California |                      |
| Have organisms been exposed to contaminants? | Not reported  |                      |
| Animals acclimated and disease-free?         | 24 h  |                      |
| Animals randomized?                          | Not reported  |                      |
| Test vessels randomized?                     | Not reported  |                      |
| Test duration                                | 48 h  |                      |
| Data for multiple times?                     | Not reported  |                      |
| Effect 1                                     | Survival  |                      |
| Control response 1                           | 100 %   |                      |
| Effect 2                                     | Immobilization (ability to swim)                                  |                      |
| Control response 2                           | Not reported  |                      |
| Temperature                                  | 13 °C   |                      |
| Test type                                    | Static  |                      |
| Photoperiod/light intensity                  | 16l:8d; Not reported  |                      |

|   | <b>Weston 2014</b>   | <b><i>E. excrucians</i></b>                             |
|---|--|---|
| <b>Parameter</b>  | <b>Value</b>   | <b>Comment</b>  |
| Dilution water  | Milli-Q purified, deionized watermade moderately hard by addition of salts | *According to EPA 821-R-02-012                          |
| pH  | Not reported*  |   |
| Hardness  | Not reported*  |   |
| Alkalinity  | Not reported*  |   |
| Conductivity  | Not reported*  |   |
| Dissolved Oxygen  | Not reported*  |   |
| Feeding   | Not reported   |   |
| Purity of test substance  | 99.50 %  | Not reported but author verified from chemical supplier |
| Concentrations measured?  | Yes  |   |
| Measured is what % of nominal?  | 66-113%  |   |
| Toxicity values calculated based on nominal or measured concentrations? | Measured   |   |
| Chemical method documented?   | GC   |   |
| Concentration of carrier (if any) in test solutions                     | Acetone, <36 µg/L  |   |
| Concentration 1 Nom; Meas (µg/L)  | Not reported; 4-7 concentrations tested; dilution factor of 2              | 3 reps, 4-6/rep   |
| Control   | Negative: 0; 0<br>Solvent: 0; 0  |   |
| LC <sub>50</sub> (95% CI) (µg/L)  | >0.436   | Method: Probit  |
| EC <sub>50</sub> (95% CI) (µg/L)  | >0.436   | Method: Probit  |

Notes: Points were not deducted for lack of water quality parameter reporting because water was prepared according to EPA 821-R-02-012 and measured during study (but not reported).

Solubility (S) of fipronil = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Organism life stage/size (5), Nominal concentrations (3), Measured concentrations (3), Minimum significant difference (2), % control at NOEC/LOEC (2), Point estimates (8). Total: 100-23 =77

Acceptability: Standard method (5), Organisms randomized (1), Temperature variation (3), Random design (2), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1). Total: 100-17 =83

**Reliability score: mean(77,83)=80**

## Water Toxicity Data Summary

*G. paripes*  
Fipronil  
MB 46030

Ali A, Nayar JK and Gu WD. (1998) Toxicity of a phenyl pyrazole insecticide, fipronil, to mosquito and chironomid midge larvae in the laboratory. *Journal of the American Mosquito Control Association*, 14(2), 216-218.

Relevance  
Score: 92.5  
Rating: R

Reliability  
Score: 60.5  
Rating: L

Relevance points taken off for: Control response (7.5).  $100-7.5=92.5$

|  | <b>Ali 1998</b>   | <b><i>G. paripes</i></b> |
|--|---|--------------------------|
| <b>Parameter</b>                             | <b>Value</b>  | <b>Comment</b>           |
| Test method cited                            | Mulla MS and Khasawinah AM. (1969) Laboratory and field evaluation of larvicides against chironomid midges. <i>Journal of Economic Entomology</i> , 62(1), 37-41. |                          |
| Phylum/subphylum                             | Arthropoda  |                          |
| Class  | Insecta   |                          |
| Order  | Diptera   |                          |
| Family                                       | Chironomidae  |                          |
| Genus  | <i>Glyptotendipes</i>   |                          |
| Species                                      | <i>paripes</i>  |                          |
| Family native to North America?              | Yes   |                          |
| Age/size at start of test/growth phase       | 4 <sup>th</sup> instar  |                          |
| Source of organisms                          | Collected from Lake Jessup, central Florida   |                          |
| Have organisms been exposed to contaminants? | No  |                          |
| Animals acclimated and disease-free?         | Yes   |                          |
| Animals randomized?                          | Not reported  |                          |
| Test vessels randomized?                     | Not reported  |                          |
| Test duration                                | 48 h  |                          |
| Data for multiple times?                     | 48 h  |                          |
| Effect 1                                     | Survival  |                          |
| Control response 1                           | Not reported  |                          |
| Temperature                                  | 26 ± 2 °C   |                          |

|   | <b>Ali 1998</b>  | <b><i>G. paripes</i></b>           |
|---|--|------------------------------------|
| <b>Parameter</b>  | <b>Value</b>   | <b>Comment</b>                     |
| Test type   | Static   |                                    |
| Photoperiod/light intensity   | 14:10d   |                                    |
| Dilution water  | Tap water  |                                    |
| pH  | Not reported   |                                    |
| Hardness  | Not reported   |                                    |
| Alkalinity  | Not reported   |                                    |
| Conductivity  | Not reported   |                                    |
| Dissolved Oxygen  | Not reported   |                                    |
| Feeding   | 1 mL 1% beef liver plus yeast (1:1) daily                                  |                                    |
| Purity of test substance  | 97.1 %   |                                    |
| Concentrations measured?  | Not reported   |                                    |
| Measured is what % of nominal?  | Not reported   |                                    |
| Toxicity values calculated based on nominal or measured concentrations? | Not reported   |                                    |
| Chemical method documented?   | Not reported   |                                    |
| Concentration of carrier (if any) in test solutions                     | Acetone, concentration not reported  |                                    |
| Concentration 1 Nom; Meas ( $\mu\text{g/L}$ )                           | 6-7 concentrations tested but not reported                                 | 3 reps, 20/rep                     |
| Control   | Negative   |                                    |
| LC <sub>50</sub> (95% CI) ( $\mu\text{g/L}$ )                           | 24 h: 0.00091 (0.00055-0.00055-0.00141)<br>48 h: 0.00042 (0.00016-0.00080) | Method: log-dose-probit regression |

Notes:

Solubility (S) of fipronil = 1650.8  $\mu\text{g/L}$ , 2S = 3301.6  $\mu\text{g/L}$ .

Reliability points taken off for:

Documentation: Analytical method (4), Nominal concentrations (3), Measured concentrations (3), Hardness (2), Alkalinity (2), Dissolved oxygen (4), Conductivity (2), pH (3), Statistical significance (2), Significance level (2), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100-31 =69

Acceptability: Control response (9), Measured concentrations within 20% nominal (4), Concentrations not > 2x solubility (4), Carrier solvent (4), Organisms randomized (1), Feeding (3), Hardness (2), Alkalinity (2), Dissolved oxygen (6), Conductivity (1), pH (2), Random design (2), Dilution factor (2), Hypothesis tests (3), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1). Total: 100-48 =52

**Reliability score: mean(69, 52)=60.5**

## Water Toxicity Data Summary

*Lampsilis fasciola*

Fipronil

MB 46030

Bringolf RB, Cope WG, Eads CB, Lazaro PR, Barnhart MC and Shea D. (2007) Acute and chronic toxicity of technical-grade pesticides to glochidia and juveniles of freshwater mussels (unionidae). *Environmental Toxicology and Chemistry*, 26(10), 2086-2093.

Relevance

Score: 85

Rating: L

Reliability

Score: 79

Rating: R

Relevance points taken off for: Point estimates (10). 100-15=85

| <b>Parameter</b>                             | <b>Bringolf 2007<br/>Value</b>  | <b><i>L. fasciola</i><br/>Comment</b> |
|--|---|---------------------------------------|
| Test method cited                            | ASTM 2006: Standard guide for conducting laboratory toxicity tests with freshwater mussels. E2455-06. |                                       |
| Phylum/subphylum                             | Mollusca  |                                       |
| Class  | Bivalvia  |                                       |
| Order  | Unionoida   |                                       |
| Family                                       | Unionidae   |                                       |
| Genus  | <i>Lampsilis</i>  |                                       |
| Species                                      | <i>fasciola</i>   |                                       |
| Family native to North America?              | Yes   |                                       |
| Age/size at start of test/growth phase       | ≤2 m, juvenile<br>Glochidia   |                                       |
| Source of organisms                          | Brooding adult females collected from rural Richland Creek, Wake County, Missouri                     |                                       |
| Have organisms been exposed to contaminants? | Not reported  |                                       |
| Animals acclimated and disease-free?         | Yes   |                                       |
| Animals randomized?                          | Not reported  |                                       |
| Test vessels randomized?                     | Not reported  |                                       |
| Test duration                                | 96 h  |                                       |
| Data for multiple times?                     | 48, 96 h  |                                       |
| Effect 1                                     | Survival  |                                       |
| Control response 1                           | >90 %   |                                       |

|   | <b>Bringolf 2007</b>  | <i>L. fasciola</i>              |
|---|---|---------------------------------|
| <b>Parameter</b>  | <b>Value</b>  | <b>Comment</b>                  |
| Temperature   | 21 ± 1 °C   |                                 |
| Test type   | Static renewal  | Renewed at 48 h                 |
| Photoperiod/light intensity   | Not reported  |                                 |
| Dilution water  | Reconstituted hard water  | ASTM 2006                       |
| pH  | 8.32-8.61   |                                 |
| Hardness  | 170-192 mg/L CaCO <sub>3</sub>  |                                 |
| Alkalinity  | 116-130 mg/L CaCO <sub>3</sub>  |                                 |
| Conductivity  | 523-625 µmhos/cm  |                                 |
| Dissolved Oxygen  | >80 %   |                                 |
| Feeding   | Not reported  |                                 |
| Purity of test substance  | 99.7 %  |                                 |
| Concentrations measured?  | Yes   |                                 |
| Measured is what % of nominal?  | Mean: 64.6 %  |                                 |
| Toxicity values calculated based on nominal or measured concentrations? | Measured  |                                 |
| Chemical method documented?   | GC  |                                 |
| Concentration of carrier (if any) in test solutions                     | Acetone, concentration not reported                                       |                                 |
| Concentration 1 Nom; Meas (µg/L)  | 5-6 concentrations tested; values not reported                            | 3 reps, 150-200 glochidia/rep   |
| Control   | Negative Solvent  |                                 |
| EC <sub>50</sub> (95% CI) (µg/L)  | Glochidia:<br>24 h: >2000<br>48 h: >2000<br><br>Juveniles:<br>96 h: >2000 | Method: Trimmed Spearman-Kärber |

Notes:

Solubility (S) of fipronil = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Nominal concentrations (3), Measured concentrations (3), Photoperiod (3), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100-13 =87

Acceptability: Measured concentrations (4), Concentrations not > 2x solubility (4), Carrier solvent (4), No prior contamination (4), Organisms randomized (1), Photoperiod (2), Random design (2), Dilution factor (2), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1), Point estimates (3). Total: 100-29 =71

**Reliability score: mean(87, 71)=79**

## Water Toxicity Data Summary

*Lampsilis siliquoidea*

Fipronil

MB 46030

Bringolf RB, Cope WG, Eads CB, Lazaro PR, Barnhart MC and Shea D. (2007) Acute and chronic toxicity of technical-grade pesticides to glochidia and juveniles of freshwater mussels (unionidae). *Environmental Toxicology and Chemistry*, 26(10), 2086-2093.

Relevance

Score: 85

Rating: L

Reliability

Score: 79

Rating: R

Relevance points taken off for: Point estimates (10). 100-15=85

| <b>Parameter</b>                       | <b>Bringolf 2007 Value</b>   | <b><i>L. siliquoidea</i> Comment</b> |
|--|--|--------------------------------------|
| Test method cited                      | ASTM 2006: Standard guide for conducting laboratory toxicity tests with freshwater mussels. E2455-06.  |                                      |
| Phylum/subphylum                       | Mollusca   |                                      |
| Class                                  | Bivalvia   |                                      |
| Order                                  | Unionoida  |                                      |
| Family                                 | Unionidae  |                                      |
| Genus                                  | <i>Lampsilis</i>   |                                      |
| Species                                | <i>siliquoidea</i>   |                                      |
| Family native to North America?        | Yes  |                                      |
| Age/size at start of test/growth phase | ≤2 m, juvenile<br>Glochidia  |                                      |
| Source of organisms                    | Brooding adult females collected from rural Silver Fork of Perche Creek, Boone County, Missouri. Juveniles were produced on the campus of Missouri State University (Springfield, MO, USA) by transformation on juvenile largemouth bass obtained from the Missouri Department of Conservation Chesapeake Hatchery |                                      |

|   | <b>Bringolf 2007</b>                                       | <b><i>L. siliquoides</i></b>                     |
|---|--|--|
| <b>Parameter</b>  | <b>Value</b>   | <b>Comment</b>                                   |
|   | (Chesapeake, Missouri)                                     |  |
| Have organisms been exposed to contaminants?                            | Not reported   |  |
| Animals acclimated and disease-free?                                    | Yes  |  |
| Animals randomized?   | Not reported   |  |
| Test vessels randomized?  | Not reported   |  |
| Test duration   | 96 h   |  |
| Data for multiple times?  | 48, 96 h   |  |
| Effect 1  | Survival   |  |
| Control response 1  | >93 %  |  |
| Temperature   | 21 ± 1 °C  |  |
| Test type   | Static renewal   | Renewed at 48 h                                  |
| Photoperiod/light intensity   | Not reported   |  |
| Dilution water  | Reconstituted hard water                                   | ASTM 2006  |
| pH  | 8.32-8.61  |  |
| Hardness  | 170-192 mg/L CaCO <sub>3</sub>                             |  |
| Alkalinity  | 116-130 mg/L CaCO <sub>3</sub>                             |  |
| Conductivity  | 523-625 µmhos/cm   |  |
| Dissolved Oxygen  | >80 %  |  |
| Feeding   | Not reported   |  |
| Purity of test substance  | 99.7 %   |  |
| Concentrations measured?  | Yes  |  |
| Measured is what % of nominal?  | Mean:<br>Juveniles: 70.5 %<br>Glochidia 64.6 %             |  |
| Toxicity values calculated based on nominal or measured concentrations? | Measured   |  |
| Chemical method documented?   | GC   |  |
| Concentration of carrier (if any) in test solutions                     | Acetone, concentration not reported                        |  |
| Concentration 1 Nom; Meas (µg/L)  | 5-6 concentrations tested; values not reported             | 3 reps, 7 juveniles/rep or 150-200 glochidia/rep |
| Control   | Negative Solvent   |  |
| EC <sub>50</sub> (95% CI) (µg/L)  | Juveniles:<br>96 h: >2000<br><br>Glochidia:<br>24 h: >2000 | Method: Trimmed Spearman-Kärber                  |

|                  | <b>Bringolf 2007</b> | <i>L. siliquoides</i> |
|------------------|----------------------|-----------------------|
| <b>Parameter</b> | <b>Value</b>         | <b>Comment</b>        |
|                  | 48 h: >2000          |                       |

Notes:

Solubility (S) of fipronil = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Nominal concentrations (3), Measured concentrations (3), Photoperiod (3), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100-13 =87

Acceptability: Measured concentrations (4), Concentrations not > 2x solubility (4), Carrier solvent (4), No prior contamination (4), Organisms randomized (1), Photoperiod (2), Random design (2), Dilution factor (2), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1), Point estimates (3). Total: 100-29 =71

**Reliability score: mean(87, 71)=79**

## Water Toxicity Data Summary

*Lumbriculus variegatus*

Fipronil

MB 46030

Putt AE. (2003c) Fipronil-Acute toxicity to oligochaetes (*Lumbriculus variegatus*) under static-renewal conditions. Springborn Smithers Laboratories, Wareham, Massachusetts. Laboratory study number 986.6162. Submitted to BASF Corporation, Research Triangle Park, North Carolina. USEPA MRID 46329903.

Relevance

Score: 85

Rating: L

Reliability

Score: 92.5

Rating: R

Relevance points taken off for: Toxicity value (15). 100-15=85

|  | <b>Putt 2003</b>          | <i>L. variegatus</i> |
|--|---------------------------|----------------------|
| <b>Parameter</b>                             | <b>Value</b>              | <b>Comment</b>       |
| Test method cited                            | ASTM Guideline E-729      |                      |
| Phylum/subphylum                             | Annelida                  |                      |
| Class  | Clitellata                |                      |
| Order  | Lumbriculida              |                      |
| Family                                       | Lumbriculidae             |                      |
| Genus  | <i>Lumbriculus</i>        |                      |
| Species                                      | <i>variegatus</i>         |                      |
| Family native to North America?              | Yes                       |                      |
| Age/size at start of test/growth phase       | 0.0039 g                  |                      |
| Source of organisms                          | Laboratory cultures       |                      |
| Have organisms been exposed to contaminants? | No                        |                      |
| Animals acclimated and disease-free?         | Yes                       |                      |
| Animals randomized?                          | Yes                       |                      |
| Test vessels randomized?                     | Not reported              |                      |
| Test duration                                | 96 h                      |                      |
| Data for multiple times?                     | 24, 48, 72, 96 h          |                      |
| Effect 1                                     | Survival                  |                      |
| Control response 1                           | 100 %                     |                      |
| Temperature                                  | 22.5 ± 0.5 °C             |                      |
| Test type                                    | Static renewal            |                      |
| Photoperiod/light intensity                  | 16l:8d/54-72 footcandles  |                      |
| Dilution water                               | Well water                |                      |
| pH   | 7.6                       |                      |
| Hardness                                     | 36 mg/L CaCO <sub>3</sub> |                      |

|   | <b>Putt 2003</b>                | <i>L. variegatus</i>   |
|---|---------------------------------|--|
| <b>Parameter</b>  | <b>Value</b>                    | <b>Comment</b>   |
| Alkalinity  | 28 mg/L CaCO <sub>3</sub>       |  |
| Conductivity  | 120 µmhos/cm                    |  |
| Dissolved Oxygen  | 6.0-9.2 mg/L                    | 69-105 %   |
| Feeding   | Not fed                         |  |
| Purity of test substance  | 99.7 %                          |  |
| Concentrations measured?  | Yes                             |  |
| Measured is what % of nominal?  | 95-100 %                        |  |
| Toxicity values calculated based on nominal or measured concentrations? | Measured                        |  |
| Chemical method documented?   | GC/ECD                          |  |
| Concentration of carrier (if any) in test solutions                     | Acetone, 0.10 mL/L              |  |
| Concentration 1 Nom; Meas (µg/L)  | 260; 250                        | 4 reps, 5/rep  |
| Concentration 2 Nom; Meas (µg/L)  | 430; 410                        |  |
| Concentration 3 Nom; Meas (µg/L)  | 720; 710                        |  |
| Concentration 4 Nom; Meas (µg/L)  | 1200; 1200                      |  |
| Concentration 5 Nom; Meas (µg/L)  | 2000; 1900                      |  |
| Control   | Negative: 0; 0<br>Solvent: 0; 0 |  |
| LC <sub>50</sub> (95% CI) (µg/L)  | >1900                           | Method:<br>Empirically estimated   |
| NOEC  | 1900                            | Method:<br>Empirically estimated<br>p: Not reported<br>MSD: Not reported |
| % control at NOEC   | 100%                            |  |

Notes:

Solubility (S) of fipronil = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Statistical significance (2), Significance level (2), Minimum significant difference (2). Total: 100- 6=94

Acceptability: Random design (2), Statistical method (2), Minimum significant difference (1), % control at LOEC (1), Point estimates (3). Total: 100-9 =91

**Reliability score: mean(94, 91)=92.5**

## Water Toxicity Data Summary

*Mercenaria mercenaria*

Fipronil

MB 46030

Overmyer JP, Rouse DR, Avants JK, Garrison AW, DeLorenzo ME, Chung KW, Key PB, Wilson WA and Black MC. (2007) Toxicity of fipronil and its enantiomers to marine and freshwater non-targets. *Journal of Environmental Science and Health Part B*, 42(5), 471-480.

Relevance

Score: 85

Rating: L

Reliability

Score: 75.5

Rating: R

Relevance points taken off for: Freshwater (15). 100-15=85

|  | <b>Overmyer et al. 2007</b>                      | <b><i>M. mercenaria</i></b> |
|--|--|-----------------------------|
| <b>Parameter</b>                             | <b>Value</b>                                     | <b>Comment</b>              |
| Test method cited                            | States that standard methods used                |                             |
| Phylum/subphylum                             | Mollusca   |                             |
| Class  | Bivalvia   |                             |
| Order  | Veneroida  |                             |
| Family                                       | Veneridae  |                             |
| Genus  | <i>Mercenaria</i>                                |                             |
| Species                                      | <i>Mercenaria</i>                                |                             |
| Family native to North America?              | Yes  |                             |
| Age/size at start of test/growth phase       | Juvenile, 212-350 µm                             |                             |
| Source of organisms                          | Atlantic Farm Inc., James Island, South Carolina |                             |
| Have organisms been exposed to contaminants? | No   |                             |
| Animals acclimated and disease-free?         | 48 h   |                             |
| Animals randomized?                          | Not reported                                     |                             |
| Test vessels randomized?                     | Not reported                                     |                             |
| Test duration                                | 96 h   | Renewed every 24 h          |
| Data for multiple times?                     | No   |                             |
| Effect 1                                     | Survival   |                             |
| Control response 1                           | >90 %  |                             |
| Temperature                                  | 25 °C  |                             |
| Test type                                    | Static   |                             |
| Photoperiod/light intensity                  | 12l:12d  |                             |
| Dilution water                               | Seawater   | 30 ‰ salinity               |

|   | <b>Overmyer et al. 2007</b>  | <b><i>M. mercenaria</i></b>                     |
|---|--|---|
| <b>Parameter</b>  | <b>Value</b>   | <b>Comment</b>                                  |
| pH  | Measured but not reported  |   |
| Hardness  | Not reported   |   |
| Alkalinity  | Not reported   |   |
| Conductivity  | Measured but not reported  |   |
| Dissolved Oxygen  | Measured but not reported  |   |
| Feeding   | Not fed  |   |
| Purity of test substance  | 98 %   |   |
| Concentrations measured?  | Yes  |   |
| Measured is what % of nominal?  | Racemate: 92-106 %   | Only highest and lowest concentrations measured |
| Toxicity values calculated based on nominal or measured concentrations? | Nominal  |   |
| Chemical method documented?   | GCMS   |   |
| Concentration of carrier (if any) in test solutions                     | Acetone, concentration not reported  |   |
| Concentration 1 Nom; Meas (µg/L)  | Racemate: 1.9; Not reported<br>(+): 37.5; 35.60<br>(-): 37.5; 34.50              | 5 reps, 30/rep                                  |
| Concentration 2 Nom; Meas (µg/L)  | Racemate: 3.9; Not reported<br>(+): 75; Not reported<br>(-): 75; Not reported    |   |
| Concentration 3 Nom; Meas (µg/L)  | Racemate: 7.8; Not reported<br>(+): Not reported<br>(-): Not reported            |   |
| Concentration 4 Nom; Meas (µg/L)  | Racemate: 15.6; Not reported<br>(+): 150; Not reported<br>(-): 150; Not reported |   |
| Concentration 5 Nom; Meas (µg/L)  | Racemate: 31.3; Not reported<br>(+): 300; Not reported<br>(-): 300; Not reported |   |
| Concentration 6 Nom; Meas (µg/L)  | Racemate: 62.5; Not reported<br>(+): 600; 638.00<br>(-): 600; 617.00             |   |
| Concentration 7 Nom; Meas (µg/L)  | Racemate: 125; Not reported  |   |
| Concentration 8 Nom; Meas (µg/L)  | Racemate: 250; Not reported  |   |
| Concentration 9 Nom; Meas (µg/L)  | Racemate: 500; Not   |   |

|                                   | <b>Overmyer et al. 2007</b>  | <b><i>M. mercenaria</i></b>     |
|-----------------------------------|--|---------------------------------|
| <b>Parameter</b>                  | <b>Value</b>   | <b>Comment</b>                  |
|                                   | reported   |                                 |
| Concentration 10 Nom; Meas (µg/L) | Racemate: 1000; Not reported   |                                 |
| Control                           | All: Negative<br>All: Solvent  |                                 |
| LC <sub>50</sub> (95% CI) (µg/L)  | Racemate: 177.00 (46.00-674.00)<br>(+): 208 (137-318)<br>(-): 187.00 (124.00-281.00) | Method: Trimmed Spearman-Kärber |

Notes:

Solubility (S) of fipronil = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Dilution water (3), Hardness (2), Alkalinity (2), Dissolved oxygen (4), Conductivity (2), pH (3), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100-20 =80

Acceptability: Carrier solvent (4), Organisms randomized (1), Acclimation (1), Dilution water (2), Hardness (2), Alkalinity (2), Dissolved oxygen (6), Temperature variation (3), Conductivity (1), pH (2), Random design (2), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1). Total: 100-29 =71

**Reliability score: mean(80, 71)=75.5**

## Water Toxicity Data Summary

*Oncorhynchus mykiss*

Fipronil-destrifluoromethyl-sulfonate

RPA 104615

Collins MK. (1993b) RPA 104615-Acute toxicity to rainbow trout (*Oncorhynchus mykiss*) under static renewal conditions. Springborn Laboratories, Inc., Wareham, Massachusetts. Laboratory study number 10566.0792.6246.103. Submitted to Rhone-Poulenc Ag Company, Research Triangle, North Carolina. USEPA MRID 43291718.

Relevance

Score: 85

Rating: L

Reliability

Score: 86

Rating: R

Relevance points taken off for: Toxicity value (15). 100-15=85

|  | <b>Collins 1993</b>                               | <b><i>O. mykiss</i></b> |
|--|---|-------------------------|
| <b>Parameter</b>                             | <b>Value</b>                                      | <b>Comment</b>          |
| Test method cited                            | FIFRA Guideline 72-1                              |                         |
| Phylum/subphylum                             | Chordata  |                         |
| Class  | Actinopterygii                                    |                         |
| Order  | Salmoniformes                                     |                         |
| Family                                       | Salmonidae  |                         |
| Genus  | <i>Oncorhynchus</i>                               |                         |
| Species                                      | <i>mykiss</i>                                     |                         |
| Family native to North America?              | Yes   |                         |
| Age/size at start of test/growth phase       | 0.85 g<br>45 mm                                   |                         |
| Source of organisms                          | Mount Lassen Trout Farm,<br>Red Bluff, California |                         |
| Have organisms been exposed to contaminants? | No  |                         |
| Animals acclimated and disease-free?         | 14 d  |                         |
| Animals randomized?                          | Yes   |                         |
| Test vessels randomized?                     | Yes   |                         |
| Test duration                                | 96 h  |                         |
| Data for multiple times?                     | 24, 48, 72, 96 h                                  |                         |
| Effect 1                                     | Survival  |                         |
| Control response 1 (mean)                    | 100 %   |                         |
| Temperature                                  | 12 ± 1 °C   |                         |
| Test type                                    | Static renewal                                    |                         |
| Photoperiod/light intensity                  | 16l:8d/970 lux                                    |                         |
| Dilution water                               | Reconstituted from                                | ASTM 1980               |

|   | <b>Collins 1993</b>       | <b><i>O. mykiss</i></b>  |
|---|---------------------------|--|
| <b>Parameter</b>  | <b>Value</b>              | <b>Comment</b>   |
|   | deionized                 |  |
| pH  | 7.5                       |  |
| Hardness  | 38 mg/L CaCO <sub>3</sub> |  |
| Alkalinity  | 23 mg/L CaCO <sub>3</sub> |  |
| Conductivity  | 110 µmhos/cm              |  |
| Dissolved Oxygen  | 3.6-11.0 mg/L             | 33-102 %   |
| Feeding   | Not fed                   |  |
| Purity of test substance  | 94.7 %                    |  |
| Concentrations measured?  | No                        |  |
| Measured is what % of nominal?  | Not reported              |  |
| Toxicity values calculated based on nominal or measured concentrations? | Nominal                   |  |
| Chemical method documented?   | Not reported              |  |
| Concentration of carrier (if any) in test solutions                     | Not used                  |  |
| Concentration 1 Nom; Meas (µg/L)  | 13,000; Not reported      | 1 reps, 10/rep   |
| Concentration 2 Nom; Meas (µg/L)  | 22,000; Not reported      |  |
| Concentration 3 Nom; Meas (µg/L)  | 36,000; Not reported      |  |
| Concentration 4 Nom; Meas (µg/L)  | 60,000; Not reported      |  |
| Concentration 5 Nom; Meas (µg/L)  | 100,000; Not reported     |  |
| Control   | Negative: 0; 0            |  |
| LC <sub>50</sub> (95% CI) (µg/L)  | 96 h: >100,000            | Method:<br>Empirically<br>estimated  |
| NOEC (µg/L)   | 100,000                   | Table 2 shows<br>mortality at this<br>level but considered<br>incidental/unrelated<br>to treatment |
| % control at NOEC   | 96 h: 100 % survival      |  |

Notes: Dilution water TOC average = 2.2 mg/L.

No mortalities related to treatment exposures were observed.

Solubility value for fipronil-destrifluoromethyl-sulfonate (RPA 104615) not available. Solubility (S) of fipronil parent compound (MB 46030) = 1650.8 µg/L, 2S = 3301.6 µg/L. Exposure concentrations exceed 2S of parent compound (fipronil, MB46030).

Reliability points taken off for:

Documentation: Measured concentrations (3), Statistical significance (2), Significance level (2), Minimum significant difference (2), Point estimates (8). Total: 100- 17=

83

Acceptability: Measured concentrations within 20% nominal (4), Adequate replication (2), Minimum significant difference (1), % control at NOEC (1), Point estimates (3). Total: 100-11=89

**Reliability score: mean(83, 89)=86**

## Water Toxicity Data Summary

*P. clarkii*  
Fipronil  
MB 46030

Overmyer JP, Rouse DR, Avants JK, Garrison AW, DeLorenzo ME, Chung KW, Key PB, Wilson WA and Black MC. (2007) Toxicity of fipronil and its enantiomers to marine and freshwater non-targets. *Journal of Environmental Science and Health Part B*, 42(5), 471-480.

Relevance  
Score: 100  
Rating: R

Reliability  
Score: 73  
Rating: L

Relevance points taken off for: none.

|  | <b>Overmyer et al. 2007</b>  | <b><i>P. clarkii</i></b> |
|--|--|--------------------------|
| <b>Parameter</b>                             | <b>Value</b>   | <b>Comment</b>           |
| Test method cited                            | States that standard methods used                                      |                          |
| Phylum/subphylum                             | Arthropoda   |                          |
| Class  | Malacostraca   |                          |
| Order  | Decapoda   |                          |
| Family                                       | Cambaridae   |                          |
| Genus  | <i>Procambarus</i>   |                          |
| Species                                      | <i>clarkii</i>   |                          |
| Family native to North America?              | Yes  |                          |
| Age/size at start of test/growth phase       | 7.1-10.5 cm  |                          |
| Source of organisms                          | Louisiana State University Agricultural Center, Baton Rouge, Louisiana |                          |
| Have organisms been exposed to contaminants? | No   |                          |
| Animals acclimated and disease-free?         | Yes  |                          |
| Animals randomized?                          | Yes  |                          |
| Test vessels randomized?                     | Not reported   |                          |
| Test duration                                | 96 h   |                          |
| Data for multiple times?                     | No   |                          |
| Effect 1                                     | Survival   |                          |
| Control response 1                           | >90 %  |                          |
| Temperature                                  | 20 °C  |                          |
| Test type                                    | Static   |                          |
| Photoperiod/light intensity                  | 16l:8d   |                          |
| Dilution water                               | Moderately hard water  |                          |

|   | <b>Overmyer et al. 2007</b>   | <b><i>P. clarkii</i></b>                        |
|---|---|---|
| <b>Parameter</b>  | <b>Value</b>  | <b>Comment</b>                                  |
| pH  | Measured but not reported   |   |
| Hardness  | Not reported  |   |
| Alkalinity  | Not reported  |   |
| Conductivity  | Measured but not reported   |   |
| Dissolved Oxygen  | Measured but not reported   | Aerated   |
| Feeding   | Not reported  |   |
| Purity of test substance  | 98 %  |   |
| Concentrations measured?  | Yes   |   |
| Measured is what % of nominal?  | 85-97 %   | Only highest and lowest concentrations measured |
| Toxicity values calculated based on nominal or measured concentrations? | Nominal   |   |
| Chemical method documented?   | GCMS  |   |
| Concentration of carrier (if any) in test solutions                     | Acetone, 0.16 %   |   |
| Concentration 1 Nom; Meas (µg/L)  | Racemate: 12.5; 12.15<br>(+): 12.5; 11.55<br>(-): 12.5; 11.10                               | 6 reps, 5/rep                                   |
| Concentration 2 Nom; Meas (µg/L)  | All: 25; Not reported   |   |
| Concentration 3 Nom; Meas (µg/L)  | All: 50; Not reported   |   |
| Concentration 4 Nom; Meas (µg/L)  | All: 100; Not reported  |   |
| Concentration 5 Nom; Meas (µg/L)  | All: 200; Not reported  |   |
| Concentration 6 Nom; Meas (µg/L)  | Racemate: 400; 371.20<br>(+): 400; 341.80<br>(-): 400; 359.70                               |   |
| Control   | All: Negative<br>All: Solvent   |   |
| LC <sub>50</sub> (95% CI) (µg/L)  | Racemate: 124.89 (87.20-179.24)<br>(+): 81.70 (62.90-106.10)<br>(-): 163.50 (124.37-214.94) | Method: Trimmed Spearman-Kärber                 |

Notes:

Solubility (S) of fipronil = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Dilution water (3), Hardness (2), Alkalinity (2), Dissolved oxygen (4), Conductivity (2), pH (3), Photoperiod (3), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100-23 =77

Acceptability: Measured concentrations within 20% nominal (4), Feeding (3), Acclimation (1), Dilution water (2), Hardness (2), Alkalinity (2), Dissolved oxygen (6), Temperature variation (3), Conductivity (1), pH (2), Random design (2), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1). Total: 100-31 =69

**Reliability score: mean(77, 69)=73**

## Water Toxicity Data Summary

*Procambarus clarkii*

Fipronil sulfide

MB 45950

Schlenk D, Huggett DB, Allgood J, Bennett E, Rimoldi J, Beeler AB, Block D, Holder AW, Hovinga R and Bedient P. (2001) Toxicity of fipronil and its degradation products to *Procambarus* sp.: Field and laboratory studies. *Archives of Environmental Contamination and Toxicology*, 41(3), 325-332.

Relevance

Score: 85

Rating: L

Reliability

Score: 68

Rating: L

Relevance points taken off for: Controls (15). 100-15=85

|  | <b>Schlenk 2001</b>   | <b><i>P. clarkii</i></b> |
|--|---|--------------------------|
| <b>Parameter</b>                             | <b>Value</b>  | <b>Comment</b>           |
| Test method cited                            | APHA (1992) Standard methods for the examination of water and wastewater, 18th ed. American Public Health Association, Washington, DC |                          |
| Phylum/subphylum                             | Arthropoda  |                          |
| Class  | Malacostraca  |                          |
| Order  | Decapoda  |                          |
| Family                                       | Cambaridae  |                          |
| Genus  | <i>Procambarus</i>  |                          |
| Species                                      | <i>clarkii</i>  |                          |
| Family native to North America?              | Yes   |                          |
| Age/size at start of test/growth phase       | 6-9 cm  |                          |
| Source of organisms                          | Quality Crayfish, Morse, Louisiana  |                          |
| Have organisms been exposed to contaminants? | No  |                          |
| Animals acclimated and disease-free?         | Yes   |                          |
| Animals randomized?                          | Not reported  |                          |
| Test vessels randomized?                     | Not reported  |                          |
| Test duration                                | 96 h  |                          |
| Data for multiple times?                     | No  |                          |
| Effect 1                                     | Survival  |                          |

|   | Schlenk 2001                                 | <i>P. clarkii</i>               |
|---|--|---------------------------------|
| Parameter   | Value  | Comment                         |
| Control response 1  | Not reported                                 |                                 |
| Temperature   | 25 °C  |                                 |
| Test type   | Static                                       |                                 |
| Photoperiod/light intensity   | 18l:6d                                       |                                 |
| Dilution water  | Deionized, reconstituted water               |                                 |
| pH  | 8.1  |                                 |
| Hardness  | 135 mg/L CaCO <sub>3</sub>                   |                                 |
| Alkalinity  | 90 mg/L CaCO <sub>3</sub>                    |                                 |
| Conductivity  | Not reported                                 |                                 |
| Dissolved Oxygen  | Not reported                                 |                                 |
| Feeding   | Not reported                                 |                                 |
| Purity of test substance  | 90 %   |                                 |
| Concentrations measured?  | Yes  |                                 |
| Measured is what % of nominal?  | 21 %   |                                 |
| Toxicity values calculated based on nominal or measured concentrations? | Calculated both ways                         |                                 |
| Chemical method documented?   | GC-ECD                                       |                                 |
| Concentration of carrier (if any) in test solutions                     | Not reported                                 |                                 |
| Concentration 1 Nom; Meas (µg/L)  | 25; 5.3                                      | 3 reps, 5/rep                   |
| Concentration 2 Nom; Meas (µg/L)  | 50; 10.5                                     |                                 |
| Concentration 3 Nom; Meas (µg/L)  | 100; 21                                      |                                 |
| Concentration 4 Nom; Meas (µg/L)  | 200; 42                                      |                                 |
| Control   | Not reported                                 |                                 |
| LC <sub>50</sub> (95% CI) (µg/L)  | Nominal: 73.7 (11.7)<br>Measured: 15.5 (2.5) | Method: Trimmed Spearman-Kärber |

Notes: Solubility value for fipronil sulfide (MB 45950) not available. Solubility (S) of fipronil parent compound (MB 46030) = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Control type (8), Dissolved oxygen (4), Statistical significance (2), Significance level (2), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100- 20=80

Acceptability: Appropriate control (6), Control response (9), Measured concentrations within 20% nominal (4), Carrier solvent (4), Organisms randomized (1), Feeding (3), Dissolved oxygen (6), Temperature variation (3), Number of concentrations (3), Random design (2), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1). Total: 100-41 =59

**Reliability score: mean(80, 56)=68**

## Water Toxicity Data Summary

*P. clarkia*  
Fipronil  
MB 46030

Schlenk D, Huggett DB, Allgood J, Bennett E, Rimoldi J, Beeler AB, Block D, Holder AW, Hovinga R and Bedient P. (2001) Toxicity of fipronil and its degradation products to *Procambarus* sp.: Field and laboratory studies. *Archives of Environmental Contamination and Toxicology*, 41(3), 325-332.

Relevance  
Score: 85  
Rating: L

Reliability  
Score: 69.5  
Rating: L

Relevance points taken off for: Controls (15). 100-15=85

|  | <b>Schlenk 2001</b>   | <b><i>P. clarkii</i></b> |
|--|---|--------------------------|
| <b>Parameter</b>                             | <b>Value</b>  | <b>Comment</b>           |
| Test method cited                            | APHA (1992) Standard methods for the examination of water and wastewater, 18th ed. American Public Health Association, Washington, DC |                          |
| Phylum/subphylum                             | Arthropoda  |                          |
| Class  | Malacostraca  |                          |
| Order  | Decapoda  |                          |
| Family                                       | Cambaridae  |                          |
| Genus  | <i>Procambarus</i>  |                          |
| Species                                      | <i>Clarkia</i>  |                          |
| Family native to North America?              | Yes   |                          |
| Age/size at start of test/growth phase       | 6-9 cm  |                          |
| Source of organisms                          | Quality Crayfish, Morse, Louisiana  |                          |
| Have organisms been exposed to contaminants? | No  |                          |
| Animals acclimated and disease-free?         | Yes   |                          |
| Animals randomized?                          | Not reported  |                          |
| Test vessels randomized?                     | Not reported  |                          |
| Test duration                                | 96 h  |                          |
| Data for multiple times?                     | No  |                          |
| Effect 1                                     | Survival  |                          |

|   | Schlenk 2001                                 | <i>P. clarkii</i>               |
|---|--|---------------------------------|
| Parameter   | Value  | Comment                         |
| Control response 1  | Not reported                                 |                                 |
| Temperature   | 25 °C  |                                 |
| Test type   | Static                                       |                                 |
| Photoperiod/light intensity   | 18l:6d                                       |                                 |
| Dilution water  | Deionized, reconstituted water               |                                 |
| pH  | 8.1  |                                 |
| Hardness  | 135 mg/L CaCO <sub>3</sub>                   |                                 |
| Alkalinity  | 90 mg/L CaCO <sub>3</sub>                    |                                 |
| Conductivity  | Not reported                                 |                                 |
| Dissolved Oxygen  | Not reported                                 |                                 |
| Feeding   | Not reported                                 |                                 |
| Purity of test substance  | 98 %   |                                 |
| Concentrations measured?  | Yes  |                                 |
| Measured is what % of nominal?  | 30 %   |                                 |
| Toxicity values calculated based on nominal or measured concentrations? | Calculated both ways                         |                                 |
| Chemical method documented?   | GC-ECD                                       |                                 |
| Concentration of carrier (if any) in test solutions                     | Not reported                                 |                                 |
| Concentration 1 Nom; Meas (µg/L)  | 1; 0.3                                       | 3 reps, 5/rep                   |
| Concentration 2 Nom; Meas (µg/L)  | 25; 7.5                                      |                                 |
| Concentration 3 Nom; Meas (µg/L)  | 50; 15                                       |                                 |
| Concentration 4 Nom; Meas (µg/L)  | 100; 30                                      |                                 |
| Concentration 5 Nom; Meas (µg/L)  | 500; 150                                     |                                 |
| Control   | Not reported                                 |                                 |
| LC <sub>50</sub> (95% CI) (µg/L)  | Nominal: 63.7 (22.4)<br>Measured: 14.3 (9.1) | Method: Trimmed Spearman-Kärber |

Notes:

Solubility (S) of fipronil = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Control type (8), Dissolved oxygen (4), Statistical significance (2), Significance level (2), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100- 20=80

Acceptability: Appropriate control (6), Control response (9), Measured concentrations within 20% nominal (4), Carrier solvent (4), Organisms randomized (1), Feeding (3), Dissolved oxygen (6), Temperature variation (3), Random design (2), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1). Total: 100-41 =59

**Reliability score: mean(80, 59)=69.5**

## Water Toxicity Data Summary

*Procambarus clarkii*

Fipronil sulfone

MB 46136

Schlenk D, Huggett DB, Allgood J, Bennett E, Rimoldi J, Beeler AB, Block D, Holder AW, Hovinga R and Bedient P. (2001) Toxicity of fipronil and its degradation products to *Procambarus* sp.: Field and laboratory studies. *Archives of Environmental Contamination and Toxicology*, 41(3), 325-332.

Relevance

Score: 85

Rating: L

Reliability

Score: 68

Rating: L

Relevance points taken off for: Controls (15). 100-15=85

|  | <b>Schlenk 2001</b>   | <b><i>P. clarkii</i></b> |
|--|---|--------------------------|
| <b>Parameter</b>                             | <b>Value</b>  | <b>Comment</b>           |
| Test method cited                            | APHA (1992) Standard methods for the examination of water and wastewater, 18th ed. American Public Health Association, Washington, DC |                          |
| Phylum/subphylum                             | Arthropoda  |                          |
| Class  | Malacostraca  |                          |
| Order  | Decapoda  |                          |
| Family                                       | Cambaridae  |                          |
| Genus  | <i>Procambarus</i>  |                          |
| Species                                      | <i>Clarkia</i>  |                          |
| Family native to North America?              | Yes   |                          |
| Age/size at start of test/growth phase       | 6-9 cm  |                          |
| Source of organisms                          | Quality Crayfish, Morse, Louisiana  |                          |
| Have organisms been exposed to contaminants? | No  |                          |
| Animals acclimated and disease-free?         | Yes   |                          |
| Animals randomized?                          | Not reported  |                          |
| Test vessels randomized?                     | Not reported  |                          |
| Test duration                                | 96 h  |                          |
| Data for multiple times?                     | No  |                          |
| Effect 1                                     | Survival  |                          |

|   | Schlenk 2001                                 | <i>P. clarkii</i>               |
|---|--|---------------------------------|
| Parameter   | Value  | Comment                         |
| Control response 1  | Not reported                                 |                                 |
| Temperature   | 25 °C  |                                 |
| Test type   | Static                                       |                                 |
| Photoperiod/light intensity   | 18l:6d                                       |                                 |
| Dilution water  | Deionized, reconstituted water               |                                 |
| pH  | 8.1  |                                 |
| Hardness  | 135 mg/L CaCO <sub>3</sub>                   |                                 |
| Alkalinity  | 90 mg/L CaCO <sub>3</sub>                    |                                 |
| Conductivity  | Not reported                                 |                                 |
| Dissolved Oxygen  | Not reported                                 |                                 |
| Feeding   | Not reported                                 |                                 |
| Purity of test substance  | 99.5 %                                       |                                 |
| Concentrations measured?  | Yes  |                                 |
| Measured is what % of nominal?  | 24 %   |                                 |
| Toxicity values calculated based on nominal or measured concentrations? | Calculated both ways                         |                                 |
| Chemical method documented?   | GC-ECD                                       |                                 |
| Concentration of carrier (if any) in test solutions                     | Not reported                                 |                                 |
| Concentration 1 Nom; Meas (µg/L)  | 25; 6  | 3 reps, 5/rep                   |
| Concentration 2 Nom; Meas (µg/L)  | 50; 12                                       |                                 |
| Concentration 3 Nom; Meas (µg/L)  | 100; 24                                      |                                 |
| Concentration 4 Nom; Meas (µg/L)  | 200; 48                                      |                                 |
| Control   | Not reported                                 |                                 |
| LC <sub>50</sub> (95% CI) (µg/L)  | Nominal: 34.0 (10.8)<br>Measured: 11.2 (2.0) | Method: Trimmed Spearman-Kärber |

Notes:

Solubility (S) value for fipronil sulfone (MB 46136) = 160 µg/L, 2S = 320 µg/L.

Reliability points taken off for:

Documentation: Control type (8), Dissolved oxygen (4), Statistical significance (2), Significance level (2), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100- 20=80

Acceptability: Appropriate control (6), Control response (9), Measured concentrations within 20% nominal (4), Carrier solvent (4), Organisms randomized (1), Feeding (3), Dissolved oxygen (6), Temperature variation (3), Number of concentrations (3), Random design (2), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1). Total: 100-41 =59

**Reliability score: mean(80, 56)=68**

## Water Toxicity Data Summary

*Procambarus clarkii*  
Fipronil desulfinyl  
MB 46513

Schlenk D, Huggett DB, Allgood J, Bennett E, Rimoldi J, Beeler AB, Block D, Holder AW, Hovinga R and Bedient P. (2001) Toxicity of fipronil and its degradation products to *Procambarus* sp.: Field and laboratory studies. *Archives of Environmental Contamination and Toxicology*, 41(3), 325-332.

Relevance  
Score: 85  
Rating: L

Reliability  
Score: 68  
Rating: L

Relevance points taken off for: Controls (15). 100-15=85

|  | <b>Schlenk 2001</b>   | <i>P. clarkii</i> |
|--|---|-------------------|
| <b>Parameter</b>                             | <b>Value</b>  | <b>Comment</b>    |
| Test method cited                            | APHA (1992) Standard methods for the examination of water and wastewater, 18th ed. American Public Health Association, Washington, DC |                   |
| Phylum/subphylum                             | Arthropoda  |                   |
| Class  | Malacostraca  |                   |
| Order  | Decapoda  |                   |
| Family                                       | Cambaridae  |                   |
| Genus  | <i>Procambarus</i>  |                   |
| Species                                      | <i>Clarkia</i>  |                   |
| Family native to North America?              | Yes   |                   |
| Age/size at start of test/growth phase       | 6-9 cm  |                   |
| Source of organisms                          | Quality Crayfish, Morse, Louisiana  |                   |
| Have organisms been exposed to contaminants? | No  |                   |
| Animals acclimated and disease-free?         | Yes   |                   |
| Animals randomized?                          | Not reported  |                   |
| Test vessels randomized?                     | Not reported  |                   |
| Test duration                                | 96 h  |                   |
| Data for multiple times?                     | No  |                   |
| Effect 1                                     | Survival  |                   |

|   | Schlenk 2001                                   | <i>P. clarkii</i>               |
|---|--|---------------------------------|
| Parameter   | Value  | Comment                         |
| Control response 1  | Not reported                                   |                                 |
| Temperature   | 25 °C  |                                 |
| Test type   | Static   |                                 |
| Photoperiod/light intensity   | 18l:6d   |                                 |
| Dilution water  | Deionized, reconstituted water                 |                                 |
| pH  | 8.1  |                                 |
| Hardness  | 135 mg/L CaCO <sub>3</sub>                     |                                 |
| Alkalinity  | 90 mg/L CaCO <sub>3</sub>                      |                                 |
| Conductivity  | Not reported                                   |                                 |
| Dissolved Oxygen  | Not reported                                   |                                 |
| Feeding   | Not reported                                   |                                 |
| Purity of test substance  | 98.5 %   |                                 |
| Concentrations measured?  | Yes  |                                 |
| Measured is what % of nominal?  | 46 %   |                                 |
| Toxicity values calculated based on nominal or measured concentrations? | Calculated both ways                           |                                 |
| Chemical method documented?   | GC-ECD   |                                 |
| Concentration of carrier (if any) in test solutions                     | Not reported                                   |                                 |
| Concentration 1 Nom; Meas (µg/L)  | 25; 11.5                                       | 3 reps, 5/rep                   |
| Concentration 2 Nom; Meas (µg/L)  | 50; 23   |                                 |
| Concentration 3 Nom; Meas (µg/L)  | 100; 46  |                                 |
| Concentration 4 Nom; Meas (µg/L)  | 200; 92  |                                 |
| Control   | Not reported                                   |                                 |
| LC <sub>50</sub> (95% CI) (µg/L)  | Nominal: 149.7 (20.5)<br>Measured: 68.6 (26.6) | Method: Trimmed Spearman-Kärber |

Notes:

Solubility (S) value for fipronil desulfinyl (MB 46513) = 950 µg/L, 2S = 1900 µg/L.

Reliability points taken off for:

Documentation: Control type (8), Dissolved oxygen (4), Statistical significance (2), Significance level (2), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100- 20=80

Acceptability: Appropriate control (6), Control response (9), Measured concentrations within 20% nominal (4), Carrier solvent (4), Organisms randomized (1), Feeding (3), Dissolved oxygen (6), Temperature variation (3), Number of concentrations (3), Random design (2), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1). Total: 100-41 =59

**Reliability score: mean(80, 56)=68**

## Water Toxicity Data Summary

*Pimephales promelas*

Fipronil

MB 46030

Beggel S, Werner I, Connon RE and Geist JP. (2010) Sublethal toxicity of commercial insecticide formulations and their active ingredients to larval fathead minnow (*Pimephales promelas*). *Science of the total environment*, 408(16), 3169-3175.

Relevance

Score: 82.5

Rating: L

Reliability

Score: 85.5

Rating: R

Relevance points taken off for: Standard method (10), Control response (7.5). 100-17.5 = 82.5

| <b>Parameter</b>                             | <b>Beggel 2010 Value</b>           | <b><i>P. promelas</i> Comment</b> |
|--|------------------------------------|-----------------------------------|
| Test method cited                            | Not reported                       |                                   |
| Phylum/subphylum                             | Chordata                           |                                   |
| Class  | Actinopterygii                     |                                   |
| Order  | Cypriniformes                      |                                   |
| Family                                       | Cyprinidae                         |                                   |
| Genus  | <i>Pimephales</i>                  |                                   |
| Species                                      | <i>promelas</i>                    |                                   |
| Family native to North America?              | Yes                                |                                   |
| Age/size at start of test/growth phase       | Larvae, 7 d post-hatch             |                                   |
| Source of organisms                          | Aquatox Inc, Hot Springs, Arkansas |                                   |
| Have organisms been exposed to contaminants? | No                                 |                                   |
| Animals acclimated and disease-free?         | Yes                                |                                   |
| Animals randomized?                          | Not reported                       |                                   |
| Test vessels randomized?                     | Yes                                |                                   |
| Test duration                                | 24 h                               |                                   |
| Data for multiple times?                     | No                                 |                                   |
| Effect 1                                     | Survival                           |                                   |
| Control response 1                           | Not reported                       |                                   |
| Temperature                                  | 23 ± 0.3 °C                        |                                   |
| Test type                                    | Static                             |                                   |
| Photoperiod/light intensity                  | 16l:8d                             |                                   |
| Dilution water                               | Reconstituted deionized water      |                                   |
| pH   | 7.51                               |                                   |

|   | <b>Beggel 2010</b>                   | <b><i>P. promelas</i></b>   |
|---|--------------------------------------|---|
| <b>Parameter</b>  | <b>Value</b>                         | <b>Comment</b>  |
| Hardness  | 80-100 mg/L CaCO <sub>3</sub>        |   |
| Alkalinity  | 57-64 mg/L CaCO <sub>3</sub>         |   |
| Conductivity  | 278 µmhos/cm                         |   |
| Dissolved Oxygen  | 7.2 mg/L                             | 87 %  |
| Feeding   | Not fed                              |   |
| Purity of test substance  | 98.5 %                               |   |
| Concentrations measured?  | Yes                                  |   |
| Measured is what % of nominal?  | Not reported                         |   |
| Toxicity values calculated based on nominal or measured concentrations? | Not reported                         |   |
| Chemical method documented?   | External testing                     | California Department of Fish and Game Water Pollution Laboratory, Rancho Cordova, California |
| Concentration of carrier (if any) in test solutions                     | 1 mL/L, methanol                     |   |
| Concentration 1 Nom; Meas (µg/L)  | 150; Not reported                    | 13 reps, 10/rep (9 reps used for swim performance, 4 used for growth)                         |
| Concentration 2 Nom; Meas (µg/L)  | 200; Not reported                    |   |
| Concentration 3 Nom; Meas (µg/L)  | 250; Not reported                    |   |
| Concentration 4 Nom; Meas (µg/L)  | 300; Not reported                    |   |
| Concentration 5 Nom; Meas (µg/L)  | 350; Not reported                    |   |
| Concentration 6 Nom; Meas (µg/L)  | 400; Not reported                    |   |
| Control   | Negative: 0; 0<br>Solvent: 0; 0      |   |
| LC <sub>50</sub> (95% CI) (µg/L)  | 398.29 (376.27-438.79)               | Method: Probit  |
| NOEC  | 300                                  | Method: ANOVA<br>p: 0.05<br>MSD: Not reported   |
| LOEC  | 350                                  |   |
| MATC (GeoMean NOEC, LOEC)   | 324                                  |   |
| % control at NOEC   | Not calculable, no raw data provided |   |
| % control at LOEC   | Not calculable, no raw data provided |   |

Notes:

Solubility (S) of fipronil = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Measured concentrations (3), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100-7 =93

Acceptability: Standard method (5), Control response (9), Measured concentrations within 20% nominal (4), Organisms randomized (1), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1). Total: 100-22 =78

**Reliability score: mean(93, 78)=85.5**

## Water Toxicity Data Summary

*Palaemonetes pugio*

Fipronil

MB 46030

Overmyer JP, Rouse DR, Avants JK, Garrison AW, DeLorenzo ME, Chung KW, Key PB, Wilson WA and Black MC. (2007) Toxicity of fipronil and its enantiomers to marine and freshwater non-targets. *Journal of Environmental Science and Health Part B*, 42(5), 471-480.

Relevance

Score: 85

Rating: L

Reliability

Score: 69

Rating: L

Relevance points taken off for: Freshwater (15). 100-15=85

|  | <b>Overmyer et al. 2007</b>   | <b><i>P. pugio</i></b> |
|--|---|------------------------|
| <b>Parameter</b>                             | <b>Value</b>  | <b>Comment</b>         |
| Test method cited                            | Stated used standard methods  |                        |
| Phylum/subphylum                             | Arthropoda/crustacea  |                        |
| Class  | Malacostraca  |                        |
| Order  | Decapoda/caridea  |                        |
| Family                                       | Palaemonidae  |                        |
| Genus  | <i>Palaemonetes</i>   |                        |
| Species                                      | <i>pugio</i>  |                        |
| Family native to North America?              | Yes   |                        |
| Age/size at start of test/growth phase       | Adults<br>Larvae, 1-2 d   |                        |
| Source of organisms                          | Adults collected from Leadenwah Creek, 32°38.930'N, 80°13.340'W), a relatively uncontaminated tidal tributary of the North Edisto River estuary, South Carolina |                        |
| Have organisms been exposed to contaminants? | No  |                        |
| Animals acclimated and disease-free?         | 7-14 d  |                        |
| Animals randomized?                          | Not reported  |                        |
| Test vessels randomized?                     | Not reported  |                        |
| Test duration                                | 96 h  | Renewed every 24 h     |
| Data for multiple times?                     | No  |                        |

|   | <b>Overmyer et al. 2007</b>  | <b><i>P. pugio</i></b>                           |
|---|--|--|
| <b>Parameter</b>  | <b>Value</b>   | <b>Comment</b>                                   |
| Effect 1  | Survival   |  |
| Control response 1  | >90 %  |  |
| Temperature   | 25 °C  |  |
| Test type   | Static   |  |
| Photoperiod/light intensity   | 16l:8d   |  |
| Dilution water  | Seawater   | 20‰ salinity                                     |
| pH  | Measured but not reported  |  |
| Hardness  | Not reported   |  |
| Alkalinity  | Not reported   |  |
| Conductivity  | Measured but not reported  |  |
| Dissolved Oxygen  | Measured but not reported  |  |
| Feeding   | Adults: not fed<br>Larvae: <i>Artemia</i> daily  |  |
| Purity of test substance  | 98 %   |  |
| Concentrations measured?  | Yes  |  |
| Measured is what % of nominal?  | Not calculable   | Inconsistent values reported in study            |
| Toxicity values calculated based on nominal or measured concentrations? | Nominal  |  |
| Chemical method documented?   | GCMS   |  |
| Concentration of carrier (if any) in test solutions                     | Acetone, concentration not reported  |  |
| Concentration 1 Nom; Meas (µg/L)  | Racemate: 0.125; Not reported*<br>(+): 0.125; Not reported*<br>(-): 0.125; Not reported*   | Adults: 3 reps, 10/rep<br>Larvae: 3 reps, 10/rep |
| Concentration 2 Nom; Meas (µg/L)  | All: 0.25; Not reported  |  |
| Concentration 3 Nom; Meas (µg/L)  | All: 0.5; Not reported   |  |
| Concentration 4 Nom; Meas (µg/L)  | All: 1.0; Not reported   |  |
| Concentration 6 Nom; Meas (µg/L)  | Racemate: 2.0; Not reported*<br>(+): 2.0; Not reported*<br>(-): 2.0; Not reported*   |  |
| Control   | All: Negative<br>All: Solvent  |  |
| LC <sub>50</sub> (95% CI) (µg/L)  | Adults:<br>Racemate: 0.32 (0.24-0.41)<br>(+): 0.54 (0.45-0.64)<br>(-): 0.32 (0.22-0.48)<br><br>Larvae:<br>Racemate: 0.68 (0.57-0.80) | Method: Trimmed Spearman-Kärber                  |

|                  | <b>Overmyer et al. 2007</b>                         | <b><i>P. pugio</i></b> |
|------------------|---|------------------------|
| <b>Parameter</b> | <b>Value</b>  | <b>Comment</b>         |
|                  | (+): 208.0 (137.00-318.00)<br>(-): 0.35 (0.29-0.43) |                        |

Notes: \*Inconsistent measured concentration values reported in study.

Solubility (S) of fipronil = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Measured concentrations (3), Dilution water (3), Hardness (2), Alkalinity (2), Dissolved oxygen (4), Conductivity (2), pH (3), Photoperiod (3), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100-26 =74

Acceptability: Measured concentrations within 20% nominal (4), Carrier solvent (4), No previous exposure (4), Organisms randomized (1), Acclimation (1), Dilution water (2), Hardness (2), Alkalinity (2), Dissolved oxygen (6), Temperature variation (3), Conductivity (1), pH (2), Random design (2), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1). Total: 100-36 =64

**Reliability score: mean(74, 64)=69**

## Water Toxicity Data Summary

*Procambarus zonangulus*

Fipronil

MB 46030

Schlenk D, Huggett DB, Allgood J, Bennett E, Rimoldi J, Beeler AB, Block D, Holder AW, Hovinga R and Bedient P. (2001) Toxicity of fipronil and its degradation products to *Procambarus* sp.: Field and laboratory studies. *Archives of Environmental Contamination and Toxicology*, 41(3), 325-332.

Relevance

Score: 85

Rating: L

Reliability

Score: 68

Rating: L

Relevance points taken off for: Controls (15). 100-15=85

|  | <b>Schlenk 2001</b>   | <b><i>P. zonangulus</i></b> |
|--|---|-----------------------------|
| <b>Parameter</b>                             | <b>Value</b>  | <b>Comment</b>              |
| Test method cited                            | APHA (1992) Standard methods for the examination of water and wastewater, 18th ed. American Public Health Association, Washington, DC |                             |
| Phylum/subphylum                             | Arthropoda  |                             |
| Class  | Malacostraca  |                             |
| Order  | Decapoda  |                             |
| Family                                       | Cambaridae  |                             |
| Genus  | <i>Procambarus</i>  |                             |
| Species                                      | <i>zonangulus</i>   |                             |
| Family native to North America?              | Yes   |                             |
| Age/size at start of test/growth phase       | 6-9 cm  |                             |
| Source of organisms                          | Quality Crayfish, Morse, Louisiana  |                             |
| Have organisms been exposed to contaminants? | No  |                             |
| Animals acclimated and disease-free?         | Yes   |                             |
| Animals randomized?                          | Not reported  |                             |
| Test vessels randomized?                     | Not reported  |                             |
| Test duration                                | 96 h  |                             |
| Data for multiple times?                     | No  |                             |
| Effect 1                                     | Survival  |                             |

|   | Schlenk 2001                                 | <i>P. zonangulus</i>            |
|---|--|---------------------------------|
| Parameter   | Value  | Comment                         |
| Control response 1  | Not reported                                 |                                 |
| Temperature   | 25 °C  |                                 |
| Test type   | Static                                       |                                 |
| Photoperiod/light intensity   | 18l:6d                                       |                                 |
| Dilution water  | Deionized, reconstituted water               |                                 |
| pH  | 8.1  |                                 |
| Hardness  | 135 mg/L CaCO <sub>3</sub>                   |                                 |
| Alkalinity  | 90 mg/L CaCO <sub>3</sub>                    |                                 |
| Conductivity  | Not reported                                 |                                 |
| Dissolved Oxygen  | Not reported                                 |                                 |
| Feeding   | Not reported                                 |                                 |
| Purity of test substance  | 98 %   |                                 |
| Concentrations measured?  | Yes  |                                 |
| Measured is what % of nominal?  | 30 %   |                                 |
| Toxicity values calculated based on nominal or measured concentrations? | Calculated both ways                         |                                 |
| Chemical method documented?   | GC-ECD                                       |                                 |
| Concentration of carrier (if any) in test solutions                     | Not reported                                 |                                 |
| Concentration 1 Nom; Meas (µg/L)  | 1; 0.3                                       | 3 reps, 5/rep                   |
| Concentration 2 Nom; Meas (µg/L)  | 25; 7.5                                      |                                 |
| Concentration 3 Nom; Meas (µg/L)  | 50; 15                                       |                                 |
| Concentration 4 Nom; Meas (µg/L)  | 100; 30                                      |                                 |
| Concentration 5 Nom; Meas (µg/L)  | 500; 150                                     |                                 |
| Control   | Not reported                                 |                                 |
| LC <sub>50</sub> (95% CI) (µg/L)  | Nominal: 65.1 (28.1)<br>Measured: 19.5 (8.4) | Method: Trimmed Spearman-Kärber |

Notes:

Solubility (S) of fipronil = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Control type (8), Dissolved oxygen (4), Statistical significance (2), Significance level (2), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100- 20=80

Acceptability: Appropriate control (6), Control response (9), Measured concentrations within 20% nominal (4), Carrier solvent (4), Organisms randomized (1), Feeding (3), Dissolved oxygen (6), Temperature variation (3), Number of concentrations (3), Random design (2), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1). Total: 100-41 =59

**Reliability score: mean(80, 56)=68**

## Water Toxicity Data Summary

*Scenedesmus obliquus*

Fipronil

MB 46030

Qu H, Ma RX, Liu DH, Wang P, Huang LD, Qiu XX and Zhou ZQ. (2014) Enantioselective toxicity and degradation of the chiral insecticide fipronil in *Scenedesmus obliquus* suspension system. *Environmental toxicology and chemistry*, 33(11), 2516-2521.

Relevance

Score: 85

Rating: L

Reliability

Score: 76.5

Rating: R

Relevance points taken off for: Controls (15). 100-15=85

|  | <b>Qu 2014</b>   | <b><i>S. obliquus</i></b>  |
|--|--|--|
| <b>Parameter</b>                             | <b>Value</b>   | <b>Comment</b>   |
| Test method cited                            | OECD guideline 201                                     |  |
| Phylum/subphylum                             | Chlorophyta  |  |
| Class  | Chlorophyceae  |  |
| Order  | Sphaeropleales   |  |
| Family                                       | Scenedesmaceae   |  |
| Genus  | <i>Scenedesmus</i>                                     |  |
| Species                                      | <i>obliquus</i>  |  |
| Family native to North America?              | Yes  |  |
| Age/size at start of test/growth phase       | Log growth phase                                       |  |
| Source of organisms                          | Institute of Hydrobiology, Chinese Academy of Sciences |  |
| Have organisms been exposed to contaminants? | No   |  |
| Animals acclimated and disease-free?         | Yes  |  |
| Animals randomized?                          | Not reported   | Given organism size and presence in growth medium, it is assumed that aliquots are inherently randomly |
| Test vessels randomized?                     | Not reported   |  |
| Test duration                                | 72 h   |  |
| Data for multiple times?                     | No   |  |
| Effect 1                                     | Cell count   |  |
| Control response 1                           | Not reported   |  |

|   | <b>Qu 2014</b>  | <i>S. obliquus</i>        |
|---|---|---------------------------|
| <b>Parameter</b>  | <b>Value</b>  | <b>Comment</b>            |
| Temperature   | 25 ± 0.5 °C   |                           |
| Test type   | Static  |                           |
| Photoperiod/light intensity   | Continuous/3000-4000 lux  |                           |
| Dilution water  | BG11 algal growth medium  |                           |
| Feeding   | Growth medium   |                           |
| Purity of test substance  | Racemate: 96.5 %<br>(+): 99.5 %<br>(-): 99.4 %  |                           |
| Concentrations measured?  | Not reported  |                           |
| Measured is what % of nominal?  | Not calculable  |                           |
| Toxicity values calculated based on nominal or measured concentrations? | Nominal   |                           |
| Chemical method documented?   | Not reported  |                           |
| Concentration of carrier (if any) in test solutions                     | Not reported  |                           |
| Concentration 1 Nom (µg/L)  | Exact concentrations not reported but nominal ranges reported:<br><br>Racemate: 10-1500<br>(+): 10-3000<br>(-): 10-1500 | 3 reps, 5000 cells/mL/rep |
| Control   | Not reported  |                           |
| EC <sub>50</sub> (95% CI) (µg/L)  | Racemate: 540 (270-1120)<br>(+): 1500 (810-2240)<br>(-): 290 (220-370)  | Method: Not reported      |

Notes: Reliability points were not taken off for water quality parameters (hardness, alkalinity, conductivity) because there is no guidance for these parameters in the test guidelines for algal/plant studies, the growth medium used requires ultrapure water, and the medium is presumably appropriate for the test species because a specific culture media was used.

Solubility (S) of fipronil = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Analytical method (4), Nominal concentrations (3), Measured concentrations (3), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100-14 =86

Acceptability: Appropriate control (6), Control response (9), Measured concentrations within 20% nominal (4), Carrier solvent (4), Random design (2), Dilution factor (2), Statistical method (2), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1). Total: 100-32 =67

**Reliability score: mean(86, 67)=76.5**

## Water Toxicity Data Summary

*Simulium vittatum*

Fipronil

MB 46030

Overmyer JP, Rouse DR, Avants JK, Garrison AW, DeLorenzo ME, Chung KW, Key PB, Wilson WA and Black MC. (2007) Toxicity of fipronil and its enantiomers to marine and freshwater non-targets. *Journal of Environmental Science and Health Part B*, 42(5), 471-480.

Relevance

Score: 100

Rating: R

Reliability

Score: 70.5

Rating: L

Relevance points taken off for: none.

|  | <b>Overmyer et al. 2007</b>            | <b><i>S. vittatum</i></b> |
|--|--|---------------------------|
| <b>Parameter</b>                             | <b>Value</b>                           | <b>Comment</b>            |
| Test method cited                            | States that standard methods used      |                           |
| Phylum/subphylum                             | Arthropoda                             |                           |
| Class  | Insecta                                |                           |
| Order  | Diptera                                |                           |
| Family                                       | Simuliidae                             |                           |
| Genus  | <i>Simulium</i>                        |                           |
| Species                                      | <i>vittatum</i>                        |                           |
| Family native to North America?              | Yes                                    |                           |
| Age/size at start of test/growth phase       | 5 <sup>th</sup> instar larvae          |                           |
| Source of organisms                          | University of Georgia, Athens, Georgia |                           |
| Have organisms been exposed to contaminants? | No                                     |                           |
| Animals acclimated and disease-free?         | Yes                                    |                           |
| Animals randomized?                          | Not reported                           |                           |
| Test vessels randomized?                     | Not reported                           |                           |
| Test duration                                | 48 h                                   |                           |
| Data for multiple times?                     | No                                     |                           |
| Effect 1                                     | Survival                               |                           |
| Control response 1                           | >90 %                                  |                           |
| Temperature                                  | 20 °C                                  |                           |
| Test type                                    | Static                                 |                           |
| Photoperiod/light intensity                  | 16l:8d                                 |                           |
| Dilution water                               | Moderately hard water                  |                           |
| pH   | Measured but not reported              |                           |

|   | <b>Overmyer et al. 2007</b>  | <i>S. vittatum</i>                              |
|---|--|---|
| <b>Parameter</b>  | <b>Value</b>   | <b>Comment</b>                                  |
| Hardness  | Not reported   |   |
| Alkalinity  | Not reported   |   |
| Conductivity  | Measured but not reported  |   |
| Dissolved Oxygen  | Measured but not reported  |   |
| Feeding   | Not reported   |   |
| Purity of test substance  | 98 %   |   |
| Concentrations measured?  | Yes  |   |
| Measured is what % of nominal?  | Racemate: 81-125 %   | Only highest and lowest concentrations measured |
| Toxicity values calculated based on nominal or measured concentrations? | Adjusted measured based on deviation   |   |
| Chemical method documented?   | GCMS   |   |
| Concentration of carrier (if any) in test solutions                     | Acetone, concentration not reported  |   |
| Concentration 1 Nom; Meas (µg/L)  | Racemate: 0.06; 0.06<br>(+): 0.06; 0.07<br>(-): 0.06; 0.07                   | 5 reps, 15/rep                                  |
| Concentration 2 Nom; Meas (µg/L)  | All: 0.125; Not reported   |   |
| Concentration 3 Nom; Meas (µg/L)  | All: 0.25; Not reported  |   |
| Concentration 4 Nom; Meas (µg/L)  | All: 0.5; Not reported   |   |
| Concentration 5 Nom; Meas (µg/L)  | All: 1.0; Not reported   |   |
| Concentration 6 Nom; Meas (µg/L)  | Racemate: 2.0; 1.62<br>(+): 2.00; 2.50<br>(-): 2.00; 2.49                    |   |
| Control   | All: Negative<br>All: Solvent  |   |
| LC <sub>50</sub> (95% CI) (µg/L)  | Racemate: 0.65 (0.60-0.70)<br>(+): 0.72 (0.66-0.78)<br>(-): 0.74 (0.69-0.81) | Method: Trimmed Spearman-Kärber                 |

Notes:

Solubility (S) of fipronil = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Dilution water (3), Hardness (2), Alkalinity (2), Dissolved oxygen (4), Conductivity (2), pH (3), Photoperiod (3), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100-23 = 77

Acceptability: Measured concentrations within 20% nominal (4), Carrier solvent (4), Organisms randomized (1), Feeding (3), Acclimation (1), Dilution water (2), Hardness (2), Alkalinity (2),

Dissolved oxygen (6), Temperature variation (3), Conductivity (1), pH (2), Random design (2), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1). Total: 100-36 =64

**Reliability score: mean(77, 64)=70.5**

## Water Toxicity Data Summary

*Taenionema* sp.  
Fipronil  
MB46030

Weston DP and Lydy MJ. (2014) Toxicity of the insecticide fipronil and its degradates to benthic macroinvertebrates of urban streams. *Environmental science & technology*, 48(2), 1290-1297.

Relevance  
Score: 75  
Rating: L

Reliability  
Score: 80  
Rating: R

Relevance points taken off for: Standard method (10), Toxicity value (15). 100-25=75

|  | <b>Weston 2014</b>  | <i>E. excrucians</i> |
|--|---|----------------------|
| <b>Parameter</b>                             | <b>Value</b>  | <b>Comment</b>       |
| Test method cited                            | Not reported  |                      |
| Phylum/subphylum                             | Anthropoda  |                      |
| Class  | Insecta   |                      |
| Order  | Plecoptera  |                      |
| Family                                       | Taeniopterygidae  |                      |
| Genus  | <i>Taenionema</i>   |                      |
| Species                                      | sp.   |                      |
| Family native to North America?              | Yes   |                      |
| Age/size at start of test/growth phase       | Not reported  |                      |
| Source of organisms                          | Urban waterbodies with minimal development in Northern California |                      |
| Have organisms been exposed to contaminants? | Not reported  |                      |
| Animals acclimated and disease-free?         | 24 h  |                      |
| Animals randomized?                          | Not reported  |                      |
| Test vessels randomized?                     | Not reported  |                      |
| Test duration                                | 96 h  |                      |
| Data for multiple times?                     | Not reported  |                      |
| Effect 1                                     | Survival  |                      |
| Control response 1                           | 100 %   |                      |
| Effect 2                                     | Immobilization (ability to cling)                                 |                      |
| Control response 2                           | Not reported  |                      |
| Temperature                                  | 8 °C  |                      |
| Test type                                    | Static  |                      |
| Photoperiod/light intensity                  | 16l:8d; Not reported  |                      |

|   | <b>Weston 2014</b>   | <b><i>E. excrucians</i></b>                             |
|---|--|---|
| <b>Parameter</b>  | <b>Value</b>   | <b>Comment</b>  |
| Dilution water  | Milli-Q purified, deionized watermade moderately hard by addition of salts | *According to EPA 821-R-02-012                          |
| pH  | Not reported*  |   |
| Hardness  | Not reported*  |   |
| Alkalinity  | Not reported*  |   |
| Conductivity  | Not reported*  |   |
| Dissolved Oxygen  | Not reported*  |   |
| Feeding   | Not reported   |   |
| Purity of test substance  | 99.50 %  | Not reported but author verified from chemical supplier |
| Concentrations measured?  | Yes  |   |
| Measured is what % of nominal?  | 66-113%  |   |
| Toxicity values calculated based on nominal or measured concentrations? | Measured   |   |
| Chemical method documented?   | GC   |   |
| Concentration of carrier (if any) in test solutions                     | Acetone, <36 µg/L  |   |
| Concentration 1 Nom; Meas (µg/L)  | Not reported; 4-7 concentrations tested; dilution factor of 2              | 3 reps, 4-6/rep   |
| Control   | Negative: 0; 0<br>Solvent: 0; 0  |   |
| LC <sub>50</sub> (95% CI) (µg/L)  | >0.184   | Method: Probit  |
| EC <sub>50</sub> (95% CI) (µg/L)  | >0.184   | Method: Probit  |

Notes: Points were not deducted for lack of water quality parameter reporting because water was prepared according to EPA 821-R-02-012 and measured during study (but not reported).

Solubility (S) of fipronil = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Organism life stage/size (5), Nominal concentrations (3), Measured concentrations (3), Minimum significant difference (2), % control at NOEC/LOEC (2), Point estimates (8). Total: 100-23 =77

Acceptability: Standard method (5), Organisms randomized (1), Temperature variation (3), Random design (2), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1), Point estimates (3). Total: 100-17 =83

**Reliability score: mean(77,83)=80**

## Water Toxicity Data Summary

*Tricorythodes* sp.

Fipronil

MB46030

Weston DP and Lydy MJ. (2014) Toxicity of the insecticide fipronil and its degradates to benthic macroinvertebrates of urban streams. *Environmental science & technology*, 48(2), 1290-1297.

Relevance

Score: 75

Rating: L

Reliability

Score: 85.5

Rating: R

Relevance points taken off for: Standard method (10), Toxicity value (15). 100-25=75

|  | <b>Weston 2014</b>  | <b><i>Tricorythodes</i></b> |
|--|---|-----------------------------|
| <b>Parameter</b>                             | <b>Value</b>  | <b>Comment</b>              |
| Test method cited                            | Not reported  |                             |
| Phylum/subphylum                             | Anthropoda  |                             |
| Class  | Insecta   |                             |
| Order  | Ephemeroptera   |                             |
| Family                                       | Leptohyphidae   |                             |
| Genus  | <i>Tricorythodes</i>  |                             |
| Species                                      | sp.   |                             |
| Family native to North America?              | Yes   |                             |
| Age/size at start of test/growth phase       | Not reported  |                             |
| Source of organisms                          | Urban waterbodies with minimal development in Northern California |                             |
| Have organisms been exposed to contaminants? | Not reported  |                             |
| Animals acclimated and disease-free?         | 24 h  |                             |
| Animals randomized?                          | Not reported  |                             |
| Test vessels randomized?                     | Not reported  |                             |
| Test duration                                | 48 h  |                             |
| Data for multiple times?                     | Not reported  |                             |
| Effect 1                                     | Survival  |                             |
| Control response 1                           | 100 %   |                             |
| Effect 2                                     | Immobilization (ability to cling)                                 |                             |
| Control response 2                           | Not reported  |                             |
| Temperature                                  | 18 °C   |                             |
| Test type                                    | Static  |                             |
| Photoperiod/light intensity                  | 16l:8d; Not reported  |                             |

|   | <b>Weston 2014</b>   | <b><i>Tricorythodes</i></b>                             |
|---|--|---|
| <b>Parameter</b>  | <b>Value</b>   | <b>Comment</b>  |
| Dilution water  | Milli-Q purified, deionized watermade moderately hard by addition of salts | *According to EPA 821-R-02-012                          |
| pH  | Not reported*  |   |
| Hardness  | Not reported*  |   |
| Alkalinity  | Not reported*  |   |
| Conductivity  | Not reported*  |   |
| Dissolved Oxygen  | Not reported*  |   |
| Feeding   | Not reported   |   |
| Purity of test substance  | 99.50 %  | Not reported but author verified from chemical supplier |
| Concentrations measured?  | Yes  |   |
| Measured is what % of nominal?  | 66-113%  |   |
| Toxicity values calculated based on nominal or measured concentrations? | Measured   |   |
| Chemical method documented?   | GC   |   |
| Concentration of carrier (if any) in test solutions                     | Acetone, <36 µg/L  |   |
| Concentration 1 Nom; Meas (µg/L)  | Not reported; 4-7 concentrations tested; dilution factor of 2              | 3 reps, 4-6/rep   |
| Control   | Negative: 0; 0<br>Solvent: 0; 0  |   |
| LC <sub>50</sub> (95% CI) (µg/L)  | >1.229   | Method: Probit  |
| EC <sub>50</sub> (95% CI) (µg/L)  | >1.229   | Method: Probit  |

Notes: Points were not deducted for lack of water quality parameter reporting because water was prepared according to EPA 821-R-02-012 and measured during study (but not reported).

Solubility (S) of fipronil = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Organism life stage/size (5), Nominal concentrations (3), Measured concentrations (3), Minimum significant difference (2), % control at NOEC/LOEC (2), Point estimates (8). Total: 100-23 =77

Acceptability: Standard method (5), Organisms randomized (1), Temperature variation (3), Random design (2), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1), Point estimates (3). Total: 100-17 =83

**Reliability score: mean(77,83)=80**

## Water Toxicity Data Summary

*Villosa constricta*

Fipronil

MB 46030

Bringolf RB, Cope WG, Eads CB, Lazaro PR, Barnhart MC and Shea D. (2007) Acute and chronic toxicity of technical-grade pesticides to glochidia and juveniles of freshwater mussels (unionidae). *Environmental Toxicology and Chemistry*, 26(10), 2086-2093.

Relevance

Score: 85

Rating: L

Reliability

Score: 79

Rating: R

Relevance points taken off for: Point estimates (10). 100-15=85

|  | <b>Bringolf 2007</b>  | <b><i>V. constricta</i></b> |
|--|---|-----------------------------|
| <b>Parameter</b>                             | <b>Value</b>  | <b>Comment</b>              |
| Test method cited                            | ASTM 2006: Standard guide for conducting laboratory toxicity tests with freshwater mussels. E2455-06. |                             |
| Phylum/subphylum                             | Mollusca  |                             |
| Class  | Bivalvia  |                             |
| Order  | Unionoida   |                             |
| Family                                       | Unionidae   |                             |
| Genus  | <i>Villosa</i>  |                             |
| Species                                      | <i>constricta</i>   |                             |
| Family native to North America?              | Yes   |                             |
| Age/size at start of test/growth phase       | Glochidia   |                             |
| Source of organisms                          | Brooding adult females collected from rural Deep Creek, Person County, Missouri                       |                             |
| Have organisms been exposed to contaminants? | Not reported  |                             |
| Animals acclimated and disease-free?         | Yes   |                             |
| Animals randomized?                          | Not reported  |                             |
| Test vessels randomized?                     | Not reported  |                             |
| Test duration                                | 96 h  |                             |
| Data for multiple times?                     | 48, 96 h  |                             |
| Effect 1                                     | Survival  |                             |
| Control response 1                           | >93 %   |                             |

|   | <b>Bringolf 2007</b>                           | <b><i>V. constricta</i></b>     |
|---|--|---------------------------------|
| <b>Parameter</b>  | <b>Value</b>                                   | <b>Comment</b>                  |
| Temperature   | 21 ± 1 °C                                      |                                 |
| Test type   | Static renewal                                 | Renewed at 48 h                 |
| Photoperiod/light intensity   | Not reported                                   |                                 |
| Dilution water  | Reconstituted hard water                       | ASTM 2006                       |
| pH  | 8.32-8.61                                      |                                 |
| Hardness  | 170-192 mg/L CaCO <sub>3</sub>                 |                                 |
| Alkalinity  | 116-130 mg/L CaCO <sub>3</sub>                 |                                 |
| Conductivity  | 523-625 µmhos/cm                               |                                 |
| Dissolved Oxygen  | >80 %  |                                 |
| Feeding   | Not reported                                   |                                 |
| Purity of test substance  | 99.7 %   |                                 |
| Concentrations measured?  | Yes  |                                 |
| Measured is what % of nominal?  | Mean: 64.6 %                                   |                                 |
| Toxicity values calculated based on nominal or measured concentrations? | Measured                                       |                                 |
| Chemical method documented?   | GC   |                                 |
| Concentration of carrier (if any) in test solutions                     | Acetone, concentration not reported            |                                 |
| Concentration 1 Nom; Meas (µg/L)  | 5-6 concentrations tested; values not reported | 3 reps, 150-200 glochidia/rep   |
| Control   | Negative Solvent                               |                                 |
| EC <sub>50</sub> (95% CI) (µg/L)  | Glochidia:<br>24 h: >2000<br>48 h: >2000       | Method: Trimmed Spearman-Kärber |

Notes:

Solubility (S) of fipronil = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Nominal concentrations (3), Measured concentrations (3), Photoperiod (3), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100-13 =87

Acceptability: Measured concentrations (4), Concentrations not > 2x solubility (4), Carrier solvent (4), No prior contamination (4), Organisms randomized (1), Photoperiod (2), Random design (2), Dilution factor (2), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1), Point estimates (3). Total: 100-29 =71

**Reliability score: mean(87, 71)=79**

## Water Toxicity Data Summary

*Xenopus laevis*

Fipronil

MB 46030

Overmyer JP, Rouse DR, Avants JK, Garrison AW, DeLorenzo ME, Chung KW, Key PB, Wilson WA and Black MC. (2007) Toxicity of fipronil and its enantiomers to marine and freshwater non-targets. *Journal of Environmental Science and Health Part B*, 42(5), 471-480.

Relevance

Score: 100

Rating: R

Reliability

Score: 72.5

Rating: L

Relevance points taken off for: none.

|  | <b>Overmyer et al. 2007</b>  | <i>X. laevis</i> |
|--|--|------------------|
| <b>Parameter</b>                             | <b>Value</b>   | <b>Comment</b>   |
| Test method cited                            | Mann, R.M.; Bidwell, J.R.<br>The acute toxicity of agricultural surfactants to the tadpoles of four Australian and two exotic frogs. <i>Environ. Pollut.</i> 2001, 114 (2), 195–205. |                  |
| Phylum/subphylum                             | Chordata   |                  |
| Class  | Amphibia   |                  |
| Order  | Anura  |                  |
| Family                                       | Pipidae  |                  |
| Genus  | <i>Xenopus</i>   |                  |
| Species                                      | <i>laevis</i>  |                  |
| Family native to North America?              | Introduced   |                  |
| Age/size at start of test/growth phase       | Tadpoles   |                  |
| Source of organisms                          | Carolina Biological, Burlington, North Carolina  |                  |
| Have organisms been exposed to contaminants? | No   |                  |
| Animals acclimated and disease-free?         | Yes  |                  |
| Animals randomized?                          | Not reported   |                  |
| Test vessels randomized?                     | Not reported   |                  |
| Test duration                                | 96 h   | Renewed at 48 h  |
| Data for multiple times?                     | No   |                  |
| Effect 1                                     | Survival   |                  |
| Control response 1                           | >90 %  |                  |

|   | <b>Overmyer et al. 2007</b>  | <b><i>X. laevis</i></b>                         |
|---|--|---|
| <b>Parameter</b>  | <b>Value</b>   | <b>Comment</b>                                  |
| Temperature   | 25 °C  |   |
| Test type   | Static   |   |
| Photoperiod/light intensity   | 16l:8d   |   |
| Dilution water  | Dechlorinated water  |   |
| pH  | Measured but not reported  |   |
| Hardness  | Not reported   |   |
| Alkalinity  | Not reported   |   |
| Conductivity  | Measured but not reported  |   |
| Dissolved Oxygen  | Measured but not reported  |   |
| Feeding   | Not reported   |   |
| Purity of test substance  | 98 %   |   |
| Concentrations measured?  | Yes  |   |
| Measured is what % of nominal?  | 80-130 %   | Only highest and lowest concentrations measured |
| Toxicity values calculated based on nominal or measured concentrations? | Nominal  |   |
| Chemical method documented?   | GCMS   |   |
| Concentration of carrier (if any) in test solutions                     | Acetone, 0.1 mL/L  |   |
| Concentration 1 Nom; Meas (µg/L)  | Racemate: 250; 200<br>(+): 250; 250<br>(-): 250; 215                           | 3 reps, 5/rep                                   |
| Concentration 2 Nom; Meas (µg/L)  | All: 500; Not reported   |   |
| Concentration 3 Nom; Meas (µg/L)  | All: 1000; Not reported  |   |
| Concentration 4 Nom; Meas (µg/L)  | Racemate: 2000; 2590<br>(+): 2000; 2030<br>(-): 2000; 2140                     |   |
| Control   | All: Negative<br>All: Solvent  |   |
| LC <sub>50</sub> (95% CI) (µg/L)  | Racemate: 850 (660-1090)<br>(+): 910 (650-1280)<br>(-): 163.50 (124.37-214.94) | Method: Trimmed Spearman-Kärber                 |

Notes:

Solubility (S) of fipronil = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Dilution water (3), Hardness (2), Alkalinity (2), Dissolved oxygen (4), Conductivity (2), pH (3), Photoperiod (3), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100-23 =77

Acceptability: Measured concentrations within 20% nominal (4), Organisms randomized (1), Feeding (3), Acclimation (1), Dilution water (2), Hardness (2), Alkalinity (2), Dissolved oxygen (6), Temperature variation (3), Conductivity (1), pH (2), Random design (2), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1). Total: 100-32 =68

**Reliability score: mean(77, 68)=72.5**

*Appendix A3 – Aqueous Toxicity Studies rated N, LN, RN*

## Water Toxicity Data Summary

*Acanthocyclops robustus*

Fipronil

MB 46030

Chaton PF, Ravanel P, Tissut M and Meyran JC. (2002) Toxicity and bioaccumulation of fipronil in the nontarget arthropodan fauna associated with subalpine mosquito breeding sites. *Ecotoxicology and Environmental Safety*, 52(1), 8-12.

Relevance

Score: R

Rating: 92.5

Reliability

Score: 56.5

Rating: N

Relevance points taken off for: Control response (7.5).  $100-7.5=92.5$

|  | <b>Chaton 2002</b>   | <i>A. robustus</i> |
|--|--|--------------------|
| <b>Parameter</b>                             | <b>Value</b>   | <b>Comment</b>     |
| Test method cited                            | WHO 1981 standard bioassay technique   |                    |
| Phylum/subphylum                             | Arthropoda   |                    |
| Class  | Maxillopoda  |                    |
| Order  | Cyclopoida   |                    |
| Family                                       | Cyclopoidae  |                    |
| Genus  | <i>Acanthocyclops</i>  |                    |
| Species                                      | <i>robustus</i>  |                    |
| Family native to North America?              | Yes  |                    |
| Age/size at start of test/growth phase       | Not reported   |                    |
| Source of organisms                          | Collected from subalpine breeding sites, specifics not reported                      |                    |
| Have organisms been exposed to contaminants? | Not reported   |                    |
| Animals acclimated and disease-free?         | Not acclimated. States they were subjected to bioassay immediately after collection. |                    |
| Animals randomized?                          | Not reported   |                    |
| Test vessels randomized?                     | Not reported   |                    |
| Test duration                                | 48 h   |                    |
| Data for multiple times?                     | Not reported   |                    |
| Effect 1                                     | Survival   |                    |
| Control response 1                           | Not reported   |                    |
| Temperature                                  | Not reported   |                    |
| Test type                                    | Static   |                    |

|   | <b>Chaton 2002</b>                  | <b><i>A. robustus</i></b> |
|---|-------------------------------------|---------------------------|
| <b>Parameter</b>  | <b>Value</b>                        | <b>Comment</b>            |
| Photoperiod/light intensity   | Not reported                        |                           |
| Dilution water  | Dechlorinated tap water             |                           |
| Feeding   | Not fed                             |                           |
| Purity of test substance  | 99.9 %                              |                           |
| Concentrations measured?  | Not reported                        |                           |
| Measured is what % of nominal?  | Not reported                        |                           |
| Toxicity values calculated based on nominal or measured concentrations? | Not reported                        |                           |
| Chemical method documented?   | Not reported                        |                           |
| Concentration of carrier (if any) in test solutions                     | Ethanol, concentration not reported |                           |
| Concentration 1 Nom; Meas (µg/L)  | 0.0004; Not reported                | 3 reps, 20/rep            |
| Concentration 2 Nom; Meas (µg/L)  | 0.004; Not reported                 |                           |
| Concentration 3 Nom; Meas (µg/L)  | 0.01; Not reported                  |                           |
| Concentration 4 Nom; Meas (µg/L)  | 0.02; Not reported                  |                           |
| Concentration 5 Nom; Meas (µg/L)  | 0.04; Not reported                  |                           |
| Concentration 6 Nom; Meas (µg/L)  | 0.1; Not reported                   |                           |
| Concentration 7 Nom; Meas (µg/L)  | 0.2; Not reported                   |                           |
| Concentration 8 Nom; Meas (µg/L)  | 0.4; Not reported                   |                           |
| Concentration 9 Nom; Meas (µg/L)  | 0.7; Not reported                   |                           |
| Concentration 10 Nom; Meas (µg/L)                                       | 0.9; Not reported                   |                           |
| Control   | Negative                            |                           |
| LC <sub>50</sub> (95% CI) (µg/L)  | 0.084 (143.9-262.1)                 | Method: log-probit        |

Notes: Water quality parameters not reported. Values reported in nM; conversion performed using Excel “=(X nM\*437.15\*1000)/10<sup>9</sup>”.

Solubility (S) of fipronil = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Organism life stage/size (5), Analytical method (4), Nominal concentrations (3), Hardness (2), Alkalinity (2), Dissolved oxygen (4), Temperature (4), Conductivity (2), pH (3), Photoperiod (3), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100-36=64

Acceptability: Control response (9), Measured concentrations within 20% nominal (4), Carrier solvent (4), No prior contamination (4), Organisms randomized (1), Acclimation (1), Hardness (2), Alkalinity (2), Dissolved oxygen (6), Temperature (6), Conductivity (1), pH (2), Photoperiod (2), Random design (2), Adequate replication (2), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1). Total: 100- 51=49

**Reliability score: mean(64, 49)=56.5**

## Water Toxicity Data Summary

### *Amphiascus tenuiremis*

Cary TL, Chandler GT, Volz DC, Walse SS and Ferry JL. (2004) Phenylpyrazole insecticide fipronil induces male infertility in the estuarine meiobenthic crustacean *Amphiascus tenuiremis*. *Environmental science & technology*, 38(2), 522-528.

Relevance

Score: 60

Rating: N

Reliability

Score: not calculated

Rating: not determined

Relevance points taken off for: Standard method (10), Freshwater (15), Toxicity value (15).  
100-40=60

|  | <b>Cary 2004</b>   | <b><i>A. tenuiremis</i></b> |
|--|--|-----------------------------|
| <b>Parameter</b>                             | <b>Value</b>   | <b>Comment</b>              |
| Test method cited                            | Not reported   |                             |
| Phylum/subphylum                             | Arthropoda   |                             |
| Class  | Copepoda   |                             |
| Order  | Harpacticoida  |                             |
| Family                                       | Miraciidae   |                             |
| Genus  | <i>Amphiascus</i>  |                             |
| Species                                      | <i>Tenuiremis</i>  |                             |
| Family native to North America?              | Yes  |                             |
| Age/size at start of test/growth phase       | Stage-I  |                             |
| Source of organisms                          | Laboratory culture   |                             |
| Have organisms been exposed to contaminants? | No   |                             |
| Animals acclimated and disease-free?         | Yes  |                             |
| Animals randomized?                          | Not reported   |                             |
| Test vessels randomized?                     | Not reported   |                             |
| Test duration                                | 24 d   |                             |
| Data for multiple times?                     |  |                             |
| Effect 1                                     | Survival   |                             |
| Control response 1                           | F <sub>0</sub> : 98.5 %<br>F <sub>1</sub> : 95.5 %   |                             |
| Effect 2                                     | Reproduction   |                             |
| Control response 2                           | 73% inhibition of reproduction when fipronil-reared males were mated with a control-reared female in fipronil-mating solution. |                             |

|   | <b>Cary 2004</b>  | <b><i>A. tenuiremis</i></b> |
|---|---|-----------------------------|
| <b>Parameter</b>  | <b>Value</b>  | <b>Comment</b>              |
|   | <p>89% inhibition of reproduction when fipronil-reared males were mated with fipronil-reared female in fipronil-mating solution.</p> <p>Control-reared males experienced no inhibition when mated with fipronil-reared females compared to mating with control-reared females when in a fipronil-mating solution.</p> <p>Fipronil-reared males mated to control- or fipronil-reared females in control-mating solution had 3-day delayed brood sac extrusion.</p> |                             |
| Temperature   | 25 ± 0 °C   |                             |
| Test type   | Static renewal  |                             |
| Photoperiod/light intensity   | 12l:12d; Not reported   |                             |
| Dilution water  | Filtered synthetic seawater   | 30 ‰, Instant Ocean         |
| pH  | Not reported  |                             |
| Hardness  | Not reported  |                             |
| Alkalinity  | Not reported  |                             |
| Conductivity  | Not reported  |                             |
| Dissolved Oxygen  | Not reported  |                             |
| Feeding   | 2 µL algae mixture every 6 d  |                             |
| Purity of test substance  | 98.0 %  |                             |
| Concentrations measured?  | Yes   |                             |
| Measured is what % of nominal?  | 105 %   |                             |
| Toxicity values calculated based on nominal or measured concentrations? | Not reported  |                             |
| Chemical method documented?   | GC-ECD  |                             |
| Concentration of carrier (if any) in test solutions                     | Acetone, 0.06 µL/L  |                             |
| Concentration 1 Nom; Meas (µg/L)  | 0.6; 0.63   | Single concentration;       |

|                                  | <b>Cary 2004</b> | <i>A. tenuiremis</i> |
|----------------------------------|------------------|----------------------|
| <b>Parameter</b>                 | <b>Value</b>     | <b>Comment</b>       |
|                                  |                  | 96/rep               |
| Control                          | Solvent: 0; 0    |                      |
| LC <sub>50</sub> (95% CI) (µg/L) | Not reported     | Method: Not reported |
| EC <sub>50</sub> (95% CI) (µg/L) | Not reported     | Method: Not reported |

Notes: Toxicity values not calculated. Single concentration tested.

Solubility (S) of fipronil = 1650.8 µg/L, 2S = 3301.6 µg/L.

## Water Toxicity Data Summary

### *Amphiascus tenuiremis*

Chandler GT, Cary TL, Volz DC, Walse SS, Ferry JL and Klosterhaus SL. (2004) Fipronil effects on estuarine copepod (*Amphiascus tenuiremis*) development, fertility, and reproduction: A rapid life-cycle assay in 96-well microplate format. *Environmental Toxicology and Chemistry*, 23(1), 117-124.

Relevance  
Score: 67.5  
Rating: N

Reliability  
Score: 66  
Rating: L

Relevance points taken off for: Standard method (10), Freshwater (15), Control response (7.5).  
100-32.5=67.5

|  | <b>Chandler 2004</b>         | <i>A. tenuiremis</i>                |
|--|------------------------------|-------------------------------------|
| <b>Parameter</b>                             | <b>Value</b>                 | <b>Comment</b>                      |
| Test method cited                            | Not reported                 |                                     |
| Phylum                                       | Anthropoda                   |                                     |
| Class  | Multicrustacea               |                                     |
| Order  | Harpacticoida                |                                     |
| Family                                       | Miraciidae                   |                                     |
| Genus  | <i>Amphiascus</i>            |                                     |
| Species                                      | <i>tenuiremis</i>            |                                     |
| Family native to North America?              | Yes                          |                                     |
| Age/size at start of test/growth phase       | Adult                        |                                     |
| Source of organisms                          | Laboratory sediment cultures |                                     |
| Have organisms been exposed to contaminants? | No                           |                                     |
| Animals acclimated and disease-free?         | Yes                          |                                     |
| Animals randomized?                          | Not reported                 |                                     |
| Test vessels randomized?                     | Not reported                 |                                     |
| Test duration                                | 96 h                         |                                     |
| Data for multiple times?                     | No                           |                                     |
| Effect 1                                     | Survival                     |                                     |
| Control response 1                           | Not reported                 |                                     |
| Temperature                                  | Not reported                 |                                     |
| Test type                                    | Static                       |                                     |
| Photoperiod/light intensity                  | 12l:12d                      |                                     |
| Dilution water                               | Artificial seawater          | 30 ‰ salinity,<br>Instant Ocean mix |
| pH   | 8.3                          |                                     |

|   | <b>Chandler 2004</b>   | <b><i>A. tenuiremis</i></b>     |
|---|--|---------------------------------|
| <b>Parameter</b>  | <b>Value</b>   | <b>Comment</b>                  |
| Dissolved Oxygen  | 8.3 mg/L   | > 90 %                          |
| Feeding   | Not fed  |                                 |
| Purity of test substance  | 98.0 %   |                                 |
| Concentrations measured?  | Yes  |                                 |
| Measured is what % of nominal?  | 63-98 %  |                                 |
| Toxicity values calculated based on nominal or measured concentrations? | Measured   |                                 |
| Chemical method documented?   | GC   |                                 |
| Concentration of carrier (if any) in test solutions                     | Acetone, <1 mL/L   |                                 |
| Concentration 1 Nom; Meas (µg/L)  | 4.3; 2.7   | 4 reps, 10 male+10 female/rep   |
| Concentration 2 Nom; Meas (µg/L)  | 7.2; 5.44  |                                 |
| Concentration 3 Nom; Meas (µg/L)  | 12.0; 10.84  |                                 |
| Concentration 4 Nom; Meas (µg/L)  | 20.0; 19.64  |                                 |
| Control   | Solvent: 0; 0  |                                 |
| LC <sub>50</sub> (95% CI) (µg/L)  | Adult: 6.8 (5.4-8.7)<br>Male: 3.5 (2.5-5.0)<br>Female: 13.0 (9.6-17.6) | Method: Trimmed Spearman-Kärber |

Notes:

Solubility (S) of fipronil = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Hardness (2), Alkalinity (2), Temperature (4), Conductivity (2), Statistical significance (2), Significance level (2), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100- 18=72

Acceptability: Standard method (5), Control response (9), Measured concentrations within 20% nominal (4), Organisms randomized (1), Adequate organisms per rep (2), Feeding (3), Hardness (2), Alkalinity (2), Temperature (6), Conductivity (1), Random design (2), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1). Total: 100- 40=60

**Reliability score: mean(72, 60)=66**

## Water Toxicity Data Summary

*Amphiascus tenuiremis*

Fipronil

MB 46030

Chronic

Chandler GT, Cary TL, Volz DC, Walse SS, Ferry JL and Klosterhaus SL. (2004) Fipronil effects on estuarine copepod (*Amphiascus tenuiremis*) development, fertility, and reproduction: A rapid life-cycle assay in 96-well microplate format. *Environmental Toxicology and Chemistry*, 23(1), 117-124.

Relevance

Score: 60

Rating: N

Reliability

Score: 72.5

Rating: L

Relevance points taken off for: Standard method (10), Freshwater (15), Toxicity value (15).  
100-40=60

|  | <b>Chandler 2004</b>          | <i>A. tenuiremis</i> |
|--|-------------------------------|----------------------|
| <b>Parameter</b>                             | <b>Value</b>                  | <b>Comment</b>       |
| Test method cited                            | Not reported                  |                      |
| Phylum                                       | Anthropoda                    |                      |
| Class  | Multicrustacea                |                      |
| Order  | Harpacticoida                 |                      |
| Family                                       | Miraciidae                    |                      |
| Genus  | <i>Amphiascus</i>             |                      |
| Species                                      | <i>tenuiremis</i>             |                      |
| Family native to North America?              | Yes                           |                      |
| Age/size at start of test/growth phase       | Stage 1 copepodites, 70-63 µm |                      |
| Source of organisms                          | Laboratory sediment cultures  |                      |
| Have organisms been exposed to contaminants? | No                            |                      |
| Animals acclimated and disease-free?         | Yes                           |                      |
| Animals randomized?                          | Yes                           |                      |
| Test vessels randomized?                     | Not reported                  |                      |
| Test duration                                | 21 d                          |                      |
| Data for multiple times?                     | 12, 21 d                      |                      |
| Effect 1                                     | Survival                      |                      |
| Control response 1                           | 12 d: 93 %<br>21 d: 91 %      |                      |
| Temperature                                  | 25°C                          |                      |
| Test type                                    | Static                        |                      |

|   | <b>Chandler 2004</b>   | <b><i>A. tenuiremis</i></b>                                  |
|---|--|--|
| <b>Parameter</b>  | <b>Value</b>   | <b>Comment</b>   |
| Photoperiod/light intensity   | 12L:12d  |  |
| Dilution water  | Artificial seawater  | 30 ‰ salinity,<br>Instant Ocean mix                          |
| Feeding   | 2 ml of a fresh, centrifuged,<br>107 cells/ml, 1:1 mixed<br>algal cell suspension of <i>I.</i><br><i>galbana</i> and <i>D. tertiolecta</i> |  |
| Purity of test substance  | 98.0 %   |  |
| Concentrations measured?  | Yes  |  |
| Measured is what % of nominal?  | 61-72 %  |  |
| Toxicity values calculated based on nominal or measured concentrations? | Measured   |  |
| Chemical method documented?   | GC   |  |
| Concentration of carrier (if any) in test solutions                     | Acetone, <1 mL/L   |  |
| Concentration 1 Nom; Meas (µg/L)  | 0.22; 0.16   | 144 test wells or 3<br>microplates, 10<br>male+10 female/rep |
| Concentration 2 Nom; Meas (µg/L)  | 0.36; 0.22   |  |
| Concentration 3 Nom; Meas (µg/L)  | 0.60; 0.42   |  |
| Control   | Solvent: 0; 0  |  |

Notes: Toxicity endpoints not reported. Raw data not included so values cannot be calculated.

Solubility (S) of fipronil = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Hardness (2), Alkalinity (2), Dissolved oxygen (4), Conductivity (2), pH (3), Minimum significant difference (2), % control at NOEC/LOEC (2), Point estimates (8). Total: 100- 25=75

Acceptability: Measured concentrations within 20% nominal (4), Hardness (2), Alkalinity (2), Dissolved oxygen (6), Conductivity (1), pH (2), Photoperiod (2), Number of concentrations (3), Random design (2), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1), Point estimates (3). Total: 100-30 =70

**Reliability score: mean(75, 70)=72.5**

## Water Toxicity Data Summary

*Crassostrea virginica*

Fipronil

MB 46030

Dionne E. (1993) MB 46030-Acute toxicity to the eastern oyster (*Crassostrea virginica*) under flow-through conditions. Springborn Laboratories, Inc., Wareham, Massachusetts. Laboratory study number 10566.0393.6269.504. Submitted to Rhone-Poulenc Ag Company, Research Triangle, North Carolina. USEPA MRID 43291701. CA DPR 157285.

**Acute study endpoint not related to survival and study only tested a single concentration so it automatically rates N and cannot be used for criteria derivation.**

## Water Toxicity Data Summary

*Daphnia magna*  
Fipronil desulfinyl  
MB 46513

Iwafune T, Yokoyama A, Nagai T and Horio T. (2011) Evaluation of the risk of mixtures of paddy insecticides and their transformation products to aquatic organisms in the Sakura River, Japan. *Environmental Toxicology and Chemistry*, 30(8), 1834-1842.

Relevance  
Score: 67.5  
Rating: N

Reliability  
Score: 62.5  
Rating: L

Relevance points taken off for: Standard method (10), Toxicity value (15), Control described (7.5).  $100 - 32.5 = 67.5$

|  | <b>Iwafune 2011</b>                  | <b><i>D. magna</i></b> |
|--|--------------------------------------|------------------------|
| <b>Parameter</b>                             | <b>Value</b>                         | <b>Comment</b>         |
| Test method cited                            | Not reported                         |                        |
| Phylum/subphylum                             | Arthropoda/Crustacea                 |                        |
| Class  | Branchiopoda                         |                        |
| Order  | Cladocera                            |                        |
| Family                                       | Daphniidae                           |                        |
| Genus  | <i>Daphnia</i>                       |                        |
| Species                                      | <i>magna</i>                         |                        |
| Family native to North America?              | Yes                                  |                        |
| Age/size at start of test/growth phase       | 1 <sup>st</sup> instar               |                        |
| Source of organisms                          | Not reported                         |                        |
| Have organisms been exposed to contaminants? | Not reported                         |                        |
| Animals acclimated and disease-free?         | Not reported                         |                        |
| Animals randomized?                          | Not reported                         |                        |
| Test vessels randomized?                     | Not reported                         |                        |
| Test duration                                | 48 h                                 |                        |
| Data for multiple times?                     | No                                   |                        |
| Effect 1                                     | Immobilization                       |                        |
| Control response 1                           | ≤5 %                                 |                        |
| Temperature                                  | 20.9 ± 0.6 °C                        |                        |
| Test type                                    | Static                               |                        |
| Photoperiod/light intensity                  | Not reported                         |                        |
| Dilution water                               | Dechlorinated and filtered tap water |                        |
| pH   | 8                                    |                        |

|   | <b>Iwafune 2011</b>              | <b><i>D. magna</i></b>    |
|---|----------------------------------|---------------------------|
| <b>Parameter</b>  | <b>Value</b>                     | <b>Comment</b>            |
| Hardness  | 70 mg/L CaCO <sub>3</sub>        |                           |
| Alkalinity  | Not reported                     |                           |
| Conductivity  | Not reported                     |                           |
| Dissolved Oxygen  | Not reported                     |                           |
| Feeding   | Not reported                     |                           |
| Purity of test substance  | >98 %                            |                           |
| Concentrations measured?  | Yes                              |                           |
| Measured is what % of nominal?  | 84.1-105.3 %                     |                           |
| Toxicity values calculated based on nominal or measured concentrations? | Measured                         |                           |
| Chemical method documented?   | LC-MS/MS                         |                           |
| Concentration of carrier (if any) in test solutions                     | Acetone or acetonitrile, ≤1 mL/L |                           |
| Concentration 1 Nom; Meas (µg/L)  | 10; Not reported                 | Reps not reported, 20/rep |
| Control   | Not reported                     |                           |
| EC <sub>50</sub> (95% CI) (µg/L)  | >9.39                            | Method: probit            |

Notes:

Solubility (S) value for fipronil desulfinyl (MB 46513) = 950 µg/L, 2S = 1900 µg/L.

Reliability points taken off for:

Documentation: Organism source (5), Nominal concentrations (3), Measured concentrations (3), Alkalinity (2), Dissolved oxygen (4), Conductivity (2), Photoperiod (3), Statistical significance (2), Significance level (2), Minimum significant difference (2), % control at NOEC/LOEC (2), Point estimates (8). Total: 100-38 =62

Acceptability: Standard method (5), No prior contamination (4), Organisms randomized (1), Feeding (3), Acclimation (1), Alkalinity (2), Dissolved oxygen (6), Conductivity (1), Photoperiod (2), Random design (2), Replication (2), Dilution factor (2), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1), Point estimates (3). Total: 100-37 =63

**Reliability score: mean(62, 63)=62.5**

## Water Toxicity Data Summary

*Daphnia magna*  
Fipronil carboxamide  
RPA 200766

Iwafune T, Yokoyama A, Nagai T and Horio T. (2011) Evaluation of the risk of mixtures of paddy insecticides and their transformation products to aquatic organisms in the Sakura River, Japan. *Environmental Toxicology and Chemistry*, 30(8), 1834-1842.

Relevance  
Score: 67.5  
Rating: N

Reliability  
Score: 62.5  
Rating: L

Relevance points taken off for: Standard method (10), Toxicity value (15), Control described (7.5).  $100 - 32.5 = 67.5$

|  | <b>Iwafune 2011</b>                  | <b><i>D. magna</i></b> |
|--|--------------------------------------|------------------------|
| <b>Parameter</b>                             | <b>Value</b>                         | <b>Comment</b>         |
| Test method cited                            | Not reported                         |                        |
| Phylum/subphylum                             | Arthropoda/Crustacea                 |                        |
| Class  | Branchiopoda                         |                        |
| Order  | Cladocera                            |                        |
| Family                                       | Daphniidae                           |                        |
| Genus  | <i>Daphnia</i>                       |                        |
| Species                                      | <i>magna</i>                         |                        |
| Family native to North America?              | Yes                                  |                        |
| Age/size at start of test/growth phase       | 1 <sup>st</sup> instar               |                        |
| Source of organisms                          | Not reported                         |                        |
| Have organisms been exposed to contaminants? | Not reported                         |                        |
| Animals acclimated and disease-free?         | Not reported                         |                        |
| Animals randomized?                          | Not reported                         |                        |
| Test vessels randomized?                     | Not reported                         |                        |
| Test duration                                | 48 h                                 |                        |
| Data for multiple times?                     | No                                   |                        |
| Effect 1                                     | Immobilization                       |                        |
| Control response 1                           | ≤5 %                                 |                        |
| Temperature                                  | 20.9 ± 0.6 °C                        |                        |
| Test type                                    | Static                               |                        |
| Photoperiod/light intensity                  | Not reported                         |                        |
| Dilution water                               | Dechlorinated and filtered tap water |                        |
| pH   | 8                                    |                        |

|   | <b>Iwafune 2011</b>              | <b><i>D. magna</i></b>    |
|---|----------------------------------|---------------------------|
| <b>Parameter</b>  | <b>Value</b>                     | <b>Comment</b>            |
| Hardness  | 70 mg/L CaCO <sub>3</sub>        |                           |
| Alkalinity  | Not reported                     |                           |
| Conductivity  | Not reported                     |                           |
| Dissolved Oxygen  | Not reported                     |                           |
| Feeding   | Not reported                     |                           |
| Purity of test substance  | >98 %                            |                           |
| Concentrations measured?  | Yes                              |                           |
| Measured is what % of nominal?  | 84.1-105.3 %                     |                           |
| Toxicity values calculated based on nominal or measured concentrations? | Measured                         |                           |
| Chemical method documented?   | LC-MS/MS                         |                           |
| Concentration of carrier (if any) in test solutions                     | Acetone or acetonitrile, ≤1 mL/L |                           |
| Concentration 1 Nom; Meas (µg/L)  | 10; Not reported                 | Reps not reported, 20/rep |
| Control   | Not reported                     |                           |
| EC <sub>50</sub> (95% CI) (µg/L)  | >9.62                            | Method: probit            |

Notes:

Solubility value for this fipronil metabolite (RPA 200766) not available. Solubility (S) of fipronil parent compound (MB 46030) = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Organism source (5), Nominal concentrations (3), Measured concentrations (3), Alkalinity (2), Dissolved oxygen (4), Conductivity (2), Photoperiod (3), Statistical significance (2), Significance level (2), Minimum significant difference (2), % control at NOEC/LOEC (2), Point estimates (8). Total: 100-38 =62

Acceptability: Standard method (5), No prior contamination (4), Organisms randomized (1), Feeding (3), Acclimation (1), Alkalinity (2), Dissolved oxygen (6), Conductivity (1), Photoperiod (2), Random design (2), Replication (2), Dilution factor (2), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1), Point estimates (3). Total: 100-37 =63

**Reliability score: mean(62, 63)=62.5**

## Water Toxicity Data Summary

*Lemna gibba*

Fipronil

MB46030

Hoberg JR. (1993) MB 46030-Toxicity to duckweed *Lemna gibba*. Springborn Laboratories, Inc., Wareham, Massachusetts. Laboratory study number 10566.0.393.6274.410. Submitted to Rhone-Poulenc Ag Company, Research Triangle, North Carolina. USEPA MRID 42918656. CA DPR 157293.

**Single concentration tested so toxicity values not determined (only > estimates). Study rates N and cannot be used for criteria derivation.**

## Water Toxicity Data Summary

*N. pelliculosa*

Fipronil

MB46030

Hoberg JR (1993) MB46030-Toxicity to the freshwater diatom, *Navicula pelliculosa*. Springborn Laboratories, Inc., Wareham, Massachusetts. Laboratory study number 10566.0393.6272.440. Submitted to Rhone-Poulenc Ag Company, Research Triangle, North Carolina. CA DPR 157294.

**Single concentration tested so toxicity values not determined (only > estimates). Study rates N and cannot be used for criteria derivation.**

## Water Toxicity Data Summary

*N. pelliculosa*

Fipronil

MB46030

Hoberg JR (1993) MB46030-Toxicity to the freshwater diatom, *Navicula pelliculosa*. Springborn Laboratories, Inc., Wareham, Massachusetts. Laboratory study number 10566.0393.6272.440. Submitted to Rhone-Poulenc Ag Company, Research Triangle, North Carolina. CA DPR 157294.

**Single concentration tested so toxicity values not determined (only > estimates). Study rates N and cannot be used for criteria derivation.**

## Water Toxicity Data Summary

*Polypedilum nubiferum*

Fipronil

MB 46030

Stevens MM, Burdett AS, Mudford EM, Helliwell S and Doran G. (2011) The acute toxicity of fipronil to two non-target invertebrates associated with mosquito breeding sites in Australia. *Acta tropica*, 117(2), 125-130.

Relevance

Score: 82.5

Rating: L

Reliability

Score: 58.5

Rating: N

Relevance points taken off for: Standard method (10), Control response (7.5). 100-17.5 = 82.5

|  | <b>Stevens 2011</b>   | <b><i>P. nubiferum</i></b> |
|--|---|----------------------------|
| <b>Parameter</b>                             | <b>Value</b>  | <b>Comment</b>             |
| Test method cited                            | Not reported  |                            |
| Phylum/subphylum                             | Arthropoda  |                            |
| Class  | Insecta   |                            |
| Order  | Diptera   |                            |
| Family                                       | Chironomidae  |                            |
| Genus  | <i>Polypedilum</i>  |                            |
| Species                                      | <i>nubiferum</i>  |                            |
| Family native to North America?              | Possibly introduced   |                            |
| Age/size at start of test/growth phase       | 4 <sup>th</sup> instar  |                            |
| Source of organisms                          | Collected from temporary pools and irrigation canals, Yanoc, New South Wales, Australia |                            |
| Have organisms been exposed to contaminants? | No  |                            |
| Animals acclimated and disease-free?         | Yes   |                            |
| Animals randomized?                          | Not reported  |                            |
| Test vessels randomized?                     | Not reported  |                            |
| Test duration                                | 48 h  |                            |
| Data for multiple times?                     | No  |                            |
| Effect 1                                     | Survival  |                            |
| Control response 1                           | Not reported  |                            |
| Temperature                                  | 25 ± 1 °C   |                            |
| Test type                                    | Static  |                            |
| Photoperiod/light intensity                  | 15L:9d  |                            |
| Dilution water                               | Martin's rearing solution   |                            |

|   | <b>Stevens 2011</b>                                | <b><i>P. nubiferum</i></b>   |
|---|--|--|
| <b>Parameter</b>  | <b>Value</b>                                       | <b>Comment</b>   |
| pH  | Not reported                                       |  |
| Hardness  | Not reported                                       |  |
| Alkalinity  | Not reported                                       |  |
| Conductivity  | Not reported                                       |  |
| Dissolved Oxygen  | Not reported                                       | Not aerated  |
| Feeding   | Finely ground fish food/brewer's yeast mixture     |  |
| Purity of test substance  | 95 %   |  |
| Concentrations measured?  | Yes  |  |
| Measured is what % of nominal?  | Not reported                                       |  |
| Toxicity values calculated based on nominal or measured concentrations? | Not reported                                       |  |
| Chemical method documented?   | GC   |  |
| Concentration of carrier (if any) in test solutions                     | Acetone, concentration not reported                |  |
| Concentration 1 Nom; Meas (µg/L)  | 6 concentrations used, concentrations not reported | 6 reps, 10/rep<br>Reps on different days/solutions/larval cultures |
| Control   | Solvent  |  |
| LC <sub>50</sub> (95% CI) (µg/L)  | Unfed: 1.00 (0.51-1.33)<br>Fed: 2.18 (1.70-2.49)   | Method: Probit   |

Notes:

Solubility (S) of fipronil = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Nominal concentrations (3), Measured concentrations (3), Hardness (2), Alkalinity (2), Dissolved oxygen (4), Conductivity (2), pH (3), Statistical significance (2), Significance level (2), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100-25 =75

Acceptability: Standard method (5), Control response (9), Measured concentrations within 20% nominal (4), Concentrations not > 2x solubility (4), Carrier solvent (4), No prior contamination (4), Organisms randomized (1), Feeding (3), Hardness (2), Alkalinity (2), Dissolved oxygen (6), Conductivity (1), pH (2), Random design (2), Dilution factor (2), Statistical method (2), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1). Total: 100-58 =42

**Reliability score: mean(75, 42)=58.5**

## Water Toxicity Data Summary

*Palaemonetes pugio*

Fipronil

MB 46030

Key PB, Chung KW, Opatkiewicz AD, Wirth EF and Fulton MH. (2003) Toxicity of the insecticides fipronil and endosulfan to selected life stages of the grass shrimp (*Palaemonetes pugio*). Bulletin of environmental contamination and toxicology, 70(3), 0533-0540.

Relevance

Score: 67.5

Rating: N

Reliability

Score: 60

Rating: L

Relevance points taken off for: Standard method (10), Freshwater (15), Control response (7.5).  
100-22.5=67.5

|  | <b>Key 2003</b>   | <b><i>P. pugio</i></b> |
|--|---|------------------------|
| <b>Parameter</b>                             | <b>Value</b>  | <b>Comment</b>         |
| Test method cited                            | Not reported  |                        |
| Phylum/subphylum                             | Arthropoda/crustacea  |                        |
| Class  | Malacostraca  |                        |
| Order  | Decapoda/caridea  |                        |
| Family                                       | Palaemonidae  |                        |
| Genus  | <i>Palaemonetes</i>   |                        |
| Species                                      | <i>Pugio</i>  |                        |
| Family native to North America?              | Yes   |                        |
| Age/size at start of test/growth phase       | Adult<br>Larvae, 1-2 d<br>Stage VI embryos  |                        |
| Source of organisms                          | Collected from Leadenwah Creek, a pristine tidal estuary (N 32°36'12"; W 80°07'00") |                        |
| Have organisms been exposed to contaminants? | Not reported  |                        |
| Animals acclimated and disease-free?         | Yes   |                        |
| Animals randomized?                          | Not reported  |                        |
| Test vessels randomized?                     | Not reported  |                        |
| Test duration                                | 96 h  |                        |
| Data for multiple times?                     | No  |                        |
| Effect 1                                     | Survival  |                        |
| Control response 1                           | Not reported  |                        |
| Temperature                                  | 25 °C   |                        |
| Test type                                    | Static renewal  |                        |

|   | <b>Key 2003</b>   | <b><i>P. pugio</i></b>  |
|---|---|---|
| <b>Parameter</b>  | <b>Value</b>  | <b>Comment</b>  |
| Photoperiod/light intensity   | 16l:8d  |   |
| Dilution water  | 20 ‰ salinity, source not reported                                      |   |
| Feeding   | Adults: not fed<br>Larvae: <i>Artemia</i> daily                         |   |
| Purity of test substance  | Not reported  |   |
| Concentrations measured?  | Not reported  |   |
| Measured is what % of nominal?  | Not reported  |   |
| Toxicity values calculated based on nominal or measured concentrations? | Not reported  |   |
| Chemical method documented?   | Not reported  |   |
| Concentration of carrier (if any) in test solutions                     | Acetone, 0.1 mL/L   |   |
| Concentration 1 Nom; Meas (µg/L)  | Adult/larvae: 0.13; Not reported<br>Embryos: 32.0; Not reported         | Adults: 2 reps, 10/rep<br>Larvae: 3 reps, 10/rep                                |
| Concentration 2 Nom; Meas (µg/L)  | Adult/larvae: 0.25; Not reported<br>Embryos: 64.0; Not reported         |   |
| Concentration 3 Nom; Meas (µg/L)  | Adult/larvae: 0.50; Not reported<br>Embryos: 128.0; Not reported        |   |
| Concentration 4 Nom; Meas (µg/L)  | Adult/larvae: 1.00; Not reported<br>Embryos: 256.0; Not reported        |   |
| Concentration 5 Nom; Meas (µg/L)  | Adult/larvae: 2.00; Not reported<br>Embryos: 512.0; Not reported        |   |
| Control   | Solvent   |   |
| LC <sub>50</sub> (95% CI) (µg/L)  | Adults: 0.32 (0.24-0.41)<br>Larvae: 0.68 (0.57-0.80)<br>Embryos: >512.0 | Method: Trimmed Spearman-Kärber   |
| NOEC  | Adults: <0.13<br>Larvae: 0.25<br>Embryos: <0.32                         | Method: Kruskal-Wallis non-parametric one-way ANOVA and Dunn's Method<br>p: .05 |

|                           | <b>Key 2003</b>   | <b><i>P. pugio</i></b> |
|---------------------------|---|------------------------|
| <b>Parameter</b>          | <b>Value</b>  | <b>Comment</b>         |
|                           |   | MSD: Not reported      |
| LOEC                      | Adults: 0.13<br>Larvae: 0.50<br>Embryos: 0.32                     |                        |
| MATC (GeoMean NOEC, LOEC) | Adults: not calculable<br>Larvae: 0.35<br>Embryos: not calculable |                        |
| % control at NOEC         | Not calculable  |                        |
| % control at LOEC         | Not calculable  |                        |

Notes: Water quality parameters measured but not reported.

Solubility (S) of fipronil = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation: Analytical method (4), Measured concentrations (3), Exposure type (5), Temperature (4), Photoperiod (3), Statistics method (5), Statistical significance (2), Significance level (2), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100- 32=68

Acceptability: Standard method (5), Control response (9), Measured concentrations within 20% nominal (4), No prior contamination (4), Organisms randomized (1), Dilution water (2), Hardness (2), Alkalinity (2), Dissolved oxygen (6), Temperature variation (3), Conductivity (1), pH (2), Random design (2), Statistical method (2), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1). Total: 100-48 =52

**Reliability score: mean(68, 52)=60**

## Water Toxicity Data Summary

*R. subcapitata*

Fipronil

MB46030

Hoberg JR (1993) MB46030-Toxicity to the freshwater green alga, *Selenastrum capricornutum*. Springborn Laboratories, Inc., Wareham, Massachusetts. Laboratory study number 10566.0393.6271.430. Submitted to Rhone-Poulenc Ag Company, Research Triangle, North Carolina. CA DPR 157291.

**Single concentration tested so toxicity values not determined (only > estimates). Study rates N and cannot be used for criteria derivation.**

## Water Toxicity Data Summary

*S. costatum*

Fipronil

MB46030

Hoberg JR (1993) MB46030-Toxicity to the marine diatom, *Skeltonema costatum*. Springborn Laboratories, Inc., Wareham, Massachusetts. Laboratory study number 10566.0393.6270.450. Submitted to Rhone-Poulenc Ag Company, Research Triangle, North Carolina. CA DPR 157295.

**Single concentration tested so toxicity values not determined (only > estimates). Study rates N and cannot be used for criteria derivation.**

# **Appendix B – Sediment Toxicity Data**

## **Summaries**

*Appendix B1 – Sediment Toxicity Studies rated RR*

## Sediment Toxicity Data Summary

*C. dilutus*  
Fipronil sulfide  
MB45950

Maul JD, Brennan AA, Harwood AD and Lydy MJ. (2008) Effect of sediment-associated pyrethroids, fipronil, and metabolites on *Chironomus tentans* growth rate, body mass, condition index, immobilization, and survival. *Environmental Toxicology and Chemistry*, 27(12), 2582-2590.

Relevance  
Score: 100  
Rating: R

Reliability  
Score: 87.5  
Rating: R

Relevance points taken off for: none

| Fipronil-sulfide                             | <b>Maul 2008</b>   | <i>C. dilutus</i> |
|--|--|-------------------|
| <b>Parameter</b>                             | <b>Value</b>   | <b>Comment</b>    |
| Test method cited                            | U.S. Environmental Protection Agency. 2000. Methods for measuring the toxicity and bioaccumulation of sediment-associated contaminants with freshwater invertebrates, 2 <sup>nd</sup> ed. EPA 600/R-99/064. Guidance Document. Washington, DC. |                   |
| Phylum                                       | Anthropoda   |                   |
| Class  | Insecta  |                   |
| Order  | Diptera  |                   |
| Family                                       | Chironomidae   |                   |
| Genus  | <i>Chironomus</i>  |                   |
| Species                                      | <i>dilutus</i>   |                   |
| Family in North America?                     | Yes  |                   |
| Age/size at start of test/growth phase       | Early to mid-fourth instar   |                   |
| Source of organisms                          | Southern Illinois University   |                   |
| Have organisms been exposed to contaminants? | No   |                   |
| Animals acclimated and disease-free?         | Yes  |                   |
| Animals randomized?                          | Yes  |                   |

|   |                                    |  |
|---|------------------------------------|--|
| Fipronil-sulfide                              | <b>Maul 2008</b>                   | <i>C. dilutus</i>                              |
| <b>Parameter</b>                              | <b>Value</b>                       | <b>Comment</b>                                 |
| Test vessels randomized?                      | Yes                                |  |
| Test duration                                 | 10 d                               |  |
| Effect 1                                      | Survival                           |  |
| Control response 1                            | 95 %                               |  |
| Effect 2                                      | Head capsule width, length         |  |
| Control response 2                            | Not reported                       |  |
| Effect 3                                      | Dry weight                         |  |
| Control response 3                            | Not reported                       |  |
| Effect 4                                      | Growth rate                        |  |
| Control response 4                            | Not reported                       |  |
| Temperature                                   | 23 ± 1 °C                          |  |
| Test type                                     | Static                             |  |
| Photoperiod/light intensity                   | 16l:8d; Not reported               |  |
| Overlying water                               | Moderately hard water              | According to standard method EPA 600/R-99/064. |
| pH  | 6.67-6.96                          |  |
| Hardness                                      | Not reported                       |  |
| Alkalinity                                    | Not reported                       |  |
| Conductivity                                  | 342-399 µS/cm                      |  |
| Dissolved Oxygen                              | 6.40-7.34 mg/L                     |  |
| TOC   | Not reported                       |  |
| DOC   | Not reported                       |  |
| Ammonia-N                                     | Not reported                       |  |
| Chemical analysis?/Method                     | GC                                 |  |
| Sediment source                               | Carbondale, Illinois               |  |
| Organic carbon                                | 0.69 %                             |  |
| Particle size distribution (sand, silt, clay) |                                    |  |
| pH  |                                    |  |
| Percent solids                                |                                    |  |
| Sediment spike procedure                      | Direct addition to sediment slurry |  |
| Carrier solvent addition; evaporated (y/n)    | Acetone; not evaporated            |  |
| Sediment spike equilibration time             | 14 d                               |  |
| Sediment to Solution ratio                    | 50 g (dry weight): 700 mL          |  |
| Sediment extraction/analysis methods          | GC                                 |  |
| Interstitial water monitored?                 | Yes                                |  |
| Interstitial water isolation method           | Centrifuge                         | 3,300 g  |

| Fipronil-sulfide  | Maul 2008                       | <i>C. dilutus</i>                                       |
|---|---------------------------------|---|
| Parameter   | Value                           | Comment   |
| Interstitial water extraction/analysis method                           | GC                              |   |
| DOC   | Not reported                    |   |
| Feeding   | 1 mL 6 g/L Tetrafin solution    |   |
| Purity of test substance  | Analytical grade                | Accustandard  |
| Measured is what % of nominal?  |                                 |   |
| Toxicity values calculated based on nominal or measured concentrations? | Measured                        | Time-weighted exposure concentration due to degradation |
| Concentration of carrier (if any) in test solutions                     | Acetone                         |   |
| Concentration 1 Nom;Meas ( $\mu\text{g/g C}$ )                          | 0.07; Not reported              | 5 reps; 10/rep  |
| Concentration 2 Nom;Meas ( $\mu\text{g/g C}$ )                          | 0.11; Not reported              |   |
| Concentration 3 Nom;Meas ( $\mu\text{g/g C}$ )                          | 0.13; Not reported              |   |
| Concentration 4 Nom;Meas ( $\mu\text{g/g C}$ )                          | 0.23; Not reported              |   |
| Concentration 5 Nom;Meas ( $\mu\text{g/g C}$ )                          | 0.33; Not reported              |   |
| Control   | Negative: 0; 0<br>Solvent: 0; 0 |   |
| LC50 (95% CI) ( $\mu\text{g/g C}$ )                                     | 0.16 (0.12-0.23)                | Method: Probit, logit, Weibull                          |
| EC50 (95% CI) ( $\mu\text{g/g C}$ )                                     | 0.06 (0.03-0.07)                | Immobilization  |
| LOEC ( $\mu\text{g/g C}$ )  | Dry weight/growth rate: 0.1     | Method: Dunnett's test<br>p: 0.05<br>MSD: Not reported  |
| NOEC ( $\mu\text{g/g C}$ )  | Dry weight/growth rate:<br>0.07 | See Figure 2B (next lowest value to reported LOEC)      |
| MATC  | 0.08                            |   |

Notes: raw data not included. Points for water quality not deducted because standard method cited in water quality explanation.

Solubility value for fipronil sulfide (MB 45950) not available. Solubility (S) of fipronil parent compound (MB 46030) = 1650.8  $\mu\text{g/L}$ , 2S = 3301.6  $\mu\text{g/L}$ .

Reliability points taken off for:

Documentation (Table 9): Sediment particle size (1), Sediment TOC (3), Minimum significant difference (2), % control at NOEC/LOEC (2). Total:  $100-8=92$

Acceptability (Table 10): Spike method (4), Equilibration time (6), Fully evaporated (4), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1). Total:  $100-17=83$

**Reliability score: Mean (92, 83)= 87.5**

## Sediment Toxicity Data Summary

*C. dilutus*  
Fipronil  
MB46030

Maul JD, Brennan AA, Harwood AD and Lydy MJ. (2008) Effect of sediment-associated pyrethroids, fipronil, and metabolites on *Chironomus tentans* growth rate, body mass, condition index, immobilization, and survival. *Environmental Toxicology and Chemistry*, 27(12), 2582-2590.

Relevance  
Score: 100  
Rating: R

Reliability  
Score: 87.5  
Rating: R

Relevance points taken off for: none

| Fipronil                                     | <b>Maul 2008</b>   | <i>C. dilutus</i> |
|--|--|-------------------|
| <b>Parameter</b>                             | <b>Value</b>   | <b>Comment</b>    |
| Test method cited                            | U.S. Environmental Protection Agency. 2000. Methods for measuring the toxicity and bioaccumulation of sediment-associated contaminants with freshwater invertebrates, 2 <sup>nd</sup> ed. EPA 600/R-99/064. Guidance Document. Washington, DC. |                   |
| Phylum                                       | Anthropoda   |                   |
| Class  | Insecta  |                   |
| Order  | Diptera  |                   |
| Family                                       | Chironomidae   |                   |
| Genus  | <i>Chironomus</i>  |                   |
| Species                                      | <i>dilutus</i>   |                   |
| Family in North America?                     | Yes  |                   |
| Age/size at start of test/growth phase       | Early to mid-fourth instar   |                   |
| Source of organisms                          | Southern Illinois University   |                   |
| Have organisms been exposed to contaminants? | No   |                   |
| Animals acclimated and disease-free?         | Yes  |                   |
| Animals randomized?                          | Yes  |                   |

|   |                                    |  |
|---|------------------------------------|--|
| Fipronil                                      | <b>Maul 2008</b>                   | <i>C. dilutus</i>                              |
| <b>Parameter</b>                              | <b>Value</b>                       | <b>Comment</b>                                 |
| Test vessels randomized?                      | Yes                                |  |
| Test duration                                 | 10 d                               |  |
| Effect 1                                      | Survival                           |  |
| Control response 1                            | 95 %                               |  |
| Effect 2                                      | Head capsule width, length         |  |
| Control response 2                            | Not reported                       |  |
| Effect 3                                      | Dry weight                         |  |
| Control response 3                            | Not reported                       |  |
| Effect 4                                      | Growth rate                        |  |
| Control response 4                            | Not reported                       |  |
| Temperature                                   | 23 ± 1 °C                          |  |
| Test type                                     | Static                             |  |
| Photoperiod/light intensity                   | 16l:8d; Not reported               |  |
| Overlying water                               | Moderately hard water              | According to standard method EPA 600/R-99/064. |
| pH  | 6.91-7.10                          |  |
| Hardness                                      | Not reported                       |  |
| Alkalinity                                    | Not reported                       |  |
| Conductivity                                  | 255-440 µS/cm                      |  |
| Dissolved Oxygen                              | 5.32-7.94 mg/L                     |  |
| TOC   | Not reported                       |  |
| DOC   | Not reported                       |  |
| Ammonia-N                                     | Not reported                       |  |
| Chemical analysis?/Method                     | GC                                 |  |
| Sediment source                               | Carbondale, Illinois               |  |
| Organic carbon                                | 0.69 %                             |  |
| Particle size distribution (sand, silt, clay) | Not reported                       |  |
| pH  | Not reported                       |  |
| Percent solids                                | Not reported                       |  |
| Sediment spike procedure                      | Direct addition to sediment slurry |  |
| Carrier solvent addition; evaporated (y/n)    | Acetone; not evaporated            |  |
| Sediment spike equilibration time             | 14 d                               |  |
| Sediment to Solution ratio                    | 50 g (dry weight): 700 mL          |  |
| Sediment extraction/analysis methods          | GC                                 |  |
| Interstitial water monitored?                 | Yes                                |  |
| Interstitial water isolation method           | Centrifuge                         | 3,300 g  |

| Fipronil  | Maul 2008                       | <i>C. dilutus</i>                                       |
|---|---------------------------------|---|
| Parameter   | Value                           | Comment   |
| Interstitial water extraction/analysis method                           | GC                              |   |
| DOC   | Not reported                    |   |
| Feeding   | 1 mL 6 g/L Tetrafin solution    |   |
| Purity of test substance  | Analytical grade                | Accustandard  |
| Measured is what % of nominal?  |                                 |   |
| Toxicity values calculated based on nominal or measured concentrations? | Measured                        | Time-weighted exposure concentration due to degradation |
| Concentration of carrier (if any) in test solutions                     | Acetone                         |   |
| Concentration 1 Nom;Meas ( $\mu\text{g/g C}$ )                          | 0.03; Not reported              | 5 reps; 10/rep  |
| Concentration 2 Nom;Meas ( $\mu\text{g/g C}$ )                          | 0.04; Not reported              |   |
| Concentration 3 Nom;Meas ( $\mu\text{g/g C}$ )                          | 0.07; Not reported              |   |
| Concentration 4 Nom;Meas ( $\mu\text{g/g C}$ )                          | 0.11; Not reported              |   |
| Concentration 5 Nom;Meas ( $\mu\text{g/g C}$ )                          | 0.15; Not reported              |   |
| Concentration 6 Nom;Meas ( $\mu\text{g/g C}$ )                          | 0.18; Not reported              |   |
| Control   | Negative: 0;0<br>Solvent: 0;0   |   |
| LC50 (95% CI) ( $\mu\text{g/g C}$ )                                     | 0.13 (0.12-0.14)                | Method: Probit, logit, Weibull                          |
| EC50 (95% CI) ( $\mu\text{g/g C}$ )                                     | 0.10 (0.08-0.11)                | Immobilization  |
| LOEC ( $\mu\text{g/g C}$ )  | Dry weight/growth rate: 0.2     | Method: Dunnett's test<br>p: 0.05<br>MSD: Not reported  |
| NOEC ( $\mu\text{g/g C}$ )  | Dry weight/growth rate:<br>0.15 | See Figure 2A (next lowest value to reported LOEC)      |
| MATC  | 0.17                            |   |

Notes: raw data not included. Points for water quality not deducted because standard method cited in water quality explanation.

Reliability points taken off for:

Documentation (Table 9): Sediment particle size (1), Sediment TOC (3), Minimum significant difference (2), % control at NOEC/LOEC (2). Total:  $100 - 8 = 92$

Acceptability (Table 10): Spike method (4), Equilibration time (6), Fully evaporated (4), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1). Total:  $100 - 17 = 83$

**Reliability score: Mean (92, 83) = 87.5**

## Sediment Toxicity Data Summary

*C. dilutus*  
Fipronil-sulfone  
MB46136

Maul JD, Brennan AA, Harwood AD and Lydy MJ. (2008) Effect of sediment-associated pyrethroids, fipronil, and metabolites on *Chironomus tentans* growth rate, body mass, condition index, immobilization, and survival. *Environmental Toxicology and Chemistry*, 27(12), 2582-2590.

Relevance  
Score: 100  
Rating: R

Reliability  
Score: 87.5  
Rating: R

Relevance points taken off for: none

|  | <b>Maul 2008</b>   | <i>C. dilutus</i> |
|--|--|-------------------|
| <b>Parameter</b>                             | <b>Value</b>   | <b>Comment</b>    |
| Test method cited                            | U.S. Environmental Protection Agency. 2000. Methods for measuring the toxicity and bioaccumulation of sediment-associated contaminants with freshwater invertebrates, 2 <sup>nd</sup> ed. EPA 600/R-99/064. Guidance Document. Washington, DC. |                   |
| Phylum                                       | Anthropoda   |                   |
| Class  | Insecta  |                   |
| Order  | Diptera  |                   |
| Family                                       | Chironomidae   |                   |
| Genus  | <i>Chironomus</i>  |                   |
| Species                                      | <i>dilutus</i>   |                   |
| Family in North America?                     | Yes  |                   |
| Age/size at start of test/growth phase       | Early to mid-fourth instar   |                   |
| Source of organisms                          | Southern Illinois University   |                   |
| Have organisms been exposed to contaminants? | No   |                   |
| Animals acclimated and disease-free?         | Yes  |                   |
| Animals randomized?                          | Yes  |                   |

|   | <b>Maul 2008</b>                   | <i>C. dilutus</i>                              |
|---|------------------------------------|--|
| <b>Parameter</b>                              | <b>Value</b>                       | <b>Comment</b>                                 |
| Test vessels randomized?                      | Yes                                |  |
| Test duration                                 | 10 d                               |  |
| Effect 1                                      | Survival                           |  |
| Control response 1                            | 95 %                               |  |
| Effect 2                                      | Head capsule width, length         |  |
| Control response 2                            | Not reported                       |  |
| Effect 3                                      | Dry weight                         |  |
| Control response 3                            | Not reported                       |  |
| Effect 4                                      | Growth rate                        |  |
| Control response 4                            | Not reported                       |  |
| Temperature                                   | 23 ± 1 °C                          |  |
| Test type                                     | Static                             |  |
| Photoperiod/light intensity                   | 16l:8d; Not reported               |  |
| Overlying water                               | Moderately hard water              | According to standard method EPA 600/R-99/064. |
| pH  | 6.67-6.96                          |  |
| Hardness                                      | Not reported                       |  |
| Alkalinity                                    | Not reported                       |  |
| Conductivity                                  | 342-399 µS/cm                      |  |
| Dissolved Oxygen                              | 6.40-7.34 mg/L                     |  |
| TOC   | Not reported                       |  |
| DOC   | Not reported                       |  |
| Ammonia-N                                     | Not reported                       |  |
| Chemical analysis?/Method                     | GC                                 |  |
| Sediment source                               | Carbondale, Illinois               |  |
| Organic carbon                                | 0.69 %                             |  |
| Particle size distribution (sand, silt, clay) | Not reported                       |  |
| pH  | Not reported                       |  |
| Percent solids                                | Not reported                       |  |
| Sediment spike procedure                      | Direct addition to sediment slurry |  |
| Carrier solvent addition; evaporated (y/n)    | Acetone; not evaporated            |  |
| Sediment spike equilibration time             | 14 d                               |  |
| Sediment to Solution ratio                    | 50 g (dry weight): 700 mL          |  |
| Sediment extraction/analysis methods          | GC                                 |  |
| Interstitial water monitored?                 | Yes                                |  |
| Interstitial water isolation method           | Centrifuge                         | 3,300 g  |

|   | <b>Maul 2008</b>                | <i>C. dilutus</i>                                       |
|---|---------------------------------|---|
| <b>Parameter</b>  | <b>Value</b>                    | <b>Comment</b>  |
| Interstitial water extraction/analysis method                           | GC                              |   |
| DOC   | Not reported                    |   |
| Feeding   | 1 mL 6 g/L Tetrafin solution    |   |
| Purity of test substance  | Analytical grade                | Accustandard  |
| Measured is what % of nominal?  | Not reported                    |   |
| Toxicity values calculated based on nominal or measured concentrations? | Measured                        | Time-weighted exposure concentration due to degradation |
| Concentration of carrier (if any) in test solutions                     | Acetone                         |   |
| Concentration 1 Nom;Meas ( $\mu\text{g/g C}$ )                          | 0.03; Not reported              | 5 reps; 10/rep  |
| Concentration 2 Nom;Meas ( $\mu\text{g/g C}$ )                          | 0.06; Not reported              |   |
| Concentration 3 Nom;Meas ( $\mu\text{g/g C}$ )                          | 0.1; Not reported               |   |
| Concentration 4 Nom;Meas ( $\mu\text{g/g C}$ )                          | 0.2; Not reported               |   |
| Control   | Negative: 0; 0<br>Solvent: 0; 0 |   |
| LC50 (95% CI) ( $\mu\text{g/g C}$ )                                     | 0.12 (0.14-0.10)                | Method: Probit, logit, Weibull                          |
| EC50 (95% CI) ( $\mu\text{g/g C}$ )                                     | 0.04 (not reported)             | Immobilization  |
| LOEC ( $\mu\text{g/g C}$ )  | Dry weight/growth rate: 0.1     | Method: Dunnett's test<br>p: 0.05<br>MSD: Not reported  |
| NOEC ( $\mu\text{g/g C}$ )  | Dry weight: 0.06                | See Figure 2C (next lowest value to reported LOEC)      |
| MATC  | 0.08                            |   |

Notes: Raw data not included. Points for water quality not deducted because standard method cited in water quality explanation.

Solubility (S) value for fipronil sulfone (MB 46136) = 160  $\mu\text{g/L}$ , 2S = 320  $\mu\text{g/L}$ .

Reliability points taken off for:

Documentation (Table 9): Sediment particle size (1), Sediment TOC (3), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100-8 =92

Acceptability (Table 10): Spike method (4), Equilibration time (6), Fully evaporated (4), Minimum significant difference (1), % control at NOEC (1), % control at LOEC (1). Total: 100-17 =83

**Reliability score: Mean (92, 83)= 87.5**

## Sediment Toxicity Data Summary

*Chironomus dilutus*

Fipronil sulfide

MB 45950

Putt, AE. (2000d) [<sup>14</sup>C]MB 45950 – Toxicity to midge (*Chironomus tentans*) during a 10-day sediment exposure. Springbord Laboratories, Inc., Wareham, Massachusetts. Laboratory project ID 10566.6536. Submitted to Aventis CropScience, Research Triangle Park, North Carolina. US EPA MRID 45084801.

Relevance

Score: 100

Rating: R

Reliability

Score: 94

Rating: R

Relevance points taken off for: none.

| <b>Fipronil-sulfide</b>                      | <b>Putt 2000</b>  | <b><i>C. dilutus</i></b> |
|--|---|--------------------------|
| <b>Parameter</b>                             | <b>Value</b>  | <b>Comment</b>           |
| Test method cited                            | Laboratory protocol that meets USEPA Ecological Effects Test Guidelines OPPTS 850.1735 Whole Sediment Acute Toxicity Invertebrates, Freshwater (1996) and ASTM Guideline E 1706-95b Standard test methods for measuring the toxicity of sediment-associated contaminants with freshwater invertebrates (1997) |                          |
| Phylum                                       | Anthropoda  |                          |
| Class  | Insecta   |                          |
| Order  | Diptera   |                          |
| Family                                       | Chironomidae  |                          |
| Genus  | <i>Chironomus</i>   |                          |
| Species                                      | <i>dilutus</i>  | Formerly <i>tentans</i>  |
| Family in North America?                     | Yes   |                          |
| Age/size at start of test/growth phase       | 8-9 d, 3 <sup>rd</sup> instar larvae  |                          |
| Source of organisms                          | Laboratory cultures   |                          |
| Have organisms been exposed to contaminants? | No  |                          |
| Animals acclimated and disease-              | Yes   |                          |

| <b>Fipronil-sulfide</b>                          | <b>Putt 2000</b>            | <b><i>C. dilutus</i></b>                                   |
|--|-----------------------------|--|
| <b>Parameter</b>                                 | <b>Value</b>                | <b>Comment</b>   |
| free?  |                             |  |
| Animals randomized?                              | Yes                         |  |
| Test vessels randomized?                         | Yes                         |  |
| Test duration                                    | 10 d                        |  |
| Effect 1   | Survival                    |  |
| Control response 1, mean controls                | 89.8 %                      |  |
| Effect 2   | Dry weight                  |  |
| Control response 2, mean controls                | 1.15                        |  |
| Temperature                                      | 23 ± 1°C                    |  |
| Test type  | Static renewal              |  |
| Photoperiod/light intensity                      | 16 l: 8 d; 540-970 lux      |  |
| Overlying water                                  | Well water                  | 175 mL   |
| pH   | 7.1                         |  |
| Hardness   | 36 mg/L CaCO <sub>3</sub>   |  |
| Alkalinity                                       | 33 mg/L CaCO <sub>3</sub>   |  |
| Conductivity                                     | 180 µS/cm                   |  |
| Dissolved Oxygen                                 | >3.4 mg/L                   | >40%   |
| TOC  | Not reported                |  |
| DOC  | Not reported                |  |
| Ammonia-N  | 0.5 mg/L                    | Ave 0,10 d   |
| Chemical analysis?/Method                        | Not reported                |  |
| Sediment source                                  | Natural sediment            | Glen Charlie Pond,<br>Wareham,<br>Massachusetts; 100<br>mL |
| pH   | 4.2                         |  |
| Organic carbon                                   | 2.9 %                       |  |
| Particle size distribution<br>(sand, silt, clay) | 97 % sand, 2% silt, 1% clay |  |
| pH   | 4.2                         |  |
| Percent solids                                   | Not reported                |  |
| Sediment spike procedure                         | Jar rolling                 |  |
| Carrier solvent addition;<br>evaporated (y/n)    | Acetone; yes                |  |
| Sediment spike equilibration<br>time             | 30 d                        |  |
| Sediment to Solution ratio                       | 100:175 mL                  |  |
| Sediment extraction/analysis<br>methods          | Not reported                |  |
| Interstitial water monitored?                    | Yes                         |  |
| Interstitial water isolation<br>method           | Centrifuge                  | 15 min, 3000 rpm   |

| <b>Fipronil-sulfide</b>   | <b>Putt 2000</b>   | <b><i>C. dilutus</i></b>                                    |
|---|--|---|
| <b>Parameter</b>  | <b>Value</b>   | <b>Comment</b>  |
| Interstitial water extraction/analysis method                           | Not reported   |   |
| DOC   | Not reported   |   |
| Feeding   | Flaked fish food suspension (4 mg/L) once daily              |   |
| Purity of test substance  | 98.8 %   |   |
| Measured is what % of nominal?  | 101-108 %  |   |
| Toxicity values calculated based on nominal or measured concentrations? | Measured   |   |
| Concentration of carrier (if any) in test solutions                     | Acetone  |   |
| Concentration 1 Nom;Meas  | 13; 15 (µg/kg)<br>0.45; 0.52 (µg/g OC)                       | 8 reps; 10 larvae/rep                                       |
| Concentration 2 Nom;Meas  | 25; 29 (µg/kg)<br>0.86; 1.00 (µg/g OC)                       |   |
| Concentration 3 Nom;Meas  | 50; 54 (µg/kg)<br>1.72; 1.86 (µg/g OC)                       |   |
| Concentration 4 Nom;Meas  | 100; 100 (µg/kg)<br>3.45; 3.45 (µg/g OC)                     |   |
| Concentration 5 Nom;Meas  | 200; 200 (µg/kg)<br>6.90; 6.90 (µg/g OC)                     |   |
| Control   | Negative: 0; 0<br>Solvent: 0; 0                              |   |
| LC50 (95% CI)   | 140 (130-140) (µg/kg)<br>4.8 (4.5-4.8) (µg/g OC)<br>Survival | Method: Computer program (Gulley 1996)                      |
| EC50 (95% CI)   | 46 (43-47) (µg/kg)<br>1.6 (1.5-1.6) (µg/g OC)<br>Growth      | Method: Computer program (Gulley 1996)                      |
| NOEC  | 29 (µg/kg)<br>1.0 (µg/g OC)                                  | Method: Williams' Test<br>p: 0.01-0.05<br>MSD: Not reported |
| LOEC  | 54 (µg/kg)<br>1.9 (µg/g OC)                                  |   |
| MATC (GeoMean NOEC, LOEC)   | 39.6 (µg/kg)<br>1.4 (µg/g OC)                                |   |
| % of control at NOEC  | 96.1 %<br>Survival   | 86 (tmt) / 89.5 (mean controls) = 96.1 %                    |

| <b>Fipronil-sulfide</b> | <b>Putt 2000</b> | <b><i>C. dilutus</i></b>               |
|-------------------------|------------------|--|
| <b>Parameter</b>        | <b>Value</b>     | <b>Comment</b>                         |
| % of control at LOEC    | 88%<br>Survival  | 79 (tmt) / 89.5 (mean controls) = 88 % |

Notes: Study reported values in ( $\mu\text{g}/\text{kg}$ ) and values were converted to  $\mu\text{g}/\text{g}$  OC using the reported % OC.

Solubility value for fipronil sulfide (MB 45950) not available. Solubility (S) of fipronil parent compound (MB 46030) =  $1650.8 \mu\text{g}/\text{L}$ ,  $2S = 3301.6 \mu\text{g}/\text{L}$ .

Reliability points taken off for:

Documentation (Table 9): Analytical method (4), Minimum significant difference (2). Total:  $100-6=94$

Acceptability (Table 10): Dissolved oxygen (5), Minimum significant difference (1). Total:  $100-6=94$

**Reliability score: Mean (94,94)=94**

## Sediment Toxicity Data Summary

*Chironomus dilutus*

Fipronil-sulfone

MB 43163

Putt, AE. (2000e) [<sup>14</sup>C]MB 43163 – Toxicity to midge (*Chironomus tentans*) during a 10-day sediment exposure. Springbord Laboratories, Inc., Wareham, Massachusetts. Laboratory project ID 10566.6537. Submitted to Aventis CropScience, Research Triangle Park, North Carolina. US EPA MRID 45175901.

Relevance

Score: 100

Rating: R

Reliability

Score: 94

Rating: R

Relevance points taken off for: none.

| <b>Fipronil-sulfone</b>                      | <b>Putt 2000</b>  | <b><i>C. dilutus</i></b> |
|--|---|--------------------------|
| <b>Parameter</b>                             | <b>Value</b>  | <b>Comment</b>           |
| Test method cited                            | Laboratory protocol that meets USEPA Ecological Effects Test Guidelines OPPTS 850.1735 Whole Sediment Acute Toxicity Invertebrates, Freshwater (1996) and ASTM Guideline E 1706-95b Standard test methods for measuring the toxicity of sediment-associated contaminants with freshwater invertebrates (1997) |                          |
| Phylum                                       | Anthropoda  |                          |
| Class  | Insecta   |                          |
| Order  | Diptera   |                          |
| Family                                       | Chironomidae  |                          |
| Genus  | <i>Chironomus</i>   |                          |
| Species                                      | <i>dilutus</i>  | Formerly <i>tentans</i>  |
| Family in North America?                     | Yes   |                          |
| Age/size at start of test/growth phase       | 10 d, 3 <sup>rd</sup> instar larvae   |                          |
| Source of organisms                          | Laboratory cultures   |                          |
| Have organisms been exposed to contaminants? | No  |                          |
| Animals acclimated and disease-              | Yes   |                          |

| <b>Fipronil-sulfone</b>                          | <b>Putt 2000</b>            | <b><i>C. dilutus</i></b>                                   |
|--|-----------------------------|--|
| <b>Parameter</b>                                 | <b>Value</b>                | <b>Comment</b>   |
| free?  |                             |  |
| Animals randomized?                              | Yes                         |  |
| Test vessels randomized?                         | Yes                         |  |
| Test duration                                    | 10 d                        |  |
| Effect 1   | Survival                    |  |
| Control response 1, mean controls                | 96.5 %                      |  |
| Effect 2   | Dry weight                  |  |
| Control response 2, mean controls                | 1.53                        |  |
| Temperature                                      | 22 ± 0.5°C                  |  |
| Test type  | Static renewal              |  |
| Photoperiod/light intensity                      | 16 l: 8 d; 540-970 lux      |  |
| Overlying water                                  | Well water                  | 175 mL   |
| pH   | 6.9                         |  |
| Hardness   | 40 mg/L CaCO <sub>3</sub>   |  |
| Alkalinity                                       | 27 mg/L CaCO <sub>3</sub>   |  |
| Conductivity                                     | 180 µS/cm                   |  |
| Dissolved Oxygen                                 | >3.4 mg/L                   | >40%   |
| TOC  | Not reported                |  |
| DOC  | Not reported                |  |
| Ammonia-N  | 0.49 mg/L                   | Ave 0,10 d   |
| Chemical analysis?/Method                        | Not reported                |  |
| Sediment source                                  | Natural sediment            | Glen Charlie Pond,<br>Wareham,<br>Massachusetts; 100<br>mL |
| pH   | 4.2                         |  |
| Organic carbon                                   | 2.9 %                       |  |
| Particle size distribution<br>(sand, silt, clay) | 97 % sand, 2% silt, 1% clay |  |
| pH   | 4.2                         |  |
| Percent solids                                   | Not reported                |  |
| Sediment spike procedure                         | Jar rolling                 |  |
| Carrier solvent addition;<br>evaporated (y/n)    | Acetone; yes                |  |
| Sediment spike equilibration<br>time             | 30 d                        |  |
| Sediment to Solution ratio                       | 100:175 mL                  |  |
| Sediment extraction/analysis<br>methods          | Not reported                |  |
| Interstitial water monitored?                    | Yes                         |  |
| Interstitial water isolation<br>method           | Centrifuge                  | 15 min, 3000 rpm   |

| <b>Fipronil-sulfone</b>   | <b>Putt 2000</b>  | <b><i>C. dilutus</i></b>                                    |
|---|---|---|
| <b>Parameter</b>  | <b>Value</b>  | <b>Comment</b>  |
| Interstitial water extraction/analysis method                           | Not reported  |   |
| DOC   | Not reported  |   |
| Feeding   | Flaked fish food suspension (4 mg/L) once daily           |   |
| Purity of test substance  | 99.7 %  |   |
| Measured is what % of nominal?  | 56-70 %   |   |
| Toxicity values calculated based on nominal or measured concentrations? | Measured  |   |
| Concentration of carrier (if any) in test solutions                     | Acetone   |   |
| Concentration 1 Nom;Meas (µg/kg)  | 13; 9.1 (µg/kg)<br>0.45; 0.52 (µg/g OC)                   | 8 reps; 10 larvae/rep                                       |
| Concentration 2 Nom;Meas (µg/kg)  | 25; 14(µg/kg)<br>0.86; 1.00 (µg/g OC)                     |   |
| Concentration 3 Nom;Meas (µg/kg)  | 50; 33 (µg/kg)<br>1.72; 1.14 (µg/g OC)                    |   |
| Concentration 4 Nom;Meas (µg/kg)  | 100; 69 (µg/kg)<br>3.45; 2.38 (µg/g OC)                   |   |
| Concentration 5 Nom;Meas (µg/kg)  | 200; 140 (µg/kg)<br>6.90; 4.83 (µg/g OC)                  |   |
| Control   | Negative: 0; 0<br>Solvent: 0; 0                           |   |
| LC50 (95% CI) (µg/kg)   | 43 (35-49) (µg/kg)<br>1.5 (1.2-1.7) (µg/g OC)<br>Survival | Method: Computer program (Gulley 1996)                      |
| EC50 (95% CI) (µg/kg)   | 47 (43-50) (µg/kg)<br>1.6 (1.5-1.7) (µg/g OC)<br>Growth   | Method: Computer program (Gulley 1996)                      |
| NOEC (µg/kg)  | 9.1 (µg/kg)<br>0.31 (µg/g OC)                             | Method: Williams' Test<br>p: 0.01-0.05<br>MSD: Not reported |
| LOEC (µg/kg)  | 14 (µg/kg)<br>0.48 (µg/g OC)                              |   |
| MATC (GeoMean NOEC, LOEC)   | 11.3 (µg/kg)<br>0.39 (µg/g OC)                            |   |
| % of control at NOEC  | 97 %<br>Survival  | 94 (tmt) / 96.5 (mean controls) = 97 %                      |
| % of control at LOEC  | 97%<br>Survival   | 94 (tmt) / 96.5 (mean controls) = 97 %                      |

Notes: Study reported values in ( $\mu\text{g}/\text{kg}$ ) and values were converted to  $\mu\text{g}/\text{g}$  OC using the reported % OC.

Solubility (S) value for fipronil sulfone (MB 46136) =  $160 \mu\text{g}/\text{L}$ ,  $2S = 320 \mu\text{g}/\text{L}$ .

Reliability points taken off for:

Documentation (Table 9): Analytical method (4), Minimum significant difference (2). Total:  $100 - 5 = 94$

Acceptability (Table 10): Dissolved oxygen (5), Minimum significant difference (1). Total:  $100 - 6 = 94$

**Reliability score: Mean (94,94)=94**

## Sediment Toxicity Data Summary

*Chironomus dilutus*  
Fipronil desulfinyl  
MB 46513

Putt, AE. (2001) [<sup>14</sup>C]MB 46513 – Toxicity to midge (*Chironomus tentans*) during a 10-day sediment exposure. Springbord Laboratories, Inc., Wareham, Massachusetts. Laboratory project ID 10566.6538. Submitted to Aventis CropScience, Research Triangle Park, North Carolina. US EPA MRID 45375901.

Relevance  
Score: 100  
Rating: R

Reliability  
Score: 94  
Rating: R

Relevance points taken off for: none.

| Fipronil-desulfinyl                          | <b>Putt 2001</b>  | <i>C. dilutus</i>       |
|--|---|-------------------------|
| <b>Parameter</b>                             | <b>Value</b>  | <b>Comment</b>          |
| Test method cited                            | Laboratory protocol that meets USEPA Ecological Effects Test Guidelines OPPTS 850.1735 Whole Sediment Acute Toxicity Invertebrates, Freshwater (1996) and ASTM Guideline E 1706-95b Standard test methods for measuring the toxicity of sediment-associated contaminants with freshwater invertebrates (1997) |                         |
| Phylum                                       | Anthropoda  |                         |
| Class  | Insecta   |                         |
| Order  | Diptera   |                         |
| Family                                       | Chironomidae  |                         |
| Genus  | <i>Chironomus</i>   |                         |
| Species                                      | <i>dilutus</i>  | Formerly <i>tentans</i> |
| Family in North America?                     | Yes   |                         |
| Age/size at start of test/growth phase       | 9 d, 3 <sup>rd</sup> instar larvae  |                         |
| Source of organisms                          | Laboratory cultures   |                         |
| Have organisms been exposed to contaminants? | No  |                         |
| Animals acclimated and disease-              | Yes   |                         |

| Fipronil-desulfinyl                           | Putt 2001                     | <i>C. dilutus</i>                                 |
|---|-------------------------------|---|
| Parameter                                     | Value                         | Comment   |
| free?   |                               |   |
| Animals randomized?                           | Yes                           |   |
| Test vessels randomized?                      | Yes                           |   |
| Test duration                                 | 10 d                          |   |
| Effect 1                                      | Survival                      |   |
| Control response 1, mean controls             | 100 %                         |   |
| Effect 2                                      | Growth (Dry weight)           |   |
| Control response 2, mean controls             | 1.24                          |   |
| Temperature                                   | 22 ± 0.5°C                    |   |
| Test type                                     | Static renewal                |   |
| Photoperiod/light intensity                   | 16 l: 8 d; 650-1100 lux       |   |
| Overlying water                               | Well water                    | 175 mL  |
| pH  | 7.6                           |   |
| Hardness                                      | 40-46 mg/L CaCO <sub>3</sub>  |   |
| Alkalinity                                    | 34 mg/L CaCO <sub>3</sub>     |   |
| Conductivity                                  | 150-160 µS/cm                 |   |
| Dissolved Oxygen                              | 7.6-8.5                       | 89-99%  |
| TOC   | Not reported                  |   |
| DOC   | Not reported                  |   |
| Ammonia-N                                     | 0.17 mg/L                     | Ave 0,10 d  |
| Chemical analysis?/Method                     | Liquid scintillation counting |   |
| Sediment source                               | Natural sediment              | Glen Charlie Pond, Wareham, Massachusetts; 100 mL |
| pH  | 4.5                           |   |
| Organic carbon                                | 2.3 %                         |   |
| Particle size distribution (sand, silt, clay) | 94 % sand, 6% silt, 0% clay   |   |
| Percent solids                                | Not reported                  |   |
| Sediment spike procedure                      | Jar rolling                   |   |
| Carrier solvent addition; evaporated (y/n)    | Acetone; yes                  |   |
| Sediment spike equilibration time             | 30 d                          |   |
| Sediment to Solution ratio                    | 100:175 mL                    |   |
| Sediment extraction/analysis methods          | Liquid scintillation counting |   |
| Interstitial water monitored?                 | Yes                           |   |
| Interstitial water isolation method           | Centrifuge                    | 15 min, 3000 rpm                                  |

| Fipronil-desulfinyl   | Putt 2001   | <i>C. dilutus</i>  |
|---|---|--|
| Parameter   | Value   | Comment  |
| Interstitial water extraction/analysis method                           | Liquid scintillation counting                           |  |
| DOC   | Not reported  |  |
| Feeding   | Flaked fish food suspension (4 mg/L) once daily         |  |
| Purity of test substance  | 97.8 %  |  |
| Measured is what % of nominal?  | 56-70 %   |  |
| Toxicity values calculated based on nominal or measured concentrations? | Measured  |  |
| Concentration of carrier (if any) in test solutions                     | Acetone   |  |
| Concentration 1 Nom;Meas (µg/kg)  | 200; 200 (µg/kg)<br>8.70; 8.70 (µg/g OC)                | 8 reps; 10 larvae/rep  |
| Concentration 2 Nom;Meas (µg/kg)  | 400; 380 (µg/kg)<br>17.39; 16.52 (µg/g OC)              |  |
| Concentration 3 Nom;Meas (µg/kg)  | 800; 790 (µg/kg)<br>34.78; 34.35 (µg/g OC)              |  |
| Concentration 4 Nom;Meas (µg/kg)  | 1600; 1500 (µg/kg)<br>69.57; 65.22 (µg/g OC)            |  |
| Concentration 5 Nom;Meas (µg/kg)  | 3200; 3200 (µg/kg)<br>139.13; 139.13 (µg/g OC)          |  |
| Control   | Negative: 0; 0<br>Solvent: 0; 0                         |  |
| LC50 (95% CI) (µg/kg)   | 1300 (710-2300) (µg/kg)<br>57 (31-100) (µg/g OC)        | Method: Dunnett's test   |
| EC50 (95% CI) (µg/kg)   | Growth<br>640 (560-720) (µg/kg)<br>28 (24-31) (µg/g OC) | Method: Steel's Many-One Rank test   |
| NOEC (µg/kg)  | Survival<br>160 (µg/kg)<br>6.96 (µg/g OC)               | Method: Steel's Many-One Rank test (growth)<br>p: 0.01-0.05<br>MSD: Not reported |
| LOEC (µg/kg)  | Survival<br>200 (µg/kg)<br>8.70 (µg/g OC)               | Method: Dunnett's test (survival)  |

| Fipronil-desulfinyl       | Putt 2001                                 | <i>C. dilutus</i>                      |
|---------------------------|---|--|
| Parameter                 | Value                                     | Comment                                |
| MATC (GeoMean NOEC, LOEC) | Survival<br>179 (µg/kg)<br>7.78 (µg/g OC) |  |
| % of control at NOEC      | 92 %<br>Survival                          | 84 (tmt) / 91.5 (mean controls) = 92 % |
| % of control at LOEC      | 100 %<br>Survival                         | 95 (tmt) / 95 (mean controls) = 100 %  |

Notes: Two identical definitive tests performed. Toxicity values established combination of tests (NOEC from first test, others from second test).

Study reported values in (µg/kg) and values were converted to µg/g OC using the reported % OC.

Solubility (S) value for fipronil desulfinyl (MB 46513) = 950 µg/L, 2S = 1900 µg/L.

Reliability points taken off for:

Documentation (Table 9): Analytical method (4), Minimum significant difference (2). Total: 100-6 =94

Acceptability (Table 10): Dissolved oxygen (5), Minimum significant difference (1). Total: 100-6 =94

**Reliability score: Mean (94,94)=94**

## Sediment Toxicity Data Summary

*Chironomus dilutus*

Fipronil

MB 46030

Putt, AE. (2003d) Fipronil-Toxicity to midge (*Chironomus tentans*) during a 10-day sediment exposure. Springborn Laboratories, Inc., Wareham, Massachusetts. Laboratory project ID 13798.6106. Submitted to Bayer CropScience, Research Triangle Park, North Carolina. US EPA MRID 45878001.

Relevance

Score: 100

Rating: R

Reliability

Score: 92

Rating: R

Relevance points taken off for: none.

| Fipronil                               | <b>Putt 2003</b>  | <i>C. dilutus</i>       |
|--|---|-------------------------|
| <b>Parameter</b>                       | <b>Value</b>  | <b>Comment</b>          |
| Test method cited                      | Laboratory protocol that meets USEPA Ecological Effects Test Guidelines OPPTS 850.1735 Whole Sediment Acute Toxicity Invertebrates, Freshwater (1996) and ASTM Guideline E 1706-95b Standard test methods for measuring the toxicity of sediment-associated contaminants with freshwater invertebrates (1997) |                         |
| Phylum                                 | Anthropoda  |                         |
| Class                                  | Insecta   |                         |
| Order                                  | Diptera   |                         |
| Family                                 | Chironomidae  |                         |
| Genus                                  | <i>Chironomus</i>   |                         |
| Species                                | <i>dilutus</i>  | Formerly <i>tentans</i> |
| Family in North America?               | Yes   |                         |
| Age/size at start of test/growth phase | 10 d, 3 <sup>rd</sup> instar larvae   |                         |
| Source of organisms                    | Laboratory cultures   |                         |
| Have organisms been exposed to         | No  |                         |

| Fipronil                                      | Putt 2003                   | <i>C. dilutus</i>                                 |
|---|-----------------------------|---|
| Parameter                                     | Value                       | Comment   |
| contaminants?                                 |                             |   |
| Animals acclimated and disease-free?          | Yes                         |   |
| Animals randomized?                           | Yes                         |   |
| Test vessels randomized?                      | Yes                         |   |
| Test duration                                 | 10 d                        |   |
| Effect 1                                      | Survival                    |   |
| Control response 1, mean controls             | Not reported                |   |
| Effect 2                                      | Growth                      |   |
| Control response 2, mean controls             | Not reported                |   |
| Temperature                                   | 23 ± 2°C                    |   |
| Test type                                     | Static renewal              |   |
| Photoperiod/light intensity                   | 16 l: 8 d; 538-861 lux      |   |
| Overlying water                               | Well water                  | 175 mL  |
| pH  | 7.55                        |   |
| Hardness                                      | 52 mg/L CaCO <sub>3</sub>   |   |
| Alkalinity                                    | 36 mg/L CaCO <sub>3</sub>   |   |
| Conductivity                                  | 180-190 µS/cm               |   |
| Dissolved Oxygen                              | >3.4 mg/L                   | >40%  |
| TOC   | Not reported                |   |
| DOC   | Not reported                |   |
| Ammonia-N                                     | 0.49 mg/L                   |   |
| Chemical analysis?/Method                     | Not reported                |   |
| Sediment source                               | Natural sediment            | Glen Charlie Pond, Wareham, Massachusetts; 100 mL |
| pH  | 5.7                         |   |
| Organic carbon                                | 2.8 %                       |   |
| Particle size distribution (sand, silt, clay) | 94 % sand, 6% silt, 0% clay |   |
| Percent solids                                | Not reported                |   |
| Sediment spike procedure                      | Jar rolling                 |   |
| Carrier solvent addition; evaporated (y/n)    | Acetone; yes                |   |
| Sediment spike equilibration time             | 7 d                         |   |
| Sediment to Solution ratio                    | 100:175 mL                  |   |
| Sediment extraction/analysis methods          | Not reported                |   |
| Interstitial water monitored?                 | Yes                         |   |
| Interstitial water isolation method           | Centrifuge                  | 30 min, 2000 rpm                                  |

| Fipronil  | Putt 2003   | <i>C. dilutus</i>                      |
|---|---|--|
| Parameter   | Value   | Comment                                |
| Interstitial water extraction/analysis method                           | Liquid scintillation counting                         |  |
| DOC   | Not reported  |  |
| Feeding   | Flaked fish food suspension (4 mg/mL) once daily      |  |
| Purity of test substance  | 98.3 %  |  |
| Measured is what % of nominal?  | 120-150 %   |  |
| Toxicity values calculated based on nominal or measured concentrations? | Measured  |  |
| Concentration of carrier (if any) in test solutions                     | Acetone   |  |
| Concentration 1 Nom;Meas  | Sediment: 6.3; 7.6 (µg/kg)<br>0.23; 0.27 (µg/g OC)    | 8 reps; 10 larvae/rep                  |
| Concentration 2 Nom;Meas  | 13; 16 (µg/kg)<br>0.46; 0.57 (µg/g OC)                |  |
| Concentration 3 Nom;Meas  | 25; 33 (µg/kg)<br>0.89; 1.18 (µg/g OC)                |  |
| Concentration 4 Nom;Meas  | 50; 68 (µg/kg)<br>1.79; 2.43 (µg/g OC)                |  |
| Concentration 5 Nom;Meas  | 100; 140 (µg/kg)<br>3.57; 5.00 (µg/g OC)              |  |
| Concentration 6 Nom;Meas  | 200; 290 (µg/kg)<br>7.14; 10.36 (µg/g OC)             |  |
| Control   | Negative: 0; 0<br>Solvent: 0; 0                       |  |
| LC50 (95% CI)   | 30 (28-32) (µg/kg)<br>1.1 (1-1.1) (µg/g OC)           | Method: Williams' Test                 |
| EC50 (95% CI)   | Growth: 50 (48-51) (µg/kg)<br>1.8 (1.7-1.8) (µg/g OC) | Method: Williams' Test                 |
| NOEC  | Survival:<br>16 (µg/kg)                               | Method: Williams' test<br>p: 0.01-0.05 |

| Fipronil                  | Putt 2003   | <i>C. dilutus</i>  |
|---------------------------|---|--|
| Parameter                 | Value   | Comment  |
|                           | 0.57 (µg/g OC)<br><br>Growth:<br>33 (µg/kg)<br>1.2 (µg/g OC)  | MSD: Not reported  |
| LOEC                      | Survival:<br>33 (µg/kg)<br>1.2 (µg/g OC)<br><br>Growth: 68 (µg/kg)<br>2.43 (µg/g OC)                        |  |
| MATC (GeoMean NOEC, LOEC) | Survival:<br>23 (µg/kg)<br>0.82 (µg/g OC)<br><br>Growth:<br>47 (µg/kg)<br>1.7 (µg/g OC)                     |  |
| % of control at NOEC      | Sediment:<br>Survival: 98 %<br>Growth: 41 %<br><br>Pore water:<br>Survival: 94 %<br>Growth: 105 %           | Sediment:<br>Survival: 94 (tmt) / 96<br>(mean controls) = 98 %<br>Growth: 39(tmt) / 96<br>(mean controls) = 41 %<br><br>Pore water:<br>Survival: 1.33 (tmt) /<br>1.41 (mean controls) =<br>94 %<br>Growth: 1.48 (tmt) /<br>1.41 (mean controls) =<br>105 % |
| % of control at LOEC      | Sediment:<br>Survival: 40 %<br>Growth: 71 %<br><br>Pore water:<br>Survival: 105 %<br>Growth: not calculable | Sediment:<br>Survival: 39 (tmt) / 96<br>(mean controls) = 40 %<br>Growth: 1.0 (tmt) / 96<br>(mean controls) = 71 %   |

| Fipronil  | Putt 2003 | <i>C. dilutus</i>   |
|-----------|-----------|---|
| Parameter | Value     | Comment   |
|           |           | Pore water:<br>Survival: 1.48 (tmt) /<br>1.41 (mean controls) =<br>105 %<br>Growth : not calculable |

Notes: Study reported values in ( $\mu\text{g}/\text{kg}$ ) and values were converted to  $\mu\text{g}/\text{g}$  OC using the reported % OC.

Reliability points taken off for:

Documentation (Table 9): Equilibration time (4), Minimum significant difference (2). Total: 100-5 =94

Acceptability (Table 10): Equilibration time (6), Temperature variation (3), Minimum significant difference (1). Total: 100-6 =90

**Reliability score: Mean (94,90)=92**

## Sediment Toxicity Data Summary

*Chironomus riparius*  
Fipronil sulfide  
MB 45950

Kolk, J. (2002) Chronic toxicity test with midge larvae (*Chironomus riparius*) in a water/sediment system. Springborn Laboratories (Europe), Horn, Switzerland. Laboratory ID 1067.006.173. Submitted to Bayer CropScience, Research Triangle Park, North Carolina. US EPA MRID 45851001.

Relevance  
Score: 100  
Rating: R

Reliability  
Score: 86  
Rating: R

Relevance points taken off for: none.

| Fipronil-sulfide                             | <b>Kolk 2002</b>   | <i>C. riparius</i>      |
|--|--|-------------------------|
| <b>Parameter</b>                             | <b>Value</b>   | <b>Comment</b>          |
| Test method cited                            | Laboratory protocol based on OECD Guidelines for testing of chemicals, proposal for a new guideline 218, sediment-water chironomid toxicity test using spike sediment (2001) |                         |
| Phylum                                       | Anthropoda   |                         |
| Class  | Insecta  |                         |
| Order  | Diptera  |                         |
| Family                                       | Chironomidae   |                         |
| Genus  | <i>Chironomus</i>  |                         |
| Species                                      | <i>riparius</i>  | Formerly <i>tentans</i> |
| Family in North America?                     | Yes  |                         |
| Age/size at start of test/growth phase       | 2-3 d, 1 <sup>st</sup> instar larvae   |                         |
| Source of organisms                          | Aquatic Research Organisms, Hampton, USA   |                         |
| Have organisms been exposed to contaminants? | No   |                         |
| Animals acclimated and disease-free?         | Yes  |                         |
| Animals randomized?                          | Yes  |                         |
| Test vessels randomized?                     | Not reported   |                         |
| Test duration                                | 28 d   |                         |
| Effect 1                                     | Cumulative emergence   |                         |

|   |   |   |
|---|---|---|
| Fipronil-sulfide                              | <b>Kolk 2002</b>  | <i>C. riparius</i>  |
| <b>Parameter</b>                              | <b>Value</b>  | <b>Comment</b>  |
| Control response 1, mean controls             | 85 %  | Table 7   |
| Effect 2                                      | Development rate  |   |
| Control response 2, mean controls             | 0.0725  |   |
| Temperature                                   | 20.5 ± 1°C  |   |
| Test type                                     | Static  |   |
| Photoperiod/light intensity                   | 16 l: 8 d; 658-982 lux  |   |
| Overlying water                               | Deionozed, reconstituted well water                               |   |
| pH  | 7.86  |   |
| Hardness                                      | 164 mg/L CaCO <sub>3</sub>  |   |
| Alkalinity                                    | 28 mg/L CaCO <sub>3</sub>   |   |
| Conductivity                                  | 490 µS/cm   |   |
| Dissolved Oxygen                              | >5 mg/L   | > 60%   |
| TOC   | Not reported  |   |
| DOC   | Not reported  |   |
| Ammonia-N                                     | Not reported  |   |
| Chemical analysis?/Method                     | Liquid scintillation counting                                     |   |
| Sediment source                               | Artificial sediment   | OECD guideline 219 (2001)                                   |
| pH  | 6.7   |   |
| Organic carbon                                | 2.18 %  |   |
| Particle size distribution (sand, silt, clay) | 72.8 % industrial sand, 19.5 kaolin clay, 7.7% sphagnum peat moss |   |
| Percent solids                                | Not reported  |   |
| Sediment spike procedure                      | Jar rolling   |   |
| Carrier solvent addition; evaporated (y/n)    | Acetone; yes  |   |
| Sediment spike equilibration time             | 10 d  |   |
| Sediment to Solution ratio                    | 75:226 mL   | Sediment: h = 1.5 cm, radius = 4 cm<br>Solution: h = 4.5 cm |
| Sediment extraction/analysis methods          | Liquid scintillation counting                                     |   |
| Interstitial water monitored?                 | Yes   |   |
| Interstitial water isolation method           | Not reported  |   |
| Interstitial water extraction/analysis method | Liquid scintillation counting                                     |   |
| DOC   | Not reported  |   |
| Feeding                                       | Tetramin suspension (0-0.3  |   |

|   |   |                        |
|---|---|------------------------|
| Fipronil-sulfide  | <b>Kolk 2002</b>  | <i>C. riparius</i>     |
| <b>Parameter</b>  | <b>Value</b>  | <b>Comment</b>         |
|   | mL/vessel/day)  |                        |
| Purity of test substance  | 99.5 %  | Radio purity           |
| Measured is what % of nominal?  | Not reported  |                        |
| Toxicity values calculated based on nominal or measured concentrations? | Nominal   |                        |
| Concentration of carrier (if any) in test solutions                     | Acetone   |                        |
| Concentration 1 Nom;Meas  | 0.16; ( $\mu\text{g}/\text{kg}$ )<br>0.01; not reported ( $\mu\text{g}/\text{g}$ OC)  | 4 reps; 16 larvae/rep  |
| Concentration 2 Nom;Meas  | 0.31; not reported ( $\mu\text{g}/\text{kg}$ )<br>0.01; not reported ( $\mu\text{g}/\text{g}$ OC)                           |                        |
| Concentration 3 Nom;Meas  | 0.63; not reported ( $\mu\text{g}/\text{kg}$ )<br>0.03; not reported ( $\mu\text{g}/\text{g}$ OC)                           |                        |
| Concentration 4 Nom;Meas  | 1.3; not reported ( $\mu\text{g}/\text{kg}$ )<br>0.06; not reported ( $\mu\text{g}/\text{g}$ OC)                            |                        |
| Concentration 5 Nom;Meas  | 2.5; not reported ( $\mu\text{g}/\text{kg}$ )<br>0.11; not reported ( $\mu\text{g}/\text{g}$ OC)                            |                        |
| Concentration 6 Nom;Meas  | 5.0; not reported ( $\mu\text{g}/\text{kg}$ )<br>0.23; not reported ( $\mu\text{g}/\text{g}$ OC)                            |                        |
| Concentration 7 Nom;Meas  | 10.0; not reported ( $\mu\text{g}/\text{kg}$ )<br>0.46; not reported ( $\mu\text{g}/\text{g}$ OC)                           |                        |
| Control   | Negative: 0; not reported<br>Solvent: 0; not reported   |                        |
| EC50 (95% CI) ( $\mu\text{g}/\text{kg}$ )                               | 3.8 (2.5-5.0) ( $\mu\text{g}/\text{kg}$ )<br>0.17 (0.11-0.23) ( $\mu\text{g}/\text{g}$ OC)<br><br>Midges that did not hatch | Method: Dunnett's test |
| NOEC ( $\mu\text{g}/\text{kg}$ )  | 2.5 ( $\mu\text{g}/\text{kg}$ )   | Method: Dunnett's test |

| Fipronil-sulfide          | <b>Kolk 2002</b>                            | <i>C. riparius</i>                        |
|---------------------------|---|---|
| <b>Parameter</b>          | <b>Value</b>                                | <b>Comment</b>                            |
|                           | 0.11 (µg/g OC)<br>Emergence and development | p: 0.05<br>MSD: Not reported              |
| LOEC (µg/kg)              | 5.0 (µg/kg)<br>0.23 (µg/g OC)               | Method: Dunnett's test                    |
| MATC (GeoMean NOEC, LOEC) | 3.8 (µg/kg)<br>0.16 (µg/g OC)               |   |
| % of control at NOEC      | 104 %<br>Emergence rate                     | 0.88 (tmt) / 0.85 (mean controls) = 104 % |
| % of control at LOEC      | 15 %<br>Survival                            | 2 (tmt) / 13.6 (mean controls) = 15 %     |

Notes: Study reported values in (µg/kg) and values were converted to µg/g OC using the reported % OC.

Solubility value for fipronil sulfide (MB 45950) not available. Solubility (S) of fipronil parent compound (MB 46030) = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation (Table 9): Measured concentrations of interstitial water/sediment (10), Sediment TOC (3), Minimum significant difference (2). Total: 100-15 =85

Acceptability (Table 10): Measured concentrations within 20% nominal (4), Equilibration time (6), Random design (2), Minimum significant difference (1). Total: 100-13 =87

**Reliability score: Mean (85,87)=86**

## Sediment Toxicity Data Summary

*Hyalella azteca*

Fipronil

MB 46030

Picard CR (2015h) 10-day toxicity test exposing freshwater amphipods (*Hyalella azteca*) to fipronil applied to sediment under static-renewal conditions following OCSPP Draft Guideline 850.1735. Smithers Viscent, Wareham, Massachusetts. Laboratory project ID13798.6350. Submitted to Bayer CropScience, Research Triangle Park, North Carolina. CA DPR study ID: 283829.

Relevance

Score: 100

Rating: R

Reliability

Score: 96

Rating: R

Relevance points taken off for: none.

| Fipronil                                     | Picard 2015h   | <i>H. azteca</i> |
|--|--|------------------|
| Parameter                                    | Value  | Comment          |
| Test method cited                            | Laboratory protocols that meet US EPA's Ecological Effects Test Guideline (Draft) OCSPP 850.1735 Whole Sediment Acute Toxicity Invertebrates, Freshwater |                  |
| Phylum                                       | Arthropoda   |                  |
| Class  | Crustacea  |                  |
| Order  | Malacostraca   |                  |
| Family                                       | Hyalellidae  |                  |
| Genus  | <i>Hyalella</i>  |                  |
| Species                                      | <i>Azteca</i>  |                  |
| Family in North America?                     | Yes  |                  |
| Age/size at start of test/growth phase       | 8 d  |                  |
| Source of organisms                          | Laboratory cultures  |                  |
| Have organisms been exposed to contaminants? | No   |                  |
| Animals acclimated and disease-free?         | 48 h   |                  |
| Animals randomized?                          | Yes  |                  |
| Test vessels randomized?                     | Yes  |                  |
| Test duration                                | 10 d   |                  |
| Effect 1                                     | Survival   |                  |
| Control response 1                           | 98.8 %   |                  |

|   |  |   |
|---|--|---|
| Fipronil  | <b>Picard 2015h</b>                      | <i>H. azteca</i>                                |
| <b>Parameter</b>  | <b>Value</b>                             | <b>Comment</b>                                  |
| Effect 2  | Dry weight                               |   |
| Control response 2  | 0.17 mg                                  |   |
| Temperature   | 23 ± 2 °C                                |   |
| Test type   | Static renewal                           |   |
| Photoperiod/light intensity   | 16 l: 8 d; 200-780 lux                   |   |
| Overlying water   | Well water                               |   |
| pH  | 7.0-7.1                                  |   |
| Hardness  | 50-56 mg/L CaCO <sub>3</sub>             |   |
| Alkalinity  | 20 mg/L CaCO <sub>3</sub>                |   |
| Conductivity  | 310-450 µS/cm                            |   |
| Dissolved Oxygen  | 3.2-6.7 mg/L                             | 37-78 %   |
| TOC   | Not reported                             |   |
| DOC   | Not reported                             |   |
| Ammonia-N   | 0.68 mg/L                                |   |
| Chemical analysis?/Method   | LC/MS/MS                                 |   |
| Sediment source   | Natural sediment                         | Glen Charlie Pond,<br>Wareham,<br>Massachusetts |
| Organic carbon  | 2.7 %                                    |   |
| Particle size distribution (sand, silt, clay)                           | 93 % sand, 5 % silt, 2 % clay            |   |
| pH  | 5.8                                      |   |
| Percent solids  | 48.48 %                                  |   |
| Sediment spike procedure  | Jar rolling                              |   |
| Carrier solvent addition; evaporated (y/n)                              | Acetone; yes                             |   |
| Sediment spike equilibration time                                       | 28 d                                     |   |
| Sediment to Solution ratio  | 100:175 mL                               |   |
| Sediment extraction/analysis methods                                    | LC/MS/MS                                 |   |
| Interstitial water monitored?   | Yes                                      |   |
| Interstitial water isolation method                                     | Centrifuge                               | 30 min; 10,000 g                                |
| Interstitial water extraction/analysis method                           | LC/MS/MS                                 |   |
| DOC   | Not reported                             |   |
| Feeding   | 1.5 mL flaked fish food suspension daily |   |
| Purity of test substance  | 93.1 %                                   |   |
| Measured is what % of nominal?  | Not reported                             |   |
| Toxicity values calculated based on nominal or measured concentrations? | Measured                                 |   |
| Concentration of carrier (if any) in                                    | Acetone                                  |   |

| Fipronil                         | Picard 2015h   | <i>H. azteca</i>   |
|----------------------------------|--|--|
| Parameter                        | Value  | Comment  |
| test solutions                   |  |  |
| Concentration 1 Nom;Meas (µg/kg) | 50; 22 (µg/kg)<br>1.85; 0.81 (µg/g OC)   | 8 reps; 10/rep   |
| Concentration 2 Nom;Meas (µg/kg) | 100; 46 (µg/kg)<br>3.70; 1.70 (µg/g OC)  |  |
| Concentration 3 Nom;Meas (µg/kg) | 200; 82 (µg/kg)<br>7.41; 3.04 (µg/g OC)  |  |
| Concentration 4 Nom;Meas (µg/kg) | 400; 180 (µg/kg)<br>14.81; 6.67 (µg/g OC)  |  |
| Concentration 5 Nom;Meas (µg/kg) | 800; 410 (µg/kg)<br>29.63; 15.19 (µg/g OC)   |  |
| Control                          | Negative: 0; 0<br>Solvent: 0; 0  |  |
| LC50 (95% CI) (µg/kg)            | 360 (310-410) (µg/kg)<br>13 (11-15) (µg/g OC)                                      | Method: CETIS program  |
| EC50 (95% CI) (µg/kg)            | Growth: >410 (µg/kg)<br>>15.19 (µg/g OC)   | Method: CETIS program  |
| NOEC (µg/kg)                     | Survival: 180 (µg/kg)<br>6.67 (µg/g OC)<br>Growth: 46 (µg/kg)<br>1.70 (µg/g OC)    | Method: Dunnett's Multiple Comparison and Bonferroni's Adjusted t Test (growth); Wilcoxon's Test with Bonferroni's Adjustment (survival)<br>p: Not reported<br>MSD: Not reported |
| LOEC (µg/kg)                     | Survival: 410 (µg/kg)<br>15.19 (µg/g OC)<br>Growth: 82 (µg/kg)<br>3.04 (µg/g OC)   |  |
| MATC (GeoMean NOEC, LOEC)        | Survival: 271.66 (µg/kg)<br>10.06 (µg/g OC)<br>Growth: 516 (µg/kg)<br>19 (µg/g OC) |  |
| % of control at NOEC             | Survival: 99 %<br>Growth: 88 %   | Survival (180): 97.8 (tmt) / 98.8 (mean controls) = 99 %<br><br>Growth (46): 0.15 (tmt) / 0.17 (mean controls) = 88 %  |
| % of control at LOEC             | Survival: 40 %   | Survival (410): 40   |

| Fipronil  | Picard 2015h | <i>H. azteca</i>   |
|-----------|--------------|--|
| Parameter | Value        | Comment  |
|           | Growth: 88 % | (tmt) / 98.8 (mean controls) = 40 %<br><br>Growth (82): 0.15 (tmt) / 0.17 (mean controls) = 88 |

Notes: Study reported values in ( $\mu\text{g}/\text{kg}$ ) and values were converted to  $\mu\text{g}/\text{g}$  OC using the reported % OC.

Solubility (S) of fipronil (MB 46030) = 1650.8 ( $\mu\text{g}/\text{L}$ , 2S = 3301.6 ( $\mu\text{g}/\text{L}$ .

Reliability points taken off for:

Documentation (Table 9): Significance level (2), Minimum significant difference (2). Total: 100-4=96

Acceptability (Table 10): Temperature variation (3), Minimum significant difference (1). Total: 100-4=96

**Reliability score: Mean (96,96)=96**

## Sediment Toxicity Data Summary

*Hyalella azteca*  
 Fipronil sulfide  
 MB 45950

Picard CR (2015a) 10-day toxicity test exposing freshwater amphipods (*Hyalella azteca*) to fipronil sulfide (MB45950) applied to sediment under static-renewal conditions following OPPTS Draft Guideline 850.1735. Springborn Viscent, Wareham, Massachusetts. Laboratory project ID13798.6353. Submitted to Bayer CropScience, Research Triangle Park, North Carolina. CA DPR study ID: 283832.

Relevance  
 Score: 100  
 Rating: R

Reliability  
 Score: 98.5  
 Rating: R

Relevance points taken off for: none.

| Fipronil-sulfide                             | <b>Picard 2015</b>  | <i>H. azteca</i> |
|--|---|------------------|
| <b>Parameter</b>                             | <b>Value</b>  | <b>Comment</b>   |
| Test method cited                            | Laboratory protocols that meet US EPA's Ecological Effects Test Guideline (Draft) OCSP 850.1735 Whole Sediment Acute Toxicity Invertebrates, Freshwater |                  |
| Phylum                                       | Arthropoda  |                  |
| Class  | Crustacea   |                  |
| Order  | Malacostraca  |                  |
| Family                                       | Hyalellidae   |                  |
| Genus  | <i>Hyalella</i>   |                  |
| Species                                      | <i>azteca</i>   |                  |
| Family in North America?                     | Yes   |                  |
| Age/size at start of test/growth phase       | 8 d   |                  |
| Source of organisms                          | Laboratory cultures   |                  |
| Have organisms been exposed to contaminants? | No  |                  |
| Animals acclimated and disease-free?         | 48 h  |                  |
| Animals randomized?                          | Yes   |                  |
| Test vessels randomized?                     | Yes   |                  |
| Test duration                                | 10 d  |                  |
| Effect 1                                     | Survival  |                  |
| Control response 1                           | 100 %   |                  |

|  |   |   |
|--|---|---|
| Fipronil-sulfide   | <b>Picard 2015</b>                          | <i>H. azteca</i>                                |
| <b>Parameter</b>   | <b>Value</b>                                | <b>Comment</b>                                  |
| Effect 2   | Dry weight                                  |   |
| Control response 2   | 0.25  |   |
| Temperature  | 23 ± 1 °C                                   |   |
| Test type  | Static renewal                              |   |
| Photoperiod/light intensity                                | 16 l: 8 d; 190-830 lux                      |   |
| Overlying water  | Well water                                  |   |
| pH   | 6.9-7.1                                     |   |
| Hardness   | 52-70 mg/L CaCO <sub>3</sub>                |   |
| Alkalinity   | 22-27 mg/L CaCO <sub>3</sub>                |   |
| Conductivity   | 320-390 µS/cm                               |   |
| Dissolved Oxygen   | 5.1 mg/L                                    | Mean; 60 %                                      |
| TOC  | Not reported                                |   |
| DOC  | Not reported                                |   |
| Ammonia-N  | 6.1 mg/L                                    |   |
| Chemical analysis?/Method                                  | LC/MS/MS                                    |   |
| Sediment source  | Natural sediment                            | Glen Charlie Pone,<br>Wareham,<br>Massachusetts |
| Organic carbon   | 2.7 %                                       |   |
| Particle size distribution<br>(sand, silt, clay)           | 93 % sand, 5 % silt, 2 %<br>clay            |   |
| pH   | 5.8   |   |
| Percent solids   | 51.14 %                                     |   |
| Sediment spike procedure                                   | Jar rolling                                 |   |
| Carrier solvent addition;<br>evaporated (y/n)              | Acetone; yes                                |   |
| Sediment spike equilibration<br>time                       | 28 d  |   |
| Sediment to Solution ratio                                 | 100:175 mL                                  |   |
| Sediment extraction/analysis<br>methods                    | LC/MS/MS                                    |   |
| Interstitial water monitored?                              | Yes   |   |
| Interstitial water isolation<br>method                     | Centrifuge                                  | 30 min; 10,000 g                                |
| Interstitial water<br>extraction/analysis method           | LC/MS/MS                                    |   |
| DOC  | Not reported                                |   |
| Feeding  | 1.0 mL flaked fish food<br>suspension daily |   |
| Purity of test substance                                   | 98.8 %                                      |   |
| Measured is what % of nominal?                             |   |   |
| Toxicity values calculated based on<br>nominal or measured | Measured                                    |   |

| Fipronil-sulfide                                    | Picard 2015   | <i>H. azteca</i>   |
|---|---|--|
| Parameter   | Value   | Comment  |
| concentrations?                                     |   |  |
| Concentration of carrier (if any) in test solutions | Acetone   |  |
| Concentration 1 Nom;Meas (µg/kg)                    | 94; 100 (µg/kg)<br>3.48; 3.70 (µg/g OC)   | 8 reps; 10/rep   |
| Concentration 2 Nom;Meas (µg/kg)                    | 190; 200 (µg/kg)<br>7.04; 7.41 (µg/g OC)  |  |
| Concentration 3 Nom;Meas (µg/kg)                    | 380; 370 (µg/kg)<br>14.07; 13.70 (µg/g OC)  |  |
| Concentration 4 Nom;Meas (µg/kg)                    | 750; 720 (µg/kg)<br>27.78; 26.67 (µg/g OC)  |  |
| Concentration 5 Nom;Meas (µg/kg)                    | 1500; 1600 (µg/kg)<br>55.56; 59.26 (µg/g OC)                                      |  |
| Control   | Negative: 0; 0<br>Solvent: 0; 0   |  |
| LC50 (95% CI) (µg/kg)                               | 1500 (1300-1700) (µg/kg)<br>56 (48-63) (µg/g OC)                                  | Method: Not reported   |
| EC50 (95% CI) (µg/kg)                               | Growth: >1600 (µg/kg)<br>>59 (µg/g OC)  | Method: Empirically  |
| NOEC (µg/kg)  | Survival: 720 (µg/kg)<br>27 (µg/g OC)<br><br>Growth: 370 (µg/kg)<br>14 (µg/g OC)  | Method: Dunnett's<br>Multiple Comparison<br>and Bonferroni's<br>Adjusted t Test<br>(growth); Wilcoxon's<br>Test with Bonferroni's<br>Adjustment (survival)<br>p:<br>MSD: |
| LOEC (µg/kg)  | Survival: 1600 (µg/kg)<br>59 (µg/g OC)<br><br>Growth: 720 (µg/kg)<br>27 (µg/g OC) |  |
| MATC (GeoMean NOEC, LOEC)                           | Survival: 1073 (µg/kg)<br>40 (µg/g OC)  |  |

| Fipronil-sulfide     | Picard 2015                         | <i>H. azteca</i>  |
|----------------------|-------------------------------------|---|
| Parameter            | Value                               | Comment   |
|                      | Growth: 516 (µg/kg)<br>19 (µg/g OC) |   |
| % of control at NOEC | Survival: 100 %<br>Growth: 92 %     | Survival: 100 (tmt) /<br>100 (mean controls) =<br>100 %<br><br>Growth: 0.23 (tmt) /<br>0.25 (mean controls) =<br>92 % |
| % of control at LOEC | Survival: 45 %<br>Growth: 56 %      | Survival: 45 (tmt) /<br>100 (mean controls) =<br>45 %<br><br>Growth: 0.14 (tmt) /<br>0.25 (mean controls) =<br>56     |

Notes: Study reported values in (µg/kg) and values were converted to µg/g OC using the reported % OC.

Solubility value for fipronil sulfide (MB 45950) not available. Solubility (S) of fipronil parent compound (MB 46030) = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation (Table 9): Minimum significant difference (2). Total: 100- 2=98

Acceptability (Table 10): Minimum significant difference (1). Total: 100- 1=99

**Reliability score: Mean (98,99 )=98.5**

## Sediment Toxicity Data Summary

*Hyalella azteca*  
Fipronil sulfone  
MB 46136

Picard CR (2015b) 10-day toxicity test exposing freshwater amphipods (*Hyalella azteca*) to fipronil sulfone (MB43136) applied to sediment under static-renewal conditions following OPPTS Draft Guideline 850.1735. Springborn Viscent, Wareham, Massachusetts. Laboratory project ID13798.6356. Submitted to Bayer CropScience, Research Triangle Park, North Carolina. CA DPR study ID: 283835.

Relevance  
Score: 100  
Rating: R

Reliability  
Score: 98.5  
Rating: R

Relevance points taken off for: none.

| Fipronil sulfone                             | <b>Picard 2015</b>  | <i>H. azteca</i> |
|--|---|------------------|
| <b>Parameter</b>                             | <b>Value</b>  | <b>Comment</b>   |
| Test method cited                            | Laboratory protocols that meet US EPA's Ecological Effects Test Guideline (Draft) OCSP 850.1735 Whole Sediment Acute Toxicity Invertebrates, Freshwater |                  |
| Phylum                                       | Arthropoda  |                  |
| Class  | Crustacea   |                  |
| Order  | Malacostraca  |                  |
| Family                                       | Hyalellidae   |                  |
| Genus  | <i>Hyalella</i>   |                  |
| Species                                      | <i>azteca</i>   |                  |
| Family in North America?                     | Yes   |                  |
| Age/size at start of test/growth phase       | 8 d<br>0.015 mg dry weight  |                  |
| Source of organisms                          | Laboratory cultures   |                  |
| Have organisms been exposed to contaminants? | No  |                  |
| Animals acclimated and disease-free?         | 48 h  |                  |
| Animals randomized?                          | Yes   |                  |
| Test vessels randomized?                     | Yes   |                  |
| Test duration                                | 10 d  |                  |
| Effect 1                                     | Survival  |                  |
| Control response 1                           | 96 %  |                  |

|  |   |   |
|--|---|---|
| Fipronil sulfone   | <b>Picard 2015</b>                          | <i>H. azteca</i>                                |
| <b>Parameter</b>   | <b>Value</b>                                | <b>Comment</b>                                  |
| Effect 2   | Dry weight                                  |   |
| Control response 2   | 0.21  |   |
| Temperature  | 23 ± 1 °C                                   |   |
| Test type  | Static renewal                              |   |
| Photoperiod/light intensity                                | 16 l: 8 d; 520-740 lux                      |   |
| Overlying water  | Well water                                  |   |
| pH   | 6.9-7.1                                     |   |
| Hardness   | 66-70 mg/L CaCO <sub>3</sub>                |   |
| Alkalinity   | 22-27 mg/L CaCO <sub>3</sub>                |   |
| Conductivity   | 360-420 µS/cm                               |   |
| Dissolved Oxygen   | 4.8 mg/L                                    | Mean 0, 10 d; 56 %                              |
| TOC  | Not reported                                |   |
| DOC  | Not reported                                |   |
| Ammonia-N  | 0.34 mg/L                                   | Mean 0, 10 d                                    |
| Chemical analysis?/Method                                  | LC/MS/MS                                    |   |
| Sediment source  | Natural sediment                            | Glen Charlie Pone,<br>Wareham,<br>Massachusetts |
| Organic carbon   | 2.7 %                                       |   |
| Particle size distribution<br>(sand, silt, clay)           | 93 % sand, 5 % silt, 2 %<br>clay            |   |
| pH   | 5.8   |   |
| Percent solids   | 47.84 %                                     |   |
| Sediment spike procedure                                   | Jar rolling                                 |   |
| Carrier solvent addition;<br>evaporated (y/n)              | Acetone; yes                                |   |
| Sediment spike equilibration<br>time                       | 28 d  |   |
| Sediment to Solution ratio                                 | 100:175 mL                                  |   |
| Sediment extraction/analysis<br>methods                    | LC/MS/MS                                    |   |
| Interstitial water monitored?                              | Yes   |   |
| Interstitial water isolation<br>method                     | Centrifuge                                  | 30 min; 10,000 g                                |
| Interstitial water<br>extraction/analysis method           | LC/MS/MS                                    |   |
| DOC  | Not reported                                |   |
| Feeding  | 1.0 mL flaked fish food<br>suspension daily |   |
| Purity of test substance                                   | 99.7 %                                      |   |
| Measured is what % of nominal?                             | Not reported                                |   |
| Toxicity values calculated based on<br>nominal or measured | Measured                                    |   |

| Fipronil sulfone                                    | Picard 2015  | <i>H. azteca</i>   |
|---|--|--|
| Parameter   | Value  | Comment  |
| concentrations?                                     |  |  |
| Concentration of carrier (if any) in test solutions | Acetone  |  |
| Concentration 1 Nom;Meas                            | 50; 48 (µg/kg)<br>1.85; 1.78 (µg/g OC)   | 8 reps; 10/rep   |
| Concentration 2 Nom;Meas                            | 100; 97 (µg/kg)<br>3.70; 3.59 (µg/g OC)  |  |
| Concentration 3 Nom;Meas                            | 200; 200 (µg/kg)<br>7.41; 7.41(µg/g OC)  |  |
| Concentration 4 Nom;Meas                            | 400; 350 (µg/kg)<br>14.81; 12.96 (µg/g OC)   |  |
| Concentration 5 Nom;Meas                            | 800; 830 (µg/kg)<br>29.63; 30.74 (µg/g OC)   |  |
| Control   | Negative: 0; 0<br>Solvent: 0; 0  |  |
| LC50 (95% CI)                                       | 280 (260-290) (µg/kg)<br>10 (10-11) (µg/g OC)  | Method: Not reported   |
| EC50 (95% CI)                                       | Growth: > 350 (µg/kg)<br>>13 (µg/g OC)   | Method: Not reported   |
| NOEC  | Survival: 97 (µg/kg)<br>3.59 (µg/g OC)<br><br>Growth: 200 (µg/kg)<br>7.41 (µg/g OC)    | Method: Dunnett's<br>Multiple Comparison<br>and Bonferroni's<br>Adjusted t Test<br>(growth); Wilcoxon's<br>Test with Bonferroni's<br>Adjustment (survival)<br>p: 0.01-0.05<br>MSD: |
| LOEC  | Survival: 200 (µg/kg)<br>7.41 (µg/g OC)<br><br>Growth: >200 (µg/kg)<br>>7.41 (µg/g OC) |  |

| Fipronil sulfone          | Picard 2015  | <i>H. azteca</i>  |
|---------------------------|--|---|
| Parameter                 | Value  | Comment   |
| MATC (GeoMean NOEC, LOEC) | Survival: 139 (µg/kg)<br>5 (µg/g OC)<br><br>Growth: not calculable |   |
| % of control at NOEC      | Survival: 103 %<br>Growth: 90 %                                    | Survival: 99 (tmt) / 96<br>(mean controls) = 103 %<br><br>Growth: 0.19 (tmt) /<br>0.21 (mean controls) =<br>90% |
| % of control at LOEC      | Survival: 95 %<br>Growth: not calculable                           | Survival: 91 (tmt) / 96<br>(mean controls) = 95 %<br><br>Growth: not calculable                                 |

Notes: Study reported values in (µg/kg) and values were converted to µg/g OC using the reported % OC.

Solubility (S) value for fipronil sulfone (MB 46136) = 160 µg/L, 2S = 320 µg/L.

Reliability points taken off for:

Documentation (Table 9): Minimum significant difference (2). Total: 100- 2=98

Acceptability (Table 10): Minimum significant difference (1). Total: 100- 1=99

**Reliability score: Mean (98,99)=98.5**

## Sediment Toxicity Data Summary

*Hyalella azteca*  
Fipronil desulfinyl  
MB 46513

Picard CR (2015c) 10-day toxicity test exposing freshwater amphipods (*Hyalella azteca*) to fipronil-desulfinyl (MB46513) applied to sediment under static-renewal conditions following OPPTS Draft Guideline 850.1735. Springborn Viscent, Wareham, Massachusetts. Laboratory project ID13798.6360. Submitted to Bayer CropScience, Research Triangle Park, North Carolina. CA DPR study ID: 283837.

Relevance

Score: 100

Rating: R

Reliability

Score: 98.5

Rating: R

Relevance points taken off for: none.

| Fipronil desulfinyl                          | <b>Picard 2015</b>  | <i>H. azteca</i> |
|--|---|------------------|
| <b>Parameter</b>                             | <b>Value</b>  | <b>Comment</b>   |
| Test method cited                            | Laboratory protocols that meet US EPA's Ecological Effects Test Guideline (Draft) OCSP 850.1735 Whole Sediment Acute Toxicity Invertebrates, Freshwater |                  |
| Phylum                                       | Arthropoda  |                  |
| Class  | Crustacea   |                  |
| Order  | Malacostraca  |                  |
| Family                                       | Hyalellidae   |                  |
| Genus  | <i>Hyalella</i>   |                  |
| Species                                      | <i>azteca</i>   |                  |
| Family in North America?                     | Yes   |                  |
| Age/size at start of test/growth phase       | 8 d   |                  |
| Source of organisms                          | Laboratory cultures   |                  |
| Have organisms been exposed to contaminants? | No  |                  |
| Animals acclimated and disease-free?         | 48 h  |                  |
| Animals randomized?                          | Yes   |                  |
| Test vessels randomized?                     | Yes   |                  |
| Test duration                                | 10 d  |                  |
| Effect 1                                     | Survival  |                  |
| Control response 1                           | 97.5 %  |                  |

|  |   |   |
|--|---|---|
| Fipronil desulfanyl  | <b>Picard 2015</b>                          | <i>H. azteca</i>                                |
| <b>Parameter</b>   | <b>Value</b>                                | <b>Comment</b>                                  |
| Effect 2   | Growth (Dry weight)                         |   |
| Control response 2   | 0.19 mg                                     |   |
| Temperature  | 23 ± 1 °C                                   |   |
| Test type  | Static renewal                              |   |
| Photoperiod/light intensity                                | 16 l: 8 d; 200-910 lux                      |   |
| Overlying water  | Well water                                  |   |
| pH   | 6.7-7.0                                     |   |
| Hardness   | 64-70 mg/L CaCO <sub>3</sub>                |   |
| Alkalinity   | 19-22 mg/L CaCO <sub>3</sub>                |   |
| Conductivity   | 370-420 µS/cm                               |   |
| Dissolved Oxygen   | >3.4 mg/L                                   | >40 %   |
| TOC  | Not reported                                |   |
| DOC  | Not reported                                |   |
| Ammonia-N  | 6.1 mg/L                                    | Mean 0, 10 d                                    |
| Chemical analysis?/Method                                  | LC/MS/MS                                    |   |
| Sediment source  | Natural sediment                            | Glen Charlie Pone,<br>Wareham,<br>Massachusetts |
| Organic carbon   | 2.7 %                                       |   |
| Particle size distribution<br>(sand, silt, clay)           | 93 % sand, 5 % silt, 2 %<br>clay            |   |
| pH   | 5.8   |   |
| Percent solids   | 48.61 %                                     |   |
| Sediment spike procedure                                   | Jar rolling                                 |   |
| Carrier solvent addition;<br>evaporated (y/n)              | Acetone; yes                                |   |
| Sediment spike equilibration<br>time                       | 28 d  |   |
| Sediment to Solution ratio                                 | 100:175 mL                                  |   |
| Sediment extraction/analysis<br>methods                    | LC/MS/MS                                    |   |
| Interstitial water monitored?                              | Yes   |   |
| Interstitial water isolation<br>method                     | Centrifuge                                  | 30 min; 10,000 g                                |
| Interstitial water<br>extraction/analysis method           | LC/MS/MS                                    |   |
| DOC  | Not reported                                |   |
| Feeding  | 1.5 mL flaked fish food<br>suspension daily |   |
| Purity of test substance                                   | 99.8 %                                      |   |
| Measured is what % of nominal?                             | 75-92 %                                     |   |
| Toxicity values calculated based on<br>nominal or measured | Measured                                    |   |

| Fipronil desulfinyl                                 | Picard 2015  | <i>H. azteca</i>   |
|---|--|--|
| Parameter   | Value  | Comment  |
| concentrations?                                     |  |  |
| Concentration of carrier (if any) in test solutions | Acetone  |  |
| Concentration 1 Nom;Meas (µg/kg)                    | 750; 560 (µg/kg)<br>28; 21 (µg/g OC)   | 8 reps; 10/rep   |
| Concentration 2 Nom;Meas (µg/kg)                    | 1500; 1300 (µg/kg)<br>56; 48 (µg/g OC)   |  |
| Concentration 3 Nom;Meas (µg/kg)                    | 3000; 2700 (µg/kg)<br>111; 100 (µg/g OC)   |  |
| Concentration 4 Nom;Meas (µg/kg)                    | 6000; 5200 (µg/kg)<br>222; 193 (µg/g OC)   |  |
| Concentration 5 Nom;Meas (µg/kg)                    | 12000; 11000 (µg/kg)<br>444; 407 (µg/g OC)   |  |
| Control   | Negative: 0; 0<br>Solvent: 0; 0  |  |
| LC50 (95% CI) (µg/kg)                               | 4900 (4500-5400) (µg/kg)<br>181 (167-200) (µg/g OC)                                  | Method: Not reported   |
| EC50 (95% CI) (µg/kg)                               | Growth: >5200 (µg/kg)<br>>193 (µg/g OC)  | Method: Not reported   |
| NOEC (µg/kg)  | Survival: 2700 (µg/kg)<br>100 (µg/g OC)<br><br>Growth: 1300 (µg/kg)<br>48 (µg/g OC)  | Method: Bonferroni's<br>Adjusted t-Test<br>(growth); Wilcoxon's<br>Test with Bonferroni's<br>Adjustment (survival)<br>p: 0.05<br>MSD: Not reported |
| LOEC (µg/kg)  | Survival: 5200 (µg/kg)<br>193 (µg/g OC)<br><br>Growth: 2700 (µg/kg)<br>100 (µg/g OC) |  |
| MATC (GeoMean NOEC, LOEC)                           | Survival: 3700 (µg/kg)   |  |

| Fipronil desulfinyl  | Picard 2015   | <i>H. azteca</i>  |
|----------------------|---|---|
| Parameter            | Value   | Comment   |
|                      | 137 (µg/g OC)<br>Growth: 1874 (µg/kg)<br>69 (µg/g OC) |   |
| % of control at NOEC | Survival: 95 %<br>Growth: 74 %                        | Survival: 92.5 (tmt) /<br>97.5 (mean controls) =<br>95 %<br><br>Growth: 0.14 (tmt) /<br>0.19 (mean controls) =<br>74 %  |
| % of control at LOEC | Survival: 47 %<br>Growth: 53 %                        | Survival: 46.25 (tmt) /<br>97.5 (mean controls) =<br>47 %<br><br>Growth: 0.10 (tmt) /<br>0.19 (mean controls) =<br>53 % |

Notes: Study reported values in (µg/kg) and values were converted to µg/g OC using the reported % OC.

Solubility (S) value for fipronil desulfinyl (MB 46513) = 950 µg/L, 2S = 1900 µg/L.

Reliability points taken off for:

Documentation (Table 9): Minimum significant difference (2). Total: 100- 2=98

Acceptability (Table 10): Minimum significant difference (1). Total: 100- 1=99

**Reliability score: Mean (98,99)=98.5**



*Appendix B2 – Sediment Toxicity Studies rated RL, LR, LL*

## Sediment Toxicity Data Summary

*Leptocheirus plumulosus*

Fipronil

MB 46030

Picard CR (2015d) 10-day toxicity test exposing estuarine amphipods (*Leptocheirus plumulosus*) to fipronil applied to sediment under static renewal conditions following OPPTS Draft Guideline 850.1740. Performed by Springborn Viscent, Wareham, MA. Laboratory project ID13798.6351. Submitted to Bayer CropScience, Research Triangle Park, North Carolina. CA DPR study ID: 283830.

Relevance

Score: 86

Rating: L

Reliability

Score: 82

Rating: R

Relevance points taken off for: Freshwater (15). 100-15=85

| Fipronil                                     | <b>Picard 2015</b>  | <i>L. plumulosus</i> |
|--|---|----------------------|
| <b>Parameter</b>                             | <b>Value</b>  | <b>Comment</b>       |
| Test method cited                            | Laboratory protocols that meet US EPA's Ecological Effects Test Guideline (Draft) OCSP 850.1740 Whole Sediment Acute Toxicity Invertebrates, Marine |                      |
| Phylum                                       | Anthropoda  |                      |
| Class  | Malacostraca  |                      |
| Order  | Amphipoda   |                      |
| Family                                       | Corophiidae   |                      |
| Genus  | Leptocheirus  |                      |
| Species                                      | <i>plumulosus</i>   |                      |
| Family in North America?                     | Yes   |                      |
| Age/size at start of test/growth phase       | Juveniles<br>2.0-4.0 mm   |                      |
| Source of organisms                          | Chesapeake Cultures, Hayes, Virginia  |                      |
| Have organisms been exposed to contaminants? | No  |                      |
| Animals acclimated and disease-free?         | 48 h  |                      |
| Animals randomized?                          | Yes   |                      |
| Test vessels randomized?                     | Yes   |                      |
| Test duration                                | 10 d  |                      |
| Effect 1                                     | Survival  |                      |

|   |                                   |  |
|---|-----------------------------------|--|
| Fipronil  | <b>Picard 2015</b>                | <i>L. plumulosus</i>                     |
| <b>Parameter</b>  | <b>Value</b>                      | <b>Comment</b>                           |
| Control response 1  | 97 %                              |  |
| Temperature   | 25 ± 1 °C                         |  |
| Test type   | Static                            |  |
| Photoperiod/light intensity   | 16 l: 8 d/500-980 lux             |  |
| Overlying water   | Natural, filtered seawater        | 21 ‰                                     |
| pH  | 7.8                               |  |
| Hardness  | Not reported                      |  |
| Alkalinity  | Not reported                      |  |
| Conductivity  | Not reported                      |  |
| Dissolved Oxygen  | Not reported                      |  |
| TOC   | 1.3 mg/L                          |  |
| DOC   | Not reported                      |  |
| Ammonia-N   | 8.29 mg/L                         |  |
| Chemical analysis?/Method   | LC/MS/MS                          |  |
| Sediment source   | Natural sediment                  | Sequim Bay,<br>Washington                |
| Organic carbon  | 3.9 %                             |  |
| Particle size distribution<br>(sand, silt, clay)                              | 35 % sand, 40% silt, 25 %<br>clay | Fine silica sand added<br>during spiking |
| pH  | 7.7                               |  |
| Percent solids  | 37.06 %                           |  |
| Sediment spike procedure  | Jar rolling                       |  |
| Carrier solvent addition;<br>evaporated (y/n)                                 | Acetone; yes                      |  |
| Sediment spike equilibration<br>time  | 9 d                               |  |
| Sediment to Solution ratio  | 175:725 mL                        |  |
| Sediment extraction/analysis<br>methods                                       | LC/MS/MS                          |  |
| Interstitial water monitored?   | LC/MS/MS                          |  |
| Interstitial water isolation<br>method  | Centrifuge                        | 30 min; 10,000g                          |
| Interstitial water<br>extraction/analysis method                              | LC/MS/MS                          |  |
| DOC   | Not reported                      |  |
| Feeding   | Not fed during exposure           |  |
| Purity of test substance  | 93.1 %                            |  |
| Measured is what % of nominal?  | 55-72 %                           |  |
| Toxicity values calculated based on<br>nominal or measured<br>concentrations? | Measured                          |  |
| Concentration of carrier (if any) in<br>test solutions                        | Acetone                           |  |

| Fipronil                  | Picard 2015                                      | <i>L. plumulosus</i>  |
|---------------------------|--|---|
| Parameter                 | Value  | Comment   |
| Concentration 1 Nom;Meas  | 5; 2.9 (µg/kg)<br>0.13; 0.07 (µg/g OC)           | 5 reps, 20/rep  |
| Concentration 2 Nom;Meas  | 10; 7.2 (µg/kg)<br>0.26; 0.18 (µg/g OC)          |   |
| Concentration 3 Nom;Meas  | 20; 12 (µg/kg)<br>0.51; 0.31 (µg/g OC)           |   |
| Concentration 4 Nom;Meas  | 40; 22 (µg/kg)<br>1.0; 0.56 (µg/g OC)            |   |
| Concentration 5 Nom;Meas  | 80; 46 (µg/kg)<br>2.1; 1.2 (µg/g OC)             |   |
| Control                   | Negative: 0; 0<br>Solvent: 0; 0                  |   |
| LC50 (95% CI) (µg/kg)     | 21 (19-22) (µg/kg)<br>0.54 (0.49-0.56) (µg/g OC) | Method: Spearman<br>Karber model  |
| NOEC (µg/kg)              | 12 (µg/kg)<br>0.31 (µg/g OC)                     | Method: Dunnett's<br>Multiple Comparison<br>Test and Bonferroni's<br>Adjusted t Test<br>p: 0.01-0.05<br>MSD: Not reported |
| LOEC (µg/kg)              | 22 (µg/kg)<br>0.56 (µg/g OC)                     |   |
| MATC (GeoMean NOEC, LOEC) | 16.3 (µg/kg)<br>0.42 (µg/g OC)                   |   |
| % of control at NOEC      | 102 %  | 99 (tmt) / 97 (mean<br>controls) = 102 %  |
| % of control at LOEC      | 36 %   | 35 (tmt) / 97 (mean<br>controls) = 36 %   |

Notes: Study reported values in (µg/kg) and values were converted to µg/g OC using the reported % OC.

Study reported values in (µg/kg) and values were converted to µg/g OC using the reported % OC.

Sediment analyzed for metabolite MB 45950 (sulfide) on day 10: 1.2, 5.4, 15 µg/kg, respectively, for 5, 20, 80 treatments. MB 45950 concentrations converted to MB 46030

equivalents and partially account for loss of fipronil in sediment attributable to degradation. Some toxicity may be due to this metabolite.

Reliability points taken off for:

Documentation (Table 9): Analytical method (4), Hardness (1), Alkalinity (1), Dissolved oxygen (2), Conductivity (1), Equilibration time (4), Minimum significant difference (2). Total: 100-15 =85

Acceptability (Table 10): Measured concentrations within 20% nominal (4), Equilibration time (6), Hardness (1), Alkalinity (1), Dissolved oxygen (5), Conductivity (1), Random design (2), Minimum significant difference (1). Total: 100-21 =79

**Reliability score: Mean (85, 79)=82**

Sediment Toxicity Data Summary

*Leptocheirus plumulosus*

Fipronil sulfide

MB 45950

Picard CR (2015e) 10-day toxicity test exposing estuarine amphipods (*Leptocheirus plumulosus*) to fipronil sulfide (MB45950) applied to sediment under static conditions following OPPTS Draft Guideline 850.1740. Performed by Springborn Viscent, Wareham, MA. Laboratory project ID13798.6354. Submitted to Bayer CropScience, Research Triangle Park, North Carolina. CA DPR study ID: 283834.

Relevance

Score: 85

Rating: L

Reliability

Score: 95.5

Rating: R

Relevance points taken off for: Freshwater (15). 100-15=85

| Fipronil sulfide                             | <b>Picard 2015</b>  | <i>L. plumulosus</i> |
|--|---|----------------------|
| <b>Parameter</b>                             | <b>Value</b>  | <b>Comment</b>       |
| Test method cited                            | Laboratory protocols that meet US EPA's Ecological Effects Test Guideline (Draft) OCSP 850.1740 Whole Sediment Acute Toxicity Invertebrates, Marine |                      |
| Phylum                                       | Anthropoda  |                      |
| Class  | Malacostraca  |                      |
| Order  | Amphipoda   |                      |
| Family                                       | Corophiidae   |                      |
| Genus  | Leptocheirus  |                      |
| Species                                      | <i>plumulosus</i>   |                      |
| Family in North America?                     | Yes   |                      |
| Age/size at start of test/growth phase       | Juveniles<br>2.0-4.0 mm   |                      |
| Source of organisms                          | Chesapeake Cultures, Hayes, Virginia  |                      |
| Have organisms been exposed to contaminants? | No  |                      |
| Animals acclimated and disease-free?         | 48 h  |                      |
| Animals randomized?                          | Yes   |                      |
| Test vessels randomized?                     | Yes   |                      |
| Test duration                                | 10 d  |                      |
| Effect 1                                     | Survival  |                      |

|   |                                   |  |
|---|-----------------------------------|--|
| Fipronil sulfide  | <b>Picard 2015</b>                | <i>L. plumulosus</i>                     |
| <b>Parameter</b>  | <b>Value</b>                      | <b>Comment</b>                           |
| Control response 1  | 99 %                              |  |
| Temperature   | 23.5 ± 0.5 °C                     |  |
| Test type   | Static                            |  |
| Photoperiod/light intensity   | 16 l:8 d/520-990 lux              |  |
| Overlying water   | Natural, filtered seawater        | 20 ‰                                     |
| pH  | 7.9                               |  |
| Hardness  | Not reported                      |  |
| Alkalinity  | Not reported                      |  |
| Conductivity  | Not reported                      |  |
| Dissolved Oxygen  | 6.9 mL                            | Mean 0, 10 d; 81 %                       |
| TOC   | Not reported                      |  |
| DOC   | Not reported                      |  |
| Ammonia-N   | 11.9 mg/L                         |  |
| Chemical analysis?/Method   | LC/MS/MS                          |  |
| Sediment source   | Natural sediment                  | Sequim Bay,<br>Washington                |
| Organic carbon  | 3.9 %                             |  |
| Particle size distribution<br>(sand, silt, clay)                              | 35 % sand, 40% silt, 25 %<br>clay | Fine silica sand added<br>during spiking |
| pH  | 7.7                               |  |
| Percent solids  | 30.08 %                           |  |
| Sediment spike procedure  | Jar rolling                       |  |
| Carrier solvent addition;<br>evaporated (y/n)                                 | Acetone; yes                      |  |
| Sediment spike equilibration<br>time  | 28 d                              |  |
| Sediment to Solution ratio  | 175:725 mL                        |  |
| Sediment extraction/analysis<br>methods                                       | LC/MS/MS                          |  |
| Interstitial water monitored?   | LC/MS/MS                          |  |
| Interstitial water isolation<br>method  | Centrifuge                        | 30 min; 10,000g                          |
| Interstitial water<br>extraction/analysis method                              | LC/MS/MS                          |  |
| DOC   | Not reported                      |  |
| Feeding   | Not fed during exposure           |  |
| Purity of test substance  | 98.8 %                            |  |
| Measured is what % of nominal?  | 90-110 %                          |  |
| Toxicity values calculated based on<br>nominal or measured<br>concentrations? | Measured                          |  |
| Concentration of carrier (if any) in<br>test solutions                        | Acetone                           |  |

| Fipronil sulfide          | Picard 2015                                   | <i>L. plumulosus</i>   |
|---------------------------|---|--|
| Parameter                 | Value   | Comment  |
| Concentration 1 Nom;Meas  | 5; 5 (µg/kg)<br>0.13; 0.13 (µg/g OC)          | 5 reps, 20/rep   |
| Concentration 2 Nom;Meas  | 10; 11 (µg/kg)<br>0.26; 0.28 (µg/g OC)        |  |
| Concentration 3 Nom;Meas  | 20; 19 (µg/kg)<br>0.51; 0.49 (µg/g OC)        |  |
| Concentration 4 Nom;Meas  | 40; 36 (µg/kg)<br>1.0; 0.92(µg/g OC)          |  |
| Concentration 5 Nom;Meas  | 80; 72 (µg/kg)<br>2.1; 1.9 (µg/g OC)          |  |
| Control                   | Negative: 0; 0<br>Solvent: 0; 0               |  |
| LC50 (95% CI)             | 54 (52-56) (µg/kg)<br>1.4 (1.3-1.4) (µg/g OC) | Method: Trimmed Spearman Karber model                                |
| NOEC                      | 36 (µg/kg)<br>0.92 (µg/g OC)                  | Method: Bonferroni's Adjusted t Test<br>p: 0.05<br>MSD: Not reported |
| LOEC                      | 72 (µg/kg)<br>1.9 (µg/g OC)                   |  |
| MATC (GeoMean NOEC, LOEC) | 51 (µg/kg)<br>1.3 (µg/g OC)                   |  |
| % of control at NOEC      | 97 %  | 96 (tmt) / 99 (mean controls) = 97 %                                 |
| % of control at LOEC      | 17 %  | 17 (tmt) / 99 (mean controls) = 17 %                                 |

Notes: Study reported values in (µg/kg) and values were converted to µg/g OC using the reported % OC.

Solubility value for fipronil sulfide (MB 45950) not available. Solubility (S) of fipronil parent compound (MB 46030) = 1650.8 µg/L, 2S = 3301.6 µg/L.

Reliability points taken off for:

Documentation (Table 9): Hardness (1), Alkalinity (1), Conductivity (1), Minimum significant difference (2). Total:  $100-5 = 95$

Acceptability (Table 10): Hardness (1), Alkalinity (1), Conductivity (1), Minimum significant difference (1). Total:  $100-4 = 96$

**Reliability score: Mean (95, 96)=95.5**

## Sediment Toxicity Data Summary

*Leptocheirus plumulosus*

Fipronil sulfone

MB 46136

Picard CR (2015f) 10-day toxicity test exposing estuarine amphipods (*Leptocheirus plumulosus*) to fipronil sulfone (MB46136) applied to sediment under static conditions following OPPTS Draft Guideline 850.1740. Performed by Springborn Viscent, Wareham, MA. Laboratory project ID13798.6357. Submitted to Bayer CropScience, Research Triangle Park, North Carolina. CA DPR study ID: 283836.

Relevance

Score: 85

Rating: L

Reliability

Score: 95.5

Rating: R

Relevance points taken off for: Freshwater (15). 100-15=85

| Fipronil sulfone                             | <b>Picard 2015</b>  | <i>L. plumulosus</i> |
|--|---|----------------------|
| <b>Parameter</b>                             | <b>Value</b>  | <b>Comment</b>       |
| Test method cited                            | Laboratory protocols that meet US EPA's Ecological Effects Test Guideline (Draft) OCSP 850.1740 Whole Sediment Acute Toxicity Invertebrates, Marine |                      |
| Phylum                                       | Anthropoda  |                      |
| Class  | Malacostraca  |                      |
| Order  | Amphipoda   |                      |
| Family                                       | Corophiidae   |                      |
| Genus  | Leptocheirus  |                      |
| Species                                      | <i>plumulosus</i>   |                      |
| Family in North America?                     | Yes   |                      |
| Age/size at start of test/growth phase       | Juveniles<br>2.0-4.0 mm   |                      |
| Source of organisms                          | Chesapeake Cultures, Hayes, Virginia  |                      |
| Have organisms been exposed to contaminants? | No  |                      |
| Animals acclimated and disease-free?         | 48 h  |                      |
| Animals randomized?                          | Yes   |                      |
| Test vessels randomized?                     | Yes   |                      |
| Test duration                                | 10 d  |                      |
| Effect 1                                     | Survival  |                      |

|   |                                   |  |
|---|-----------------------------------|--|
| Fipronil sulfone  | <b>Picard 2015</b>                | <i>L. plumulosus</i>                     |
| <b>Parameter</b>  | <b>Value</b>                      | <b>Comment</b>                           |
| Control response 1  | 99 %                              |  |
| Temperature   | 25 ± 1 °C                         |  |
| Test type   | Static                            |  |
| Photoperiod/light intensity   | 16 l:8 d/550-980 lux              |  |
| Overlying water   | Natural, filtered seawater        | 20-21 ‰                                  |
| pH  | 7.7                               |  |
| Hardness  | Not reported                      |  |
| Alkalinity  | Not reported                      |  |
| Conductivity  | Not reported                      |  |
| Dissolved Oxygen  | 6.5 mL                            | Mean 0, 10 d; 76 %                       |
| TOC   | Not reported                      |  |
| DOC   | Not reported                      |  |
| Ammonia-N   | 14.5 mg/L                         |  |
| Chemical analysis?/Method   | LC/MS/MS                          |  |
| Sediment source   | Natural sediment                  | Sequim Bay,<br>Washington                |
| Organic carbon  | 3.9 %                             |  |
| Particle size distribution<br>(sand, silt, clay)                              | 35 % sand, 40% silt, 25 %<br>clay | Fine silica sand added<br>during spiking |
| pH  | 7.7                               |  |
| Percent solids  | 35.09 %                           |  |
| Sediment spike procedure  | Jar rolling                       |  |
| Carrier solvent addition;<br>evaporated (y/n)                                 | Acetone; yes                      |  |
| Sediment spike equilibration<br>time  | 28 d                              |  |
| Sediment to Solution ratio  | 175:725 mL                        |  |
| Sediment extraction/analysis<br>methods                                       | LC/MS/MS                          |  |
| Interstitial water monitored?   | LC/MS/MS                          |  |
| Interstitial water isolation<br>method  | Centrifuge                        | 30 min; 10,000g                          |
| Interstitial water<br>extraction/analysis method                              | LC/MS/MS                          |  |
| DOC   | Not reported                      |  |
| Feeding   | Not fed during exposure           |  |
| Purity of test substance  | 99.7 %                            |  |
| Measured is what % of nominal?  | 68-100 %                          |  |
| Toxicity values calculated based on<br>nominal or measured<br>concentrations? | Measured                          |  |
| Concentration of carrier (if any) in<br>test solutions                        | Acetone                           |  |

| Fipronil sulfone          | Picard 2015                                      | <i>L. plumulosus</i>   |
|---------------------------|--|--|
| Parameter                 | Value  | Comment  |
| Concentration 1 Nom;Meas  | 5; 3.4 (µg/kg)<br>0.13; 0.09 (µg/g OC)           | 5 reps, 20/rep   |
| Concentration 2 Nom;Meas  | 10; 9.2 (µg/kg)<br>0.26; 0.24 (µg/g OC)          |  |
| Concentration 3 Nom;Meas  | 20; 20 (µg/kg)<br>0.51; 0.51 (µg/g OC)           |  |
| Concentration 4 Nom;Meas  | 40; 38 (µg/kg)<br>1.0; 0.97 (µg/g OC)            |  |
| Concentration 5 Nom;Meas  | 80; 79 (µg/kg)<br>2.1; 2.0 (µg/g OC)             |  |
| Control                   | Negative: 0; 0<br>Solvent: 0; 0                  |  |
| LC50 (95% CI)             | 27 (27-28) (µg/kg)<br>0.69 (0.69-0.72) (µg/g OC) | Method: Not reported   |
| NOEC                      | 20 (µg/kg)<br>0.51 (µg/g OC)                     | Method: Wilcoxon's<br>Test with Bonferroni's<br>Adjustment<br>p: 0.05<br>MSD: Not reported |
| LOEC                      | 38 (µg/kg)<br>0.97 (µg/g OC)                     |  |
| MATC (GeoMean NOEC, LOEC) | 27 (µg/kg)<br>0.71 (µg/g OC)                     |  |
| % of control at NOEC      | 98 %   | 97 (tmt) / 99 (mean<br>controls) = 98 %  |
| % of control at LOEC      | 1 %  | 1 (tmt) / 99 (mean<br>controls) = 1 %  |

Notes: Study reported values in (µg/kg) and values were converted to µg/g OC using the reported % OC.

Solubility (S) value for fipronil sulfone (MB 46136) = 160 µg/L, 2S = 320 µg/L.

Reliability points taken off for:

Documentation (Table 9): Hardness (1), Alkalinity (1), Conductivity (1), Minimum significant difference (2). Total:  $100-5 = 95$

Acceptability (Table 10): Hardness (1), Alkalinity (1), Conductivity (1), Minimum significant difference (1). Total:  $100-4 = 96$

**Reliability score: Mean (95, 96)=95.5**

## Sediment Toxicity Data Summary

*Leptocheirus plumulosus*

Fipronil desulfinyl

MB 46513

Picard CR (2015g) 10-day toxicity test exposing estuarine amphipods (*Leptocheirus plumulosus*) to fipronil desulfinyl (MB46513) applied to sediment under static conditions following OPPTS Draft Guideline 850.1740. Performed by Springborn Viscent, Wareham, MA. Laboratory project ID13798.6361. Submitted to Bayer CropScience, Research Triangle Park, North Carolina. CA DPR study ID: 283838.

Relevance

Score: 85

Rating: L

Reliability

Score: 95.5

Rating: R

Relevance points taken off for: Freshwater (15). 100-15=85

| Fipronil desulfinyl                          | <b>Picard 2015</b>  | <i>L. plumulosus</i> |
|--|---|----------------------|
| <b>Parameter</b>                             | <b>Value</b>  | <b>Comment</b>       |
| Test method cited                            | Laboratory protocols that meet US EPA's Ecological Effects Test Guideline (Draft) OCSP 850.1740 Whole Sediment Acute Toxicity Invertebrates, Marine |                      |
| Phylum                                       | Anthropoda  |                      |
| Class  | Malacostraca  |                      |
| Order  | Amphipoda   |                      |
| Family                                       | Corophiidae   |                      |
| Genus  | Leptocheirus  |                      |
| Species                                      | <i>plumulosus</i>   |                      |
| Family in North America?                     | Yes   |                      |
| Age/size at start of test/growth phase       | Juveniles<br>2.0-4.0 mm   |                      |
| Source of organisms                          | Chesapeake Cultures, Hayes, Virginia  |                      |
| Have organisms been exposed to contaminants? | No  |                      |
| Animals acclimated and disease-free?         | 48 h  |                      |
| Animals randomized?                          | Yes   |                      |
| Test vessels randomized?                     | Yes   |                      |
| Test duration                                | 10 d  |                      |
| Effect 1                                     | Survival  |                      |

|   |                                   |  |
|---|-----------------------------------|--|
| Fipronil desulfanyl   | <b>Picard 2015</b>                | <i>L. plumulosus</i>                     |
| <b>Parameter</b>  | <b>Value</b>                      | <b>Comment</b>                           |
| Control response 1  | 99 %                              |  |
| Temperature   | 24.5 ± 0.5 °C                     |  |
| Test type   | Static                            |  |
| Photoperiod/light intensity   | 16 l:8 d/600-790 lux              |  |
| Overlying water   | Natural, filtered seawater        | 20 ‰                                     |
| pH  | 7.8                               |  |
| Hardness  | Not reported                      |  |
| Alkalinity  | Not reported                      |  |
| Conductivity  | Not reported                      |  |
| Dissolved Oxygen  | >4.4 mL                           | > 60 %                                   |
| TOC   | Not reported                      |  |
| DOC   | Not reported                      |  |
| Ammonia-N   | 15 mg/L                           |  |
| Chemical analysis?/Method   | LC/MS/MS                          |  |
| Sediment source   | Natural sediment                  | Sequim Bay,<br>Washington                |
| Organic carbon  | 3.9 %                             |  |
| Particle size distribution<br>(sand, silt, clay)                              | 35 % sand, 40% silt, 25 %<br>clay | Fine silica sand added<br>during spiking |
| pH  | 7.7                               |  |
| Percent solids  | 35.36 %                           |  |
| Sediment spike procedure  | Jar rolling                       |  |
| Carrier solvent addition;<br>evaporated (y/n)                                 | Acetone; yes                      |  |
| Sediment spike equilibration<br>time  | 29 d                              |  |
| Sediment to Solution ratio  | 175:725 mL                        |  |
| Sediment extraction/analysis<br>methods                                       | LC/MS/MS                          |  |
| Interstitial water monitored?   | LC/MS/MS                          |  |
| Interstitial water isolation<br>method  | Centrifuge                        | 30 min; 10,000g                          |
| Interstitial water<br>extraction/analysis method                              | LC/MS/MS                          |  |
| DOC   | Not reported                      |  |
| Feeding   | Not fed during exposure           |  |
| Purity of test substance  | 97.8 %                            |  |
| Measured is what % of nominal?  | 80-97 %                           |  |
| Toxicity values calculated based on<br>nominal or measured<br>concentrations? | Measured                          |  |
| Concentration of carrier (if any) in<br>test solutions                        | Acetone                           |  |

| Fipronil desulfinyl       | Picard 2015                                      | <i>L. plumulosus</i>   |
|---------------------------|--|--|
| Parameter                 | Value  | Comment  |
| Concentration 1 Nom;Meas  | 31; 30 (µg/kg)<br>1.1; 1.0 (µg/g OC)             | 5 reps, 20/rep   |
| Concentration 2 Nom;Meas  | 62; 61 (µg/kg)<br>2.1; 2.1 (µg/g OC)             |  |
| Concentration 3 Nom;Meas  | 130; 120 (µg/kg)<br>4.5; 4.1 (µg/g OC)           |  |
| Concentration 4 Nom;Meas  | 250; 200 (µg/kg)<br>8.6; 6.9 (µg/g OC)           |  |
| Concentration 5 Nom;Meas  | 500; 470 (µg/kg)<br>17; 16 (µg/g OC)             |  |
| Control                   | Negative: 0; 0<br>Solvent: 0; 0                  |  |
| LC50 (95% CI)             | 240 (220-250) (µg/kg)<br>8.3 (7.6-8.6) (µg/g OC) | Method: Not reported   |
| NOEC                      | 120 (µg/kg)<br>4.1 (µg/g OC)                     | Method: Steel's One-Many Rank Sum Test<br>p: 0.05<br>MSD: Not reported |
| LOEC                      | 200 (µg/kg)<br>6.9 (µg/g OC)                     |  |
| MATC (GeoMean NOEC, LOEC) | 155 (µg/kg)<br>5.3 (µg/g OC)                     |  |
| % of control at NOEC      | 97 %   | 96 (tmt) / 99 (mean controls) = 97 %                                   |
| % of control at LOEC      | 65 %   | 64 (tmt) / 99 (mean controls) = 65 %                                   |

Notes: Study reported values in (µg/kg) and values were converted to µg/g OC using the reported % OC.

Solubility (S) value for fipronil desulfinyl (MB 46513) = 950 µg/L, 2S = 1900 µg/L.

Reliability points taken off for:

Documentation (Table 9): Hardness (1), Alkalinity (1), Conductivity (1), Minimum significant difference (2). Total:  $100-5 = 95$

Acceptability (Table 10): Hardness (1), Alkalinity (1), Conductivity (1), Minimum significant difference (1). Total:  $100-4 = 96$

**Reliability score: Mean (95, 96)=95.5**

## Sediment Toxicity Data Summary

*Mysidopsis bahia*

Fipronil

MB 46030

Cafarella MA. (2005) Fipronil: Life-cycle toxicity test with mysids (*Americamysis bahia*) under static conditions in a water-sediment system. Springborn Smithers Laboratories, Wareham, Massachusetts. Laboratory project ID 986.6163. Submitted to BASF Corporation, Research Triangle Park, North Carolina. US EPA MRID 46619103.

Relevance

Score: 70

Rating: L

Reliability

Score: 82

Rating: R

Relevance points taken off for: Freshwater (15), Toxicity value bioavailability (15). 100-30=70

| Fipronil                                     | <b>Cafarella 2005</b>  | <i>M. bahia</i> |
|--|--|-----------------|
| <b>Parameter</b>                             | <b>Value</b>   | <b>Comment</b>  |
| Test method cited                            | OPPTS 850.1350 Mysid Chronic Toxicity Test (1996), EPA's Pesticide Assessment Guidelines Subdivision E (1982), Standard Guide for Conduction Life-Cycle Toxicity Tests with Saltwater Mysids (ASTM 1994) |                 |
| Phylum                                       | Arthropoda/Crustacea   |                 |
| Class  | Malacostraca   |                 |
| Order  | Mysida   |                 |
| Family                                       | Mysidae  |                 |
| Genus  | <i>Americamysis</i>  |                 |
| Species                                      | <i>bahia</i>   |                 |
| Family in North America?                     | Yes  |                 |
| Age/size at start of test/growth phase       | 21 d, <24 hr   |                 |
| Source of organisms                          | Laboratory cultures  |                 |
| Have organisms been exposed to contaminants? | No   |                 |
| Animals acclimated and disease-free?         | Yes  |                 |
| Animals randomized?                          | Yes  |                 |
| Test vessels randomized?                     | Not reported   |                 |
| Test duration                                | 28 d   |                 |

| Fipronil                    | Cafarella 2005  | <i>M. bahia</i>   |
|-----------------------------|---|---|
| Parameter                   | Value   | Comment   |
| Effect 1                    | 21 d old: female survival   | Reproduction calculated as the ratio of number offspring:total number females/chamber/day |
| Control response 1          | 92 %  |   |
| Effect 2                    | 21 d old: reproduction  | Number offspring per reproductive day   |
| Control response 2          | 0.59  |   |
| Effect 3                    | <24 h old: female survival  |   |
| Control response 3          | 100 %   |   |
| Effect 4                    | <24 h old: reproduction   | Number offspring per reproductive day   |
| Control response 4          | 2.2   |   |
| Effect 5                    | <24 h old: growth   |   |
| Control response 5          | Male length: 7.7 mm<br>Male weight: 0.94 mg<br>Female length: 7.9 mm<br>Female weight: 1.2 mg |   |
| Effect 6                    | <24 h old: number adults and juveniles  |   |
| Control response 6          | Juveniles: 489<br>Adults: 46  |   |
| Temperature                 | 25 ± 2 °C   |   |
| Test type                   | Static  |   |
| Photoperiod/light intensity | 16 l: 8 d/720-1250 lux  |   |
| Overlying water             | Filtered seawater diluted with well water   | Salinity: 20 ± 1 ‰  |
| pH                          | 7.9   |   |
| Hardness                    | Not reported  |   |
| Alkalinity                  | Not reported  |   |
| Conductivity                | 27,000 µmhos/cm   |   |
| Dissolved Oxygen            | > 4.3 mg/L  | >60 %   |
| TOC                         | Not reported  |   |
| DOC                         | Not reported  | Microalga added to control ammonia  |
| Ammonia-N                   | 18.6 mg/L   | Field collected microalga in each vessel to control ammonia levels                        |
| Chemical analysis?/Method   | LC/MS/MS  |   |
| Sediment source             | Natural marine sediment   | Little Harbor Beach, Wareham,   |

| Fipronil  | Cafarella 2005  | <i>M. bahia</i>  |
|---|---|--|
| Parameter   | Value   | Comment  |
|   |   | Massachusetts  |
| Organic carbon  | 2.7 %   |  |
| Particle size distribution<br>(sand, silt, clay)                              | 77 % sand, 14 % silt, 9 %<br>clay   |  |
| pH  | 7.7   |  |
| Percent solids  | Not reported  |  |
| Sediment spike procedure  | Sediment not spiked,<br>fipronil added to overlying<br>water                        |  |
| Carrier solvent addition;<br>evaporated (y/n)                                 | Acetone, not evaporated   |  |
| Sediment spike equilibration<br>time  | Not reported  |  |
| Sediment to Solution ratio  | 16,000 mL:750 mL  |  |
| Sediment extraction/analysis<br>methods                                       | LC/MS/MS  |  |
| Interstitial water monitored?   | Not reported  |  |
| Interstitial water isolation<br>method  | Not reported  |  |
| Interstitial water<br>extraction/analysis method                              | Not reported  |  |
| DOC   | Not reported  |  |
| Feeding   | Live <i>Artemia salina</i> nauplii<br>twice daily and daily<br>supplements of Selco |  |
| Purity of test substance  | 99.7 %  |  |
| Measured is what % of nominal?  | Overlying water: 73-112 %<br>Sediment: 75-81 %                                      |  |
| Toxicity values calculated based on<br>nominal or measured<br>concentrations? | Nominal   |  |
| Concentration of carrier (if any) in<br>test solutions                        | Acetone   |  |
| Concentration 1 Nom;Meas (µg/L)   | Overlying water: 0.015;<br>0.0170 <sup>a</sup> , <LOQ <sup>b</sup>                  | 4 reps, 50 mysids/rep<br><sup>a</sup> day 0<br><sup>b</sup> day 28 |
| Concentration 2 Nom;Meas (µg/L)   | Overlying water 0.030;<br>0.031, 0.0042 <sup>b</sup>                                | <sup>a</sup> day 0<br><sup>b</sup> day 28                          |
| Concentration 3 Nom;Meas (µg/L)   | Overlying water 0.060;<br>0.055 <sup>a</sup> , <LOQ <sup>b</sup>                    | <sup>a</sup> day 0<br><sup>b</sup> day 28                          |
| Control   | Solvent: 0; not reported  |  |
| NOEC (µg/L)   | 0.060   | Method: Williams'<br>Test, Bonferroni's T-                         |

| Fipronil                  | Cafarella 2005  | <i>M. bahia</i>   |
|---------------------------|---|---|
| Parameter                 | Value   | Comment   |
|                           |   | test<br>p: 0.01-0.05<br>MSD:  |
| LOEC (µg/L)               | Survival, reproduction,<br>growth: >0.060   |   |
| MATC (GeoMean NOEC, LOEC) | Not calculable  |   |
| % of control at NOEC      | <p>21 d old:<br/>female survival = 97 %<br/>reproduction = 75 %</p> <p>&lt;24 h old:<br/>female survival = 95 %<br/>reproduction= 91 %<br/>male length: 99 %<br/>male weight: 94 %<br/>female length: 103 %<br/>female weight: 108 %<br/>number juveniles: 74 %<br/>number adults: 91 %</p> | <p>21 d old female<br/>survival: 89 (tmt) / 92<br/>(control) = 97 %</p> <p>21 d old reproduction:<br/>0.44 (tmt) / 0.59<br/>(control) = 75 %</p> <p>&lt;24 h old female<br/>survival: 95 (tmt) / 100<br/>(control) = 95 %</p> <p>&lt;24 h old<br/>reproduction: 2.0 (tmt)<br/>/ 2.2 (control) = 91 %</p> <p>Male length: 7.6 (tmt) /<br/>7.7 (control) = 99 %</p> <p>Male weight: 0.88<br/>(tmt) / 0.94 (control) =<br/>94 %</p> <p>Female length: 8.1<br/>(tmt) / 7.9 (control) =<br/>103 %</p> <p>Female weight: 1.3<br/>(tmt) / 1.2 (control) =<br/>108 %</p> <p>&lt;24 h old number of<br/>juveniles: 362 (tmt) /<br/>489 (control) = 74 %</p> <p>&lt;24 h old number of<br/>adults: 42 (tmt) / 46<br/>(control) = 91 %</p> |

|                      |                       |                 |
|----------------------|-----------------------|-----------------|
| Fipronil             | <b>Cafarella 2005</b> | <i>M. bahia</i> |
| <b>Parameter</b>     | <b>Value</b>          | <b>Comment</b>  |
| % of control at LOEC | Not calculable        |                 |

Reliability points taken off for:

Documentation (Table 9): Hardness (1), Alkalinity (1), Spike method (4), Equilibration time (4), Minimum significant difference (2), Point estimates (8). Total: 100-20 = 80

Acceptability (Table 10): Spike method (4), Equilibration time (6), Hardness (1), Alkalinity (1), Random design (2), Minimum significant difference (1), % control at NOEC (1). Total: 100-16 =84

**Reliability score: Mean (80, 84)= 82**

# **Appendix C – Ecosystem Rating Tables**

*Appendix C1 – Ecosystem Toxicity Studies rated R*

Fipronil  
MB 46030

Wirth EF, Pennington PL, Lawton JC, DeLorenzo ME, Bearden D, Shaddrix B, Sivertsen S and Fulton MH. (2004) The effects of the contemporary-use insecticide (fipronil) in an estuarine mesocosm. *Environmental Pollution*, 131(3), 365-371.

Study duration: 28 days. No significant effects on added fish (*Cyprinidon variegatus*), clams (*Mercenaria mercenaria*), or oysters (*Crassostrea virginica*) but grass shrimp (*Palaemonetes pugio*) experienced measured toxicity.

Documentation and acceptability (reliability) evaluation for data derived from aquatic outdoor field and indoor model ecosystems experiments. Include notes next to each parameter. Adapted from ECOTOX 2006; Table from TenBrook et al. 2010.

| Parameter <sup>a</sup>  | Score <sup>b</sup> | Points   |
|---|--------------------|----------|
| Results published or in signed, dated format <b>Peer review journal</b>   | 5                  | <b>5</b> |
| Exposure duration and sample regime adequately described  | 6                  | <b>6</b> |
| Unimpacted site (Score 7 for artificial systems) <b>Stated undeveloped site</b>   | 7                  | <b>7</b> |
| Adequate range of organisms in system (1 <sup>o</sup> producers, 1 <sup>o</sup> , 2 <sup>o</sup> consumers) <b>Environmental microflora/fauna in collected sediment; introduced fish, shrimp, oysters</b> | 6                  | <b>6</b> |
| Chemical  |                    |          |
| Grade or purity stated <b>ChemService, website = 99.5 %</b>   | 6                  | <b>6</b> |
| Concentrations measured/estimated and reported <b>Estimated: 0.15, 0.355, 5 µg/L</b>  | 8                  | <b>8</b> |
| Analysis method stated <b>GC-ECD</b>  | 2                  | <b>2</b> |
| Habitat described (e.g., pond, lake, ditch, artificial, lentic, lotic) <b>Estuary</b>   | 6                  | <b>6</b> |
| Water quality   |                    |          |
| Source identified <b>Natural seawater from Bohicket Creek, South Carolina</b>   | 2                  | <b>2</b> |
| Hardness reported <b>Not reported</b>   | 1                  | <b>0</b> |
| Alkalinity reported <b>Not reported</b>   | 1                  | <b>0</b> |
| Dissolved oxygen reported <b>5.9-8.8 mg/L</b>   | 2                  | <b>2</b> |
| Temperature reported <b>28.5-31.1</b>   | 2                  | <b>2</b> |
| Conductivity reported <b>Not reported</b>   | 1                  | <b>0</b> |
| pH reported <b>7.7-8.1</b>  | 1                  | <b>1</b> |
| Photoperiod reported <b>Not reported</b>  | 1                  | <b>0</b> |

| Parameter <sup>a</sup>   | Score <sup>b</sup> | Points    |
|--|--------------------|-----------|
| Organic carbon reported <b>Not reported</b>                                      | 2                  | <b>0</b>  |
| Chemical fate reported   | 3                  | <b>0</b>  |
| Geographic location identified (Score 2 for indoor systems) <b>Not reported</b>  | 2                  | <b>2</b>  |
| Pesticide application  |                    |           |
| Type reported (e.g., spray, dilutor, injection) <b>Via Sump</b>                  | 2                  | <b>2</b>  |
| Frequency reported <b>Once, time=0</b>   | 2                  | <b>2</b>  |
| Date/season reported (Score 2 for indoor systems) <b>90 days past March 2001</b> | 2                  | <b>2</b>  |
| Test endpoints   |                    |           |
| Species abundance reported <b>Shrimp population structure reported</b>           | 3                  | <b>3</b>  |
| Species diversity reported   | 3                  | <b>0</b>  |
| Biomass reported   | 2                  | <b>0</b>  |
| Ecosystem recovery reported  | 2                  | <b>0</b>  |
| Statistics   |                    |           |
| Methods identified <b>Probit</b>   | 2                  | <b>2</b>  |
| At least 2 replicates <b>2</b>   | 3                  | <b>3</b>  |
| At least 2 test concentrations and 1 control <b>3 plus control</b>               | 3                  | <b>3</b>  |
| Dose-response relationship observed <b>Shrimp</b>                                | 2                  | <b>2</b>  |
| Hypothesis tests   |                    |           |
| NOEC determined <b>Shrimp LC<sub>50</sub> = 0.357 µg/L</b>                       | 4                  | <b>0</b>  |
| Significance level stated <b>Not reported</b>                                    | 2                  | <b>0</b>  |
| Minimum significant difference reported <b>Not reported</b>                      | 2                  | <b>0</b>  |
| % of control at NOEC and/or LOEC reported or calculable <b>Not reported</b>      | 2                  | <b>0</b>  |
| <b>Total Reliability</b>   | 100                | <b>74</b> |

LOEC = lowest observed effect concentration, NOEC = no observed effect concentration.

<sup>a</sup>Compiled from RIVM 2001, USEPA 1985 and 2003a, ECOTOX 2006, CCME 1995, ANZECC and ARMCANZ 2000, OECD 1995a, and van der Hoeven et al. 1997.

<sup>b</sup>Weighting based on ECOTOX 2006 and on data quality criteria in RIVM 2001 and OECD 1995a.

*Appendix C2 – Ecosystem Toxicity Studies rated L*

Fipronil  
MB 46030

Walse SS, Pennington PL, Scott GI and Ferry JL. (2004) The fate of fipronil in modular estuarine mesocosms. *Journal of Environmental Monitoring*, 6(1), 58-64.

Study focused on degradate formation in aqueous and sediment phases. No toxicity values were calculated.

Documentation and acceptability (reliability) evaluation for data derived from aquatic outdoor field and indoor model ecosystems experiments. Include notes next to each parameter. Adapted from ECOTOX 2006; Table from TenBrook et al. 2010.

| Parameter <sup>a</sup>  | Score <sup>b</sup> | Points   |
|---|--------------------|----------|
| Results published or in signed, dated format <b>Peer review journal</b>   | 5                  | <b>5</b> |
| Exposure duration and sample regime adequately described  | 6                  | <b>6</b> |
| Unimpacted site (Score 7 for artificial systems) <b>Stated pristine site</b>  | 7                  | <b>7</b> |
| Adequate range of organisms in system (1° producers, 1°, 2° consumers) <b>Epibenthic, benthic, meiobenthic flora/fauna from environmental sediments</b> | 6                  | <b>6</b> |
| Chemical  |                    |          |
| Grade or purity stated <b>98 %</b>  | 6                  | <b>6</b> |
| Concentrations measured/estimated and reported <b>Not reported</b>  | 8                  | <b>0</b> |
| Analysis method stated <b>GC-MS</b>   | 2                  | <b>2</b> |
| Habitat described (e.g., pond, lake, ditch, artificial, lentic, lotic) <b>Tidal marsh</b>   | 6                  | <b>6</b> |
| Water quality   |                    |          |
| Source identified <b>Seawater collected from Cherry Point Boat Landing, Wadmalaw Island, South Carolina</b>   | 2                  | <b>2</b> |
| Hardness reported <b>Not reported</b>   | 1                  | <b>0</b> |
| Alkalinity reported <b>Not reported</b>   | 1                  | <b>0</b> |
| Dissolved oxygen reported <b>6.2 mg/L</b>   | 2                  | <b>2</b> |
| Temperature reported <b>28.6°C</b>  | 2                  | <b>2</b> |
| Conductivity reported <b>Not reported</b>   | 1                  | <b>0</b> |
| pH reported <b>7.8</b>  | 1                  | <b>1</b> |
| Photoperiod reported <b>Measured but not reported</b>   | 1                  | <b>1</b> |
| Organic carbon reported <b>3.8-4.1 %</b>  | 2                  | <b>2</b> |

| Parameter <sup>a</sup>  | Score <sup>b</sup> | Points    |
|---|--------------------|-----------|
| Chemical fate reported  | 3                  | 3         |
| Geographic location identified (Score 2 for indoor systems) <b>Indoor</b> | 2                  | 2         |
| Pesticide application   |                    |           |
| Type reported (e.g., spray, dilutor, injection) <b>Not reported</b>       | 2                  | 0         |
| Frequency reported <b>Once</b>  | 2                  | 2         |
| Date/season reported (Score 2 for indoor systems) <b>Indoor</b>           | 2                  | 2         |
| Test endpoints  |                    |           |
| Species abundance reported <b>Measured but not reported</b>               | 3                  | 3         |
| Species diversity reported <b>Not reported</b>                            | 3                  | 0         |
| Biomass reported <b>Not reported</b>                                      | 2                  | 0         |
| Ecosystem recovery reported <b>Not reported</b>                           | 2                  | 0         |
| Statistics  |                    |           |
| Methods identified <b>Not reported</b>                                    | 2                  | 0         |
| At least 2 replicates <b>3</b>  | 3                  | 3         |
| At least 2 test concentrations and 1 control <b>One plus control</b>      | 3                  | 0         |
| Dose-response relationship observed <b>Not reported</b>                   | 2                  | 0         |
| Hypothesis tests  |                    |           |
| NOEC determined   | 4                  | 0         |
| Significance level stated   | 2                  | 0         |
| Minimum significant difference reported                                   | 2                  | 0         |
| % of control at NOEC and/or LOEC reported or calculable                   | 2                  | 0         |
| <b>Total Reliability</b>  | 100                | <b>63</b> |

LOEC = lowest observed effect concentration, NOEC = no observed effect concentration.

<sup>a</sup>Compiled from RIVM 2001, USEPA 1985 and 2003a, ECOTOX 2006, CCME 1995, ANZECC and ARMCANZ 2000, OECD 1995a, and van der Hoeven et al. 1997.

<sup>b</sup>Weighting based on ECOTOX 2006 and on data quality criteria in RIVM 2001 and OECD 1995a.

*Appendix C3 – Ecosystem Toxicity Studies rated N*

Fipronil

MB 46030

Aajoud A, Ravanel P and Tissut M. (2003) Fipronil metabolism and dissipation in a simplified aquatic ecosystem. *Journal of agricultural and food chemistry*, 51(5), 1347-1352.

Study conducted ecosystem component with environmental organic matter and microbes to determine fate. Toxicity of fipronil and its degradates to *Aedes aegypti* was determined in separate bioassays with LD<sub>50</sub> values given.

Documentation and acceptability (reliability) evaluation for data derived from aquatic outdoor field and indoor model ecosystems experiments. Include notes next to each parameter. Adapted from ECOTOX 2006; Table from TenBrook et al. 2010.

| Parameter <sup>a</sup>   | Score <sup>b</sup> | Points |
|--|--------------------|--------|
| Results published or in signed, dated format <b>Published peer review article</b>  | 5                  | 5      |
| Exposure duration and sample regime adequately described   | 6                  | 6      |
| Unimpacted site (Score 7 for artificial systems) <b>Indoor microcosm</b>   | 7                  | 7      |
| Adequate range of organisms in system (1° producers, 1°, 2° consumers)<br><b>Range of unidentified environmental microorganisms and algae; mosquito larvae toxicity tested after</b> | 6                  | 6      |
| Chemical   |                    |        |
| Grade or purity stated <b>99.3 %</b>   | 6                  | 6      |
| Concentrations measured/estimated and reported <b>Not reported</b>   | 8                  | 0      |
| Analysis method stated <b>GLC</b>  | 2                  | 2      |
| Habitat described (e.g., pond, lake, ditch, artificial, lentic, lotic) <b>Pond</b>   | 6                  | 6      |
| Water quality  |                    |        |
| Source identified <b>Distilled</b>   | 2                  | 2      |
| Hardness reported <b>Not reported</b>  | 1                  | 0      |
| Alkalinity reported <b>Not reported</b>  | 1                  | 0      |
| Dissolved oxygen reported <b>Not reported</b>  | 2                  | 0      |
| Temperature reported <b>Not reported</b>   | 2                  | 0      |
| Conductivity reported <b>Not reported</b>  | 1                  | 0      |
| pH reported <b>7-7.2</b>   | 1                  | 1      |
| Photoperiod reported <b>Not reported</b>   | 1                  | 0      |

| Parameter <sup>a</sup>  | Score <sup>b</sup> | Points |
|---|--------------------|--------|
| Organic carbon reported <b>Not reported</b>                                   | 2                  | 0      |
| Chemical fate reported  | 3                  | 3      |
| Geographic location identified (Score 2 for indoor systems) <b>Indoor</b>     | 2                  | 2      |
| Pesticide application   |                    |        |
| Type reported (e.g., spray, dilutor, injection) <b>Single dose in solvent</b> | 2                  | 2      |
| Frequency reported <b>Once</b>  | 2                  | 2      |
| Date/season reported (Score 2 for indoor systems) <b>Indoor</b>               | 2                  | 2      |
| Test endpoints  |                    |        |
| Species abundance reported <b>Not reported</b>                                | 3                  | 0      |
| Species diversity reported <b>Not reported</b>                                | 3                  | 0      |
| Biomass reported <b>Not reported</b>  | 2                  | 0      |
| Ecosystem recovery reported <b>Not reported</b>                               | 2                  | 0      |
| Statistics  |                    |        |
| Methods identified <b>Log-probit</b>  | 2                  | 2      |
| At least 2 replicates <b>Not reported</b>                                     | 3                  | 0      |
| At least 2 test concentrations and 1 control <b>Single concentration</b>      | 3                  | 0      |
| Dose-response relationship observed <b>Not reported</b>                       | 2                  | 0      |
| Hypothesis tests  |                    |        |
| NOEC determined <b>Not reported</b>   | 4                  | 0      |
| Significance level stated <b>Not reported</b>                                 | 2                  | 0      |
| Minimum significant difference reported <b>Not reported</b>                   | 2                  | 0      |
| % of control at NOEC and/or LOEC reported or calculable <b>Not reported</b>   | 2                  | 0      |
| <b>Total Reliability</b>  | 100                | 54     |

LOEC = lowest observed effect concentration, NOEC = no observed effect concentration.

<sup>a</sup>Compiled from RIVM 2001, USEPA 1985 and 2003a, ECOTOX 2006, CCME 1995, ANZECC and ARMCANZ 2000, OECD 1995a, and van der Hoeven et al. 1997.

<sup>b</sup>Weighting based on ECOTOX 2006 and on data quality criteria in RIVM 2001 and OECD 1995a.

# **Appendix D – Wildlife Rating Tables**

*Appendix D1 – Wildlife Toxicity Studies rated R*

Fipronil  
MB 46030

Wirth EF, Pennington PL, Lawton JC, DeLorenzo ME, Bearden D, Shaddrix B, Sivertsen S and Fulton MH. (2004) The effects of the contemporary-use insecticide (fipronil) in an estuarine mesocosm. *Environmental Pollution*, 131(3), 365-371.

Study duration: 28 days. No significant effects on added fish (*Cyprinidon variegatus*), clams (*Mercenaria mercenaria*), or oysters (*Crassostrea virginica*) but grass shrimp (*Palaemonetes pugio*) experienced measured toxicity.

Documentation and acceptability (reliability) evaluation for data derived from aquatic outdoor field and indoor model ecosystems experiments. Include notes next to each parameter. Adapted from ECOTOX 2006; Table from TenBrook et al. 2010.

| Parameter <sup>a</sup>  | Score <sup>b</sup> | Points   |
|---|--------------------|----------|
| Results published or in signed, dated format <b>Peer review journal</b>                     | 5                  | <b>5</b> |
| Exposure duration and sample regime adequately described                                    | 6                  | <b>6</b> |
| Unimpacted site (Score 7 for artificial systems) <b>Stated undeveloped site</b>             | 7                  | <b>7</b> |
| Adequate range of organisms in system (1° producers, 1°, 2° consumers) <b>Environmental</b> | 6                  | <b>6</b> |

| Parameter <sup>a</sup>  | Score <sup>b</sup> | Points |
|---|--------------------|--------|
| <b>microflora/fauna in collected sediment; introduced fish, shrimp, oysters</b>       |                    |        |
| Chemical  |                    |        |
| Grade or purity stated <b>ChemService, website = 99.5 %</b>                           | 6                  | 6      |
| Concentrations measured/estimated and reported <b>Estimated: 0.15, 0.355, 5 µg/L</b>  | 8                  | 8      |
| Analysis method stated <b>GC-ECD</b>  | 2                  | 2      |
| Habitat described (e.g., pond, lake, ditch, artificial, lentic, lotic) <b>Estuary</b> | 6                  | 6      |
| Water quality   |                    |        |
| Source identified <b>Natural seawater from Bohicket Creek, South Carolina</b>         | 2                  | 2      |
| Hardness reported <b>Not reported</b>   | 1                  | 0      |
| Alkalinity reported <b>Not reported</b>   | 1                  | 0      |
| Dissolved oxygen reported <b>5.9-8.8 mg/L</b>   | 2                  | 2      |
| Temperature reported <b>28.5-31.1</b>   | 2                  | 2      |
| Conductivity reported <b>Not reported</b>   | 1                  | 0      |
| pH reported <b>7.7-8.1</b>  | 1                  | 1      |
| Photoperiod reported <b>Not reported</b>  | 1                  | 0      |
| Organic carbon reported <b>Not reported</b>   | 2                  | 0      |
| Chemical fate reported  | 3                  | 0      |
| Geographic location identified (Score 2 for indoor systems) <b>Not reported</b>       | 2                  | 2      |
| Pesticide application   |                    |        |
| Type reported (e.g., spray, dilutor, injection) <b>Via Sump</b>                       | 2                  | 2      |
| Frequency reported <b>Once, time=0</b>  | 2                  | 2      |
| Date/season reported (Score 2 for indoor systems) <b>90 days past March 2001</b>      | 2                  | 2      |
| Test endpoints  |                    |        |
| Species abundance reported <b>Shrimp population structure reported</b>                | 3                  | 3      |
| Species diversity reported  | 3                  | 0      |
| Biomass reported  | 2                  | 0      |
| Ecosystem recovery reported   | 2                  | 0      |
| Statistics  |                    |        |

| Parameter <sup>a</sup>  | Score <sup>b</sup> | Points |
|---|--------------------|--------|
| Methods identified <b>Probit</b>  | 2                  | 2      |
| At least 2 replicates <b>2</b>  | 3                  | 3      |
| At least 2 test concentrations and 1 control <b>3 plus control</b>          | 3                  | 3      |
| Dose-response relationship observed <b>Shrimp</b>                           | 2                  | 2      |
| Hypothesis tests  |                    |        |
| NOEC determined <b>Shrimp LC<sub>50</sub> = 0.357 µg/L</b>                  | 4                  | 0      |
| Significance level stated <b>Not reported</b>                               | 2                  | 0      |
| Minimum significant difference reported <b>Not reported</b>                 | 2                  | 0      |
| % of control at NOEC and/or LOEC reported or calculable <b>Not reported</b> | 2                  | 0      |
| <b>Total Reliability</b>  | 100                | 74     |

LOEC = lowest observed effect concentration, NOEC = no observed effect concentration.

<sup>a</sup>Compiled from RIVM 2001, USEPA 1985 and 2003a, ECOTOX 2006, CCME 1995, ANZECC and ARMCANZ 2000, OECD 1995a, and van der Hoeven et al. 1997.

<sup>b</sup>Weighting based on ECOTOX 2006 and on data quality criteria in RIVM 2001 and OECD 1995a.

## *Appendix D2 – Wildlife Toxicity Studies rated L*

Fipronil  
MB 46030

Walse SS, Pennington PL, Scott GI and Ferry JL. (2004) The fate of fipronil in modular estuarine mesocosms. *Journal of Environmental Monitoring*, 6(1), 58-64.

Study focused on degradate formation in aqueous and sediment phases. No toxicity values were calculated.

Documentation and acceptability (reliability) evaluation for data derived from aquatic outdoor field and indoor model ecosystems experiments. Include notes next to each parameter. Adapted from ECOTOX 2006; Table from TenBrook et al. 2010.

| Parameter <sup>a</sup>  | Score <sup>b</sup> | Points   |
|---|--------------------|----------|
| Results published or in signed, dated format <b>Peer review journal</b>   | 5                  | <b>5</b> |
| Exposure duration and sample regime adequately described  | 6                  | <b>6</b> |
| Unimpacted site (Score 7 for artificial systems) <b>Stated pristine site</b>  | 7                  | <b>7</b> |
| Adequate range of organisms in system (1° producers, 1°, 2° consumers) <b>Epibenthic, benthic, meiobenthic flora/fauna from environmental sediments</b> | 6                  | <b>6</b> |

| Parameter <sup>a</sup>  | Score <sup>b</sup> | Points   |
|---|--------------------|----------|
| Chemical  |                    |          |
| Grade or purity stated <b>98 %</b>  | 6                  | <b>6</b> |
| Concentrations measured/estimated and reported <b>Not reported</b>  | 8                  | <b>0</b> |
| Analysis method stated <b>GC-MS</b>   | 2                  | <b>2</b> |
| Habitat described (e.g., pond, lake, ditch, artificial, lentic, lotic) <b>Tidal marsh</b>                   | 6                  | <b>6</b> |
| Water quality   |                    |          |
| Source identified <b>Seawater collected from Cherry Point Boat Landing, Wadmalaw Island, South Carolina</b> | 2                  | <b>2</b> |
| Hardness reported <b>Not reported</b>   | 1                  | <b>0</b> |
| Alkalinity reported <b>Not reported</b>   | 1                  | <b>0</b> |
| Dissolved oxygen reported <b>6.2 mg/L</b>   | 2                  | <b>2</b> |
| Temperature reported <b>28.6°C</b>  | 2                  | <b>2</b> |
| Conductivity reported <b>Not reported</b>   | 1                  | <b>0</b> |
| pH reported <b>7.8</b>  | 1                  | <b>1</b> |
| Photoperiod reported <b>Measured but not reported</b>   | 1                  | <b>1</b> |
| Organic carbon reported <b>3.8-4.1 %</b>  | 2                  | <b>2</b> |
| Chemical fate reported  | 3                  | <b>3</b> |
| Geographic location identified (Score 2 for indoor systems) <b>Indoor</b>                                   | 2                  | <b>2</b> |
| Pesticide application   |                    |          |
| Type reported (e.g., spray, dilutor, injection) <b>Not reported</b>   | 2                  | <b>0</b> |
| Frequency reported <b>Once</b>  | 2                  | <b>2</b> |
| Date/season reported (Score 2 for indoor systems) <b>Indoor</b>   | 2                  | <b>2</b> |
| Test endpoints  |                    |          |
| Species abundance reported <b>Measured but not reported</b>   | 3                  | <b>3</b> |
| Species diversity reported <b>Not reported</b>  | 3                  | <b>0</b> |
| Biomass reported <b>Not reported</b>  | 2                  | <b>0</b> |
| Ecosystem recovery reported <b>Not reported</b>   | 2                  | <b>0</b> |
| Statistics  |                    |          |

| Parameter <sup>a</sup>   | Score <sup>b</sup> | Points    |
|--|--------------------|-----------|
| Methods identified <b>Not reported</b>                               | 2                  | <b>0</b>  |
| At least 2 replicates <b>3</b>                                       | 3                  | <b>3</b>  |
| At least 2 test concentrations and 1 control <b>One plus control</b> | 3                  | <b>0</b>  |
| Dose-response relationship observed <b>Not reported</b>              | 2                  | <b>0</b>  |
| Hypothesis tests   |                    |           |
| NOEC determined  | 4                  | <b>0</b>  |
| Significance level stated  | 2                  | <b>0</b>  |
| Minimum significant difference reported                              | 2                  | <b>0</b>  |
| % of control at NOEC and/or LOEC reported or calculable              | 2                  | <b>0</b>  |
| <b>Total Reliability</b>   | 100                | <b>63</b> |

LOEC = lowest observed effect concentration, NOEC = no observed effect concentration.

<sup>a</sup>Compiled from RIVM 2001, USEPA 1985 and 2003a, ECOTOX 2006, CCME 1995, ANZECC and ARMCANZ 2000, OECD 1995a, and van der Hoeven et al. 1997.

<sup>b</sup>Weighting based on ECOTOX 2006 and on data quality criteria in RIVM 2001 and OECD 1995a.

## *Appendix D3 – Wildlife Toxicity Studies rated N*

Fipronil  
MB 46030

Aajoud A, Ravanel P and Tissut M. (2003) Fipronil metabolism and dissipation in a simplified aquatic ecosystem. *Journal of agricultural and food chemistry*, 51(5), 1347-1352.

Study conducted ecosystem component with environmental organic matter and microbes to determine fate. Toxicity of fipronil and its degradates to *Aedes aegypti* was determined in separate bioassays with LD<sub>50</sub> values given.

Documentation and acceptability (reliability) evaluation for data derived from aquatic outdoor field and indoor model ecosystems experiments. Include notes next to each parameter. Adapted from ECOTOX 2006; Table from TenBrook et al. 2010.

| Parameter <sup>a</sup>  | Score <sup>b</sup> | Points   |
|---|--------------------|----------|
| Results published or in signed, dated format <b>Published peer review article</b> | 5                  | <b>5</b> |
| Exposure duration and sample regime adequately described                          | 6                  | <b>6</b> |

| Parameter <sup>a</sup>   | Score <sup>b</sup> | Points |
|--|--------------------|--------|
| Unimpacted site (Score 7 for artificial systems) <b>Indoor microcosm</b>   | 7                  | 7      |
| Adequate range of organisms in system (1° producers, 1°, 2° consumers)<br><b>Range of unidentified environmental microorganisms and algae; mosquito larvae toxicity tested after</b> | 6                  | 6      |
| Chemical   |                    |        |
| Grade or purity stated <b>99.3 %</b>   | 6                  | 6      |
| Concentrations measured/estimated and reported <b>Not reported</b>   | 8                  | 0      |
| Analysis method stated <b>GLC</b>  | 2                  | 2      |
| Habitat described (e.g., pond, lake, ditch, artificial, lentic, lotic) <b>Pond</b>   | 6                  | 6      |
| Water quality  |                    |        |
| Source identified <b>Distilled</b>   | 2                  | 2      |
| Hardness reported <b>Not reported</b>  | 1                  | 0      |
| Alkalinity reported <b>Not reported</b>  | 1                  | 0      |
| Dissolved oxygen reported <b>Not reported</b>  | 2                  | 0      |
| Temperature reported <b>Not reported</b>   | 2                  | 0      |
| Conductivity reported <b>Not reported</b>  | 1                  | 0      |
| pH reported <b>7-7.2</b>   | 1                  | 1      |
| Photoperiod reported <b>Not reported</b>   | 1                  | 0      |
| Organic carbon reported <b>Not reported</b>  | 2                  | 0      |
| Chemical fate reported   | 3                  | 3      |
| Geographic location identified (Score 2 for indoor systems) <b>Indoor</b>  | 2                  | 2      |
| Pesticide application  |                    |        |
| Type reported (e.g., spray, dilutor, injection) <b>Single dose in solvent</b>  | 2                  | 2      |
| Frequency reported <b>Once</b>   | 2                  | 2      |
| Date/season reported (Score 2 for indoor systems) <b>Indoor</b>  | 2                  | 2      |
| Test endpoints   |                    |        |
| Species abundance reported <b>Not reported</b>   | 3                  | 0      |
| Species diversity reported <b>Not reported</b>   | 3                  | 0      |
| Biomass reported <b>Not reported</b>   | 2                  | 0      |

| Parameter <sup>a</sup>  | Score <sup>b</sup> | Points    |
|---|--------------------|-----------|
| Ecosystem recovery reported <b>Not reported</b>                             | 2                  | <b>0</b>  |
| Statistics  |                    |           |
| Methods identified <b>Log-probit</b>  | 2                  | <b>2</b>  |
| At least 2 replicates <b>Not reported</b>                                   | 3                  | <b>0</b>  |
| At least 2 test concentrations and 1 control <b>Single concentration</b>    | 3                  | <b>0</b>  |
| Dose-response relationship observed <b>Not reported</b>                     | 2                  | <b>0</b>  |
| Hypothesis tests  |                    |           |
| NOEC determined <b>Not reported</b>   | 4                  | <b>0</b>  |
| Significance level stated <b>Not reported</b>                               | 2                  | <b>0</b>  |
| Minimum significant difference reported <b>Not reported</b>                 | 2                  | <b>0</b>  |
| % of control at NOEC and/or LOEC reported or calculable <b>Not reported</b> | 2                  | <b>0</b>  |
| <b>Total Reliability</b>  | 100                | <b>54</b> |

LOEC = lowest observed effect concentration, NOEC = no observed effect concentration.

<sup>a</sup>Compiled from RIVM 2001, USEPA 1985 and 2003a, ECOTOX 2006, CCME 1995, ANZECC and ARMCANZ 2000, OECD 1995a, and van der Hoeven et al. 1997.

<sup>b</sup>Weighting based on ECOTOX 2006 and on data quality criteria in RIVM 2001 and OECD 1995a.

# **Appendix E – Acute WQC Fit Test for fipronil**

Fipronil—Burr III SSD

|    | SMAV   | Omit   |        |        |        |        |        |        |        |        |        |        |        |        |        |        |        |        |        |
|----|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|
|    |        | one    | 1      | 2      | 3      | 4      | 5      | 6      | 7      | 8      | 9      | 10     | 11     | 12     | 13     | 14     | 15     | 16     | 17     |
| 1  | 0.0519 |        |        | 0.0519 | 0.0519 | 0.0519 | 0.0519 | 0.0519 | 0.0519 | 0.0519 | 0.0519 | 0.0519 | 0.0519 | 0.0519 | 0.0519 | 0.0519 | 0.0519 | 0.0519 | 0.0519 |
| 2  | 0.03   | 0.03   |        | 0.03   | 0.03   | 0.03   | 0.03   | 0.03   | 0.03   | 0.03   | 0.03   | 0.03   | 0.03   | 0.03   | 0.03   | 0.03   | 0.03   | 0.03   | 0.03   |
| 3  | 33.3   | 33.3   | 33.3   |        | 33.3   | 33.3   | 33.3   | 33.3   | 33.3   | 33.3   | 33.3   | 33.3   | 33.3   | 33.3   | 33.3   | 33.3   | 33.3   | 33.3   | 33.3   |
| 4  | 190    | 190    | 190    | 190    |        | 190    | 190    | 190    | 190    | 190    | 190    | 190    | 190    | 190    | 190    | 190    | 190    | 190    | 190    |
| 5  | 0.0707 | 0.0707 | 0.0707 | 0.0707 | 0.0707 |        | 0.0707 | 0.0707 | 0.0707 | 0.0707 | 2.0707 | 0.0707 | 0.0707 | 0.0707 | 0.0707 | 0.0707 | 3.0707 | 0.0707 | 0.0707 |
| 6  | 0.267  | 0.267  | 0.267  | 0.267  | 0.267  | 0.267  |        | 0.267  | 0.267  | 0.267  | 0.267  | 0.267  | 0.267  | 0.267  | 0.267  | 0.267  | 0.267  | 0.267  | 0.267  |
| 7  | 0.46   | 0.46   | 0.46   | 0.46   | 0.46   | 0.46   | 0.46   |        | 0.46   | 0.46   | 0.46   | 0.46   | 0.46   | 0.46   | 0.46   | 0.46   | 0.46   | 0.46   | 0.46   |
| 8  | 0.727  | 0.727  | 0.727  | 0.727  | 0.727  | 0.727  | 0.727  | 0.727  |        | 0.727  | 0.727  | 0.727  | 0.727  | 0.727  | 0.727  | 0.727  | 0.727  | 0.727  | 0.727  |
| 9  | 0.602  | 0.602  | 0.602  | 0.602  | 0.602  | 0.602  | 0.602  | 0.602  | 0.602  |        | 0.602  | 0.602  | 0.602  | 0.602  | 0.602  | 0.602  | 0.602  | 0.602  | 0.602  |
| 10 | 560    | 560    | 560    | 560    | 560    | 560    | 560    | 560    | 560    | 560    |        | 560    | 560    | 560    | 560    | 560    | 560    | 560    | 560    |
| 11 | 0.101  | 0.101  | 0.101  | 0.101  | 0.101  | 0.101  | 0.101  | 0.101  | 0.101  | 0.101  | 0.101  |        | 0.101  | 0.101  | 0.101  | 0.101  | 0.101  | 0.101  | 0.101  |
| 12 | 85.2   | 85.2   | 85.2   | 85.2   | 85.2   | 85.2   | 85.2   | 85.2   | 85.2   | 85.2   | 85.2   | 85.2   |        | 85.2   | 85.2   | 85.2   | 85.2   | 85.2   | 85.2   |
| 13 | 0.0634 | 0.0634 | 0.0634 | 0.0634 | 0.0634 | 0.0634 | 0.0634 | 0.0634 | 0.0634 | 0.0634 | 0.0634 | 0.0634 | 0.0634 |        | 0.0634 | 0.0634 | 0.0634 | 0.0634 | 0.0634 |
| 14 | 248    | 248    | 248    | 248    | 248    | 248    | 248    | 248    | 248    | 248    | 248    | 248    | 248    | 248    |        | 248    | 248    | 248    | 248    |
| 15 | 208    | 208    | 208    | 208    | 208    | 208    | 208    | 208    | 208    | 208    | 208    | 208    | 208    | 208    | 208    | 208    |        | 208    | 208    |
| 16 | 0.589  | 0.589  | 0.589  | 0.589  | 0.589  | 0.589  | 0.589  | 0.589  | 0.589  | 0.589  | 0.589  | 0.589  | 0.589  | 0.589  | 0.589  | 0.589  | 0.589  |        | 0.589  |
| 17 | 0.19   | 0.19   | 0.19   | 0.19   | 0.19   | 0.19   | 0.19   | 0.19   | 0.19   | 0.19   | 0.19   | 0.19   | 0.19   | 0.19   | 0.19   | 0.19   | 0.19   | 0.19   |        |

Fipronil—Burr III SSD

| <b>Omitted point, xi:</b>                | 0.0519 | 0.03  | 33.3  | 190   | 0.0707 | 0.267 | 0.46  | 0.727 | 0.602 | 560   | 0.101 | 85.2  | 0.0634 | 248   | 208   | 0.589 | 0.19  |
|--|--------|-------|-------|-------|--------|-------|-------|-------|-------|-------|-------|-------|--------|-------|-------|-------|-------|
|  | 1      | 2     | 3     | 4     | 5      | 6     | 7     | 8     | 9     | 10    | 11    | 12    | 13     | 14    | 15    | 16    | 17    |
| <b>median 5th percentile</b><br>Burr III | 0.039  | 0.039 | 0.024 | 0.026 | 0.031  | 0.025 | 0.024 | 0.024 | 0.024 | 0.027 | 0.029 | 0.025 | 0.031  | 0.026 | 0.026 | 0.024 | 0.026 |
| <b>percentile</b>                        | 93     | 96    | 16    | 7.8   | 89     | 72    | 65    | 58    | 61    | 4.7   | 85    | 11    | 90     | 6.9   | 7.5   | 61    | 77    |
| <b>F-i(xi)</b>                           | 0.93   | 0.96  | 0.16  | 0.078 | 0.89   | 0.72  | 0.65  | 0.58  | 0.61  | 0.047 | 0.85  | 0.11  | 0.9    | 0.069 | 0.075 | 0.61  | 0.77  |
| <b>1-F(xi)</b>                           | 0.07   | 0.04  | 0.84  | 0.922 | 0.11   | 0.28  | 0.35  | 0.42  | 0.39  | 0.953 | 0.15  | 0.89  | 0.1    | 0.931 | 0.925 | 0.39  | 0.23  |
| <b>Min of F-i(xi) or 1-F(xi)</b>         | 0.07   | 0.04  | 0.16  | 0.078 | 0.11   | 0.28  | 0.35  | 0.42  | 0.39  | 0.047 | 0.15  | 0.11  | 0.1    | 0.069 | 0.075 | 0.39  | 0.23  |
| <b>p<sub>i</sub> =2(min)</b>             | 0.14   | 0.08  | 0.32  | 0.156 | 0.22   | 0.56  | 0.7   | 0.84  | 0.78  | 0.094 | 0.3   | 0.22  | 0.2    | 0.138 | 0.15  | 0.78  | 0.46  |

Fipronil—Burr III SSD

| $p_i$  | $\ln(p_i)$ | Fisher test statistic               |            |
|--------|------------|-------------------------------------|------------|
|        |            | $-2 \times \text{Sum of } \ln(p_i)$ | $X^2_{2n}$ |
| 0.1400 | -1.9661    | 43.9144                             | 0.1188     |
| 0.0800 | -2.5257    |                                     |            |
| 0.3200 | -1.1394    |                                     |            |
| 0.1560 | -1.8579    |                                     |            |
| 0.2200 | -1.5141    |                                     |            |
| 0.5600 | -0.5798    |                                     |            |
| 0.7000 | -0.3567    |                                     |            |
| 0.8400 | -0.1744    |                                     |            |
| 0.7800 | -0.2485    |                                     |            |
| 0.0940 | -2.3645    |                                     |            |
| 0.3000 | -1.2040    |                                     |            |
| 0.2200 | -1.5141    |                                     |            |
| 0.2000 | -1.6094    |                                     |            |
| 0.1380 | -1.9805    |                                     |            |
| 0.1500 | -1.8971    |                                     |            |
| 0.7800 | -0.2485    |                                     |            |
| 0.4600 | -0.7765    |                                     |            |

if  $X^2 < 0.05$     significant lack of fit  
 if  $X^2 > 0.05$     fit (no significant lack of fit)

# **Appendix F – Acute WQC Fit Test for fipronil-sulfone, Burr**

## **III SSD**

Fipronil-sulfone—Burr III SSD

|    | SMAV   | Omit one | 2      | 3      | 4      | 5      | 6      | 7      | 8      | 9      | 10     | 11     | 12     | 13     | 14     | 15     |
|----|--------|----------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|
|    |        | 1        |        |        |        |        |        |        |        |        |        |        |        |        |        |        |
| 1  | 0.0079 |          | 0.0079 | 0.0079 | 0.0079 | 0.0079 | 0.0079 | 0.0079 | 0.0079 | 0.0079 | 0.0079 | 0.0079 | 0.0079 | 0.0079 | 0.0079 | 0.0079 |
| 2  | 0.0313 | 0.0313   |        | 0.0313 | 0.0313 | 0.0313 | 0.0313 | 0.0313 | 0.0313 | 0.0313 | 0.0313 | 0.0313 | 0.0313 | 0.0313 | 0.0313 | 0.0313 |
| 3  | 0.0474 | 0.0474   | 0.0474 |        | 0.0474 | 0.0474 | 0.0474 | 0.0474 | 0.0474 | 0.0474 | 0.0474 | 0.0474 | 0.0474 | 0.0474 | 0.0474 | 0.0474 |
| 4  | 0.0717 | 0.0717   | 0.0717 | 0.0717 |        | 0.0717 | 0.0717 | 0.0717 | 0.0717 | 0.0717 | 0.0717 | 0.0717 | 0.0717 | 0.0717 | 0.0717 | 0.0717 |
| 5  | 0.0729 | 0.0729   | 0.0729 | 0.0729 | 0.0729 |        | 0.0729 | 0.0729 | 0.0729 | 0.0729 | 2.0707 | 0.0729 | 0.0729 | 0.0729 | 0.0729 | 3.0707 |
| 6  | 0.0738 | 0.0738   | 0.0738 | 0.0738 | 0.0738 | 0.0738 |        | 0.0738 | 0.0738 | 0.0738 | 0.0738 | 0.0738 | 0.0738 | 0.0738 | 0.0738 | 0.0738 |
| 7  | 0.075  | 0.075    | 0.075  | 0.075  | 0.075  | 0.075  | 0.075  |        | 0.075  | 0.075  | 0.075  | 0.075  | 0.075  | 0.075  | 0.075  | 0.075  |
| 8  | 0.0926 | 0.0926   | 0.0738 | 0.0738 | 0.0738 | 0.0738 | 0.0738 | 0.0738 |        | 0.0738 | 0.0738 | 0.0738 | 0.0738 | 0.0738 | 0.0738 | 0.0738 |
| 9  | 0.0959 | 0.0959   | 0.0959 | 0.0959 | 0.0959 | 0.0959 | 0.0959 | 0.0959 | 0.0959 |        | 0.0959 | 0.0959 | 0.0959 | 0.0959 | 0.0959 | 0.0959 |
| 10 | 0.155  | 0.155    | 0.155  | 0.155  | 0.155  | 0.155  | 0.155  | 0.155  | 0.155  | 0.155  |        | 0.155  | 0.155  | 0.155  | 0.155  | 0.155  |
| 11 | 0.159  | 0.159    | 0.159  | 0.159  | 0.159  | 0.159  | 0.159  | 0.159  | 0.159  | 0.159  | 0.159  |        | 0.159  | 0.159  | 0.159  | 0.159  |
| 12 | 0.163  | 0.163    | 0.163  | 0.163  | 0.163  | 0.163  | 0.163  | 0.163  | 0.163  | 0.163  | 0.163  | 0.163  |        | 0.163  | 0.163  | 0.163  |
| 13 | 25     | 25       | 25     | 25     | 25     | 25     | 25     | 25     | 25     | 25     | 25     | 25     | 25     |        | 25     | 25     |
| 14 | 29     | 29       | 29     | 29     | 29     | 29     | 29     | 29     | 29     | 29     | 29     | 29     | 29     | 29     |        | 29     |
| 15 | 39     | 39       | 39     | 39     | 39     | 39     | 39     | 39     | 39     | 39     | 39     | 39     | 39     | 39     | 39     |        |

Fipronil-sulfone—Burr III SSD

|                                       |        |        |        |        |        |        |       |        |        |       |       |       |        |       |        |
|---------------------------------------|--------|--------|--------|--------|--------|--------|-------|--------|--------|-------|-------|-------|--------|-------|--------|
| <b>Omitted point, xi:</b>             | 0.0079 | 0.0313 | 0.0474 | 0.0717 | 0.0729 | 0.0738 | 0.075 | 0.0926 | 0.0959 | 0.155 | 0.159 | 0.163 | 25     | 29    | 39     |
| <b>median 5th percentile Burr III</b> | 0.027  | 0.027  | 0.024  | 0.022  | 0.022  | 0.0738 | 0.022 | 0.022  | 0.022  | 0.021 | 0.021 | 0.021 | 0.026  | 0.026 | 0.027  |
| <b>percentile</b>                     | 100    | 93     | 85     | 75     | 74     | 74     | 73    | 68     | 67     | 54    | 54    | 53    | 0.71   | 0.6   | 0.42   |
| <b>F-i(xi)</b>                        | 1      | 0.93   | 0.85   | 0.75   | 0.74   | 0.74   | 0.73  | 0.68   | 0.67   | 0.54  | 0.54  | 0.53  | 0.0071 | 0.006 | 0.0042 |
| <b>1-F(xi)</b>                        | 0      | 0.07   | 0.15   | 0.25   | 0.26   | 0.26   | 0.27  | 0.32   | 0.33   | 0.46  | 0.46  | 0.47  | 0.9929 | 0.994 | 0.9958 |
| <b>Min of F-i(xi) or 1-F(xi)</b>      | 0      | 0.07   | 0.15   | 0.25   | 0.26   | 0.26   | 0.27  | 0.32   | 0.33   | 0.46  | 0.46  | 0.47  | 0.0071 | 0.006 | 0.0042 |
| <b>p<sub>i</sub> =2(min)</b>          | 0      | 0.14   | 0.3    | 0.5    | 0.52   | 0.52   | 0.54  | 0.64   | 0.66   | 0.92  | 0.92  | 0.94  | 0.0142 | 0.012 | 0.0084 |

Fipronil-sulfone—Burr III SSD

| p <sub>i</sub> | ln(p <sub>i</sub> ) | Fisher test statistic          |            |
|----------------|---------------------|--------------------------------|------------|
|                |                     | -2*Sum of ln (p <sub>i</sub> ) | $X^2_{2n}$ |
| 0.0000         | n/a                 | 43.3633                        | 0.0867     |
| 0.1400         | -1.9661             |                                |            |
| 0.3000         | -1.2040             |                                |            |
| 0.5000         | -0.6931             |                                |            |
| 0.5200         | -0.6539             |                                |            |
| 0.5000         | -0.6931             |                                |            |
| 0.5200         | -0.6539             |                                |            |
| 0.5200         | -0.6539             |                                |            |
| 0.5400         | -0.6162             |                                |            |
| 0.6400         | -0.4463             |                                |            |
| 0.6600         | -0.4155             |                                |            |
| 0.9200         | -0.0834             |                                |            |
| 0.9200         | -0.0834             |                                |            |
| 0.9400         | -0.0619             |                                |            |
| 0.0142         | -4.2545             |                                |            |
| 0.0120         | -4.4228             |                                |            |
| 0.0084         | -4.7795             |                                |            |

# **Appendix G – Acute WQC Fit Test for fipronil-sulfone, log- logistic**

Fipronil-sulfone—Log-logistic SSD

|    | SMAV   | Omit<br>one | 1      | 2      | 3      | 4      | 5      | 6      | 7      | 8      | 9      | 10     | 11     | 12     | 13     | 14     | 15     |
|----|--------|-------------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|
| 1  | 0.0079 |             |        | 0.0079 | 0.0079 | 0.0079 | 0.0079 | 0.0079 | 0.0079 | 0.0079 | 0.0079 | 0.0079 | 0.0079 | 0.0079 | 0.0079 | 0.0079 | 0.0079 |
| 2  | 0.0313 | 0.0313      |        |        | 0.0313 | 0.0313 | 0.0313 | 0.0313 | 0.0313 | 0.0313 | 0.0313 | 0.0313 | 0.0313 | 0.0313 | 0.0313 | 0.0313 | 0.0313 |
| 3  | 0.0474 | 0.0474      | 0.0474 |        |        | 0.0474 | 0.0474 | 0.0474 | 0.0474 | 0.0474 | 0.0474 | 0.0474 | 0.0474 | 0.0474 | 0.0474 | 0.0474 | 0.0474 |
| 4  | 0.0717 | 0.0717      | 0.0717 | 0.0717 |        |        | 0.0717 | 0.0717 | 0.0717 | 0.0717 | 0.0717 | 0.0717 | 0.0717 | 0.0717 | 0.0717 | 0.0717 | 0.0717 |
| 5  | 0.0729 | 0.0729      | 0.0729 | 0.0729 | 0.0729 |        |        | 0.0729 | 0.0729 | 0.0729 | 0.0729 | 0.0729 | 0.0729 | 0.0729 | 0.0729 | 0.0729 | 0.0729 |
| 6  | 0.0738 | 0.0738      | 0.0738 | 0.0738 | 0.0738 | 0.0738 |        |        | 0.0738 | 0.0738 | 0.0738 | 0.0738 | 0.0738 | 0.0738 | 0.0738 | 0.0738 | 0.0738 |
| 7  | 0.075  | 0.075       | 0.075  | 0.075  | 0.075  | 0.075  | 0.075  |        |        | 0.075  | 0.075  | 0.075  | 0.075  | 0.075  | 0.075  | 0.075  | 0.075  |
| 8  | 0.0926 | 0.0926      | 0.0926 | 0.0926 | 0.0926 | 0.0926 | 0.0926 | 0.0926 |        |        | 0.0926 | 0.0926 | 0.0926 | 0.0926 | 0.0926 | 0.0926 | 0.0926 |
| 9  | 0.0959 | 0.0959      | 0.0959 | 0.0959 | 0.0959 | 0.0959 | 0.0959 | 0.0959 | 0.0959 |        |        | 0.0959 | 0.0959 | 0.0959 | 0.0959 | 0.0959 | 0.0959 |
| 10 | 0.155  | 0.155       | 0.155  | 0.155  | 0.155  | 0.155  | 0.155  | 0.155  | 0.155  | 0.155  |        |        | 0.155  | 0.155  | 0.155  | 0.155  | 0.155  |
| 11 | 0.159  | 0.159       | 0.159  | 0.159  | 0.159  | 0.159  | 0.159  | 0.159  | 0.159  | 0.159  | 0.159  |        |        | 0.159  | 0.159  | 0.159  | 0.159  |
| 12 | 0.163  | 0.163       | 0.163  | 0.163  | 0.163  | 0.163  | 0.163  | 0.163  | 0.163  | 0.163  | 0.163  | 0.163  |        |        | 0.163  | 0.163  | 0.163  |
| 13 | 25     | 25          | 25     | 25     | 25     | 25     | 25     | 25     | 25     | 25     | 25     | 25     | 25     |        |        | 25     | 25     |
| 14 | 29     | 29          | 29     | 29     | 29     | 29     | 29     | 29     | 29     | 29     | 29     | 29     | 29     | 29     |        |        | 29     |
| 15 | 39     | 39          | 39     | 39     | 39     | 39     | 39     | 39     | 39     | 39     | 39     | 39     | 39     | 39     | 39     |        |        |

Fipronil-sulfone—Log-logistic SSD

|                           |          |          |          |          |          |          |          |         |         |          |          |          |          |          |          |
|---------------------------|----------|----------|----------|----------|----------|----------|----------|---------|---------|----------|----------|----------|----------|----------|----------|
| Omitted point, xi:        | 0.0079   | 0.0313   | 0.0474   | 0.0717   | 0.0729   | 0.0738   | 0.075    | 0.0926  | 0.0959  | 0.155    | 0.159    | 0.163    | 25       | 29       | 39       |
| median                    |          |          |          |          |          |          |          |         |         |          |          |          |          |          |          |
| 5th percentile            | 0.003906 | 0.002816 | 0.002707 | 0.002551 | 0.002545 | 0.002541 | 0.002536 | 0.00247 | 0.00246 | 0.002339 | 0.002333 | 0.002328 | 0.002949 | 0.003042 | 0.003254 |
| Log-logistic percentile   | 7.83     | 20.02    | 24.95    | 30.78    | 31.03    | 31.21    | 31.45    | 34.68   | 35.23   | 43.05    | 43.47    | 43.89    | 97.46    | 97.82    | 98.42    |
| F-i(xi)                   | 0.0783   | 0.2002   | 0.2495   | 0.3078   | 0.3103   | 0.3121   | 0.3145   | 0.3468  | 0.3523  | 0.4305   | 0.4347   | 0.4389   | 0.9746   | 0.9782   | 0.9842   |
| 1-F(xi)                   | 0.9217   | 0.7998   | 0.7505   | 0.6922   | 0.6897   | 0.6879   | 0.6855   | 0.6532  | 0.6477  | 0.5695   | 0.5653   | 0.5611   | 0.0254   | 0.0218   | 0.0158   |
| Min of F-i(xi) or 1-F(xi) | 0.0783   | 0.2002   | 0.2495   | 0.3078   | 0.3103   | 0.3121   | 0.3145   | 0.3468  | 0.3523  | 0.4305   | 0.4347   | 0.4389   | 0.0254   | 0.0218   | 0.0158   |
| pi =2(min)                | 0.1566   | 0.4004   | 0.499    | 0.6156   | 0.6206   | 0.6242   | 0.629    | 0.6936  | 0.7046  | 0.861    | 0.8694   | 0.8778   | 0.0508   | 0.0436   | 0.0316   |

Fipronil-sulfone—Log-logistic SSD

| p <sub>i</sub> | ln(p <sub>i</sub> ) | Fisher test statistic          |                              |
|----------------|---------------------|--------------------------------|------------------------------|
|                |                     | -2*Sum of ln (p <sub>i</sub> ) | X <sup>2</sup> <sub>2n</sub> |
| 0.1566         | -1.8541             | 32.1295                        | 0.3615                       |
| 0.4004         | -0.9153             |                                |                              |
| 0.4990         | -0.6951             |                                |                              |
| 0.6156         | -0.4852             |                                |                              |
| 0.6206         | -0.4771             |                                |                              |
| 0.6242         | -0.4713             |                                |                              |
| 0.6290         | -0.4636             |                                |                              |
| 0.6936         | -0.3659             |                                |                              |
| 0.7046         | -0.3501             |                                |                              |
| 0.8610         | -0.1497             |                                |                              |
| 0.8694         | -0.1400             |                                |                              |
| 0.8778         | -0.1303             |                                |                              |
| 0.0508         | -2.9799             |                                |                              |
| 0.0436         | -3.1327             |                                |                              |
| 0.0316         | -3.4546             |                                |                              |

if X<sup>2</sup> < 0.05      significant lack of fit  
 if X<sup>2</sup> > 0.05      fit (no significant lack of fit)