

MEMORANDUM

To: Bay Area Clean Water Agencies
From: Jennifer Jackson, Jackson Environmental Consulting
Date: January 6, 2014
Subject: Fipronil in Wastewater, Summary of Available Data & Information

Fipronil is an insecticide registered for use to control ants, cockroaches, fleas, termites and other insects both indoors and outdoors in urban areas. It is highly toxic to aquatic species with observable effects known to occur at concentrations as low as 5 parts per trillion (U.S. EPA OPP 2011). As summarized below, Fipronil and its degradates have been detected in wastewater influent, effluent, and biosolids. Though data are limited, detection frequencies exceeded 80% in low detection limit studies. Most fipronil (>80%) appears to pass through municipal wastewater treatment plants; to the extent removal occurs, it appears to involve transfer to biosolids. In effluent, concentrations of fipronil and its degradates approached and in some cases exceeded effect thresholds for sensitive species.

Residential & Urban Uses

Fipronil has limited registered uses in residential and urban settings, including flea and tick treatments for pets, gels for crack and crevice treatment, ant and cockroach baits, outdoor perimeter sprays for ant control, and outdoor subsurface/soil injection for termites control. Some of these uses have the potential to impact wastewater, though the pathways to the sanitary sewer may not be immediately obvious. For example, flea treatment for pets involves applying a gel into pets' fur; transfer of fipronil to wastewater may occur when the pet owner washes his hands after applying the gel or after subsequent contacts with the pet, and when the animal is bathed (e.g., by a groomer), and/or when pet bedding, carpets, and other surfaces contacted by the pet are washed. A 2009 study by Mahler, et al. showed that house dust in homes where pets have been flea-treated had higher concentrations of fipronil than homes where pets had not been treated. Another 2009 study, by Stout, et al. showed that 40% of household floors were contaminated with fipronil. If contaminated indoor surfaces are cleaned, there can be an indirect pathway to the sanitary sewer, whenever rags, mops and sponges are washed out. The use of gels to treat cracks and crevices may also be a source of fipronil in wastewater, though again, the pathway is indirect: wastewater may become contaminated with fipronil when hands, rags, sponges and other tools are washed.

Soil injection to control termites and perimeter sprays to control ants present potential indirect pathways to the sanitary sewer. If a compromised sewer line passes through soil that is being or has been injected with fipronil, there is the potential for fipronil to infiltrate the sewer system. While this pathway has not been proven, Shuai et al. (2011) showed that fipronil can be mobile in soil, particularly when the organic carbon content of the soil is low. Fipronil may be tracked indoors after outdoor applications. A 2009 study by Mahler et al. showed that if a termite treatment had occurred in the prior two years (applications via trench or soil injection), indoor house dust had higher concentrations of fipronil than those homes that had not had treatments. As noted above, housecleaning may transfer dust containing fipronil to wastewater when rags and mops are washed.

Fipronil & Degradates Wastewater Monitoring Data

A literature review was conducted and California POTWs were informally surveyed to obtain wastewater fipronil monitoring data. Very limited data were identified, including results from multiple samples collected by one California Publicly Owned Treatment Works (POTW), a US Geological Survey (USGS) sampling survey of nine Columbia River POTWs (Morace 2012), and a national POTW sampling study conducted by researchers from Johns Hopkins University that provided fipronil data from nine POTWs (Heidler and Halden 2009). These data are summarized in Tables 1, 2, and 3. Table 4 summarizes results from each sample from the California POTW and Morace (2012).

The California POTW data are unpublished. The California POTW collected six whole effluent grab samples between March and September 2013. The samples were analyzed by Caltest Analytical Laboratory for fipronil and three degradates – fipronil desulfinyl, fipronil sulfide and fipronil sulfone. Chemical analysis employed methods previously developed for pyrethroid insecticides (see Markle and Van Buuren 2013), involving sample preparation by EPA Method SW846 3510C and chemical analysis by modified EPA method 8270 (gas chromatography/mass spectroscopy using negative chemical ionization mode, with selective ion monitoring mode of detection and quantification). The laboratory reporting limit was 1.5 ng/L for each analyte.

Morace (2012) describes the sampling of nine Oregon and Washington POTWs, which was conducted by a US Geological Survey (USGS) reconnaissance team. USGS sampling procedures are described in in the full report (Morace 2012). Unlike most POTW samples (which are analyzed as whole water samples that include both suspended solids and water), the USGS samples were filtered on-site, separating the suspended solids from the water prior to analysis. USGS analyzed the solids separately from the liquid fraction. Effluent water was analyzed for fipronil, fipronil desulfinyl, fipronil sulfide and fipronil sulfone. Effluent solids analyses included fipronil and only two degradates, fipronil desulfinyl and fipronil sulfide. Notably, filtered

***Fipronil in Wastewater: A Summary of Available Data & Information
December 2013***

effluent solids are not biosolids or sewage sludge, as filtered effluent solids were suspended in the water column and were not subjected to solids treatment. Results for the solids and water were reported separately; the USGS did not sum the data to estimate total effluent concentrations. In Tables 1 and 2, only the USGS water phase data are included in the “wastewater effluent” summary. Tables 1 and 2 summarize the USGS solids data on the basis of solids-associated fipronil concentration per liter of water in the sample. From these data, it appears that negligible quantities of fipronil had partitioned onto solids in these samples.

Table 1. Summary of Fipronil in Effluent and Effluent Solids* of Ten POTWs

	Wastewater Effluent <i>(15 data points)</i>	Effluent Solids* <i>(10 data points)</i>
Samples with fipronil detections	87%	90%
Mean Concentration Detected ^a	36 ng/L	0.5 ng/L
Median Concentration Detected	16 ng/L	0.2 ng/L
Range of Detected Concentrations	ND – 130 ng/L	ND – 1.4 ng/L
EPA Benchmark	11 ng/L	Not applicable
Lowest published toxicity data ^b	LC 50 = 140 ng/L LOEC = 5 ng/L <i>Americamysis bahia</i>	Not applicable

* Solids-associated fipronil concentration per liter of water in the sample. Solids are filtered out of effluent; they are not biosolids/sewage sludge.

^a Mean and median calculations included all numeric estimates issued by the laboratory. The calculation did not include samples where presence was determined but not estimated nor quantified with a numeric value.

^b U.S. EPA OPP 2011.

Table 2. Summary of Fipronil Degradates in Effluent and Effluent Solids* of Ten POTWs

	Wastewater Effluent <i>(45 data points)</i>	Effluent Solids* <i>(22 data points)</i>
Samples with fipronil detections	82%	82%
Mean Concentration Detected ^a	14 ng/L (Total concentration of all measured degradates)	0.06 ng/L (Total concentration of all measured degradates)
Median Concentration Detected ^a	13 ng/L (Total concentration of all measured degradates)	0.04 ng/L (Total concentration of all measured degradates)
Range of Detected Concentrations	ND – 24 ng/L (Individual degradates)	ND – 0.08 ng/L (Individual degradates)
EPA Benchmark ^b	37-590 ng/L	Not applicable
Lowest published toxicity data ^c	LC 50 = 77-1500 ng/L LOEC = 2.6 – 8.7 ng/L <i>Americamysis bahia</i>	Not applicable

* Solids-associated fipronil concentration per liter of water in the sample. Solids are filtered out of effluent; these are not biosolids/sewage sludge.

^a Mean and median calculations included all numeric estimates issued by the laboratory. The calculation did not include samples where presence was determined but not estimated nor quantified with a numeric value.

^b Aquatic invertebrates, chronic

^c U.S. EPA OPP 2011.

The 2009 Johns Hopkins study measured fipronil in influent, effluent and biosolids. Degradates were not measured in this study. Methods are detailed in Heidler and Halden (2009). Unlike the two above studies, Heidler and Halden collected 24-hour composite samples. Although the study involved 25 POTWs across the country, samples from only nine POTWs were analyzed for fipronil. Effluent and biosolids data from this study are summarized in Table 3. Heidler and Halden also calculated mass balances for fipronil, which showed that most fipronil passes

through wastewater treatment systems. Measured fipronil reductions between influent and effluent averaged <20%. Removal from the water phase appeared to occur primarily as a consequence of partitioning into biosolids.

Table 3. Summary of Heidler & Halden (2009) Results

	Wastewater Effluent <i>(9 data points)</i>	Biosolids* <i>(9 data points)</i>
Detection of fipronil	100%	100%
Median Detection Concentration	30 ng/L	60 µg/kg (anaerobic digestion) 130 µg/kg (aerobic digestion)
Range of Detected Concentrations	<10 – 70 ng/L	3 – 160 µg/kg
EPA Benchmark ^a	11 ng/L	Not applicable
Lowest published toxicity data ^b	LC 50 = 140 ng/L LOEC = 5 ng/L <i>Americamysis bahia</i>	

*Digested biosolids, prior to dewatering, wet weight.

^a Aquatic invertebrates, chronic

^b U.S. EPA OPP 2011.

Another study conducted by Hope, et al. of the Oregon Department of Environmental Quality surveyed 52 Oregon POTWs for 406 persistent organic compounds, including fipronil. Fipronil was not detected in any of the samples, likely due to the high detection limit of 10,000 ng/L, which is three orders of magnitude higher than the above studies' detection limits and higher than the maximum fipronil detections reported in the above studies.

Finally, Weston and Lydy have a forthcoming study that includes fipronil monitoring data for two California POTWs as well as new fipronil toxicity data.

References

- Heidler, J. and R. Halden. 2009. "Fate of organohalogens in US wastewater treatment plants and estimated chemical releases to soils nationwide from biosolids recycling." *Journal of Environmental Monitoring* 11(12): 2207–2215.
- Hope, B.K., L. Pillsbury and B. Boling. 2012. "A State-Wide Survey in Oregon (USA) of Trace Metals and Organic Chemicals in Municipal Effluent." *Science of the Total Environment* 417-418:263-72.
- Mahler, B.J., P.C. VanMetre, J.T. Wilson, M. Musgrove, S.D. Zauggand and M.R. Burkhardt. 2009. "Fipronil and its Degradates in Indoor and Outdoor Dust." *Environmental Science & Technology* 43(15): 5665–5670.
- Markle, J. and B. H. van Buuren. 2013. Pyrethroid Pesticides in Municipal Wastewater: A Baseline Survey of Publicly Owned Treatment Works Facilities In California 2011-2013. Prepared for the Pyrethroid Working Group.
- Morace, J.L. 2012. Reconnaissance of contaminants in selected wastewater-treatment-plant effluent and stormwater runoff entering the Columbia River, Columbia River Basin, Washington and Oregon, 2008–10: U.S. Geological Survey Scientific Investigations Report 2012–5068, 68 p.
- Shuai, X., J. Chen, R. Chittaranjan. 2011. "Adsorption, transport and degradation of fipronil termiticide in three Hawaii soils." *Pest Management Science* 68: 731–739.
- Stout, D.M., K.D. Bradham, P.P. Egeghy, P. Jones, C.W. Croghan, P.A. Ashley, E. Pinzer, W. Friedman, M.C. Brinkman, M.G. Nishioka and D.C. Cox. 2009. "American Healthy Homes Survey: A National Study of Residential Pesticides Measured from Floor Wipes." *Environmental Science & Technology* 43(12): 4294–4300.
- U.S. EPA Office of Pesticide Programs (OPP). 2011. Problem Formulation for the Environmental Fate and Ecological Risk, Endangered Species, and Drinking Water Assessments in Support of the Registration Review of Fipronil.

Table 4. Fipronil Monitoring Data for 10 POTWs

POTW #	Location	Treatment	Matrix	Constituent	Sample Type	Container	Number of Liters Filtered	Sample Date	MDL (ng/L)	RL (ng/L)	Result (ng/L)	Qualifiers
1	Confidential CA POTW	Advanced secondary with fixed-film reactor nitrification, dissolved air flotation, dual media filtration, chlorine disinfection and dechlorination.	Whole Effluent	fipronil	composite	glass		April 9-15, 2013	0.5	1.5	4.2	
1	Confidential CA POTW	Advanced secondary with fixed-film reactor nitrification, dissolved air flotation, dual media filtration, chlorine disinfection and dechlorination.	Whole Effluent	fipronil	composite	glass		April 22-28, 2013	0.5	1.5	5	
1	Confidential CA POTW	Advanced secondary with fixed-film reactor nitrification, dissolved air flotation, dual media filtration, chlorine disinfection and dechlorination.	Whole Effluent	fipronil	composite	glass		May 6-12, 2013	0.5	1.5	4.1	
1	Confidential CA POTW	Advanced secondary with fixed-film reactor nitrification, dissolved air flotation, dual media filtration, chlorine disinfection and dechlorination.	Whole Effluent	fipronil	composite	glass		May 20-26, 2013	0.5	1.5	1	J
1	Confidential CA POTW	Advanced secondary with fixed-film reactor nitrification, dissolved air flotation, dual media filtration, chlorine disinfection and dechlorination.	Whole Effluent	fipronil	composite	glass		July 15-21, 2013	0.5	1.5	1.1	J
1	Confidential CA POTW	Advanced secondary with fixed-film reactor nitrification, dissolved air flotation, dual media filtration, chlorine disinfection and dechlorination.	Whole Effluent	fipronil	composite	glass		Sept 9 - 15, 2013	0.5	1.5	1.9	
1	Confidential CA POTW	Advanced secondary with fixed-film reactor nitrification, dissolved air flotation, dual media filtration, chlorine disinfection and dechlorination.	Whole Effluent	fipronil desulfinyl	composite	glass		April 9-15, 2013	0.5	1.5	3.9	
1	Confidential CA POTW	Advanced secondary with fixed-film reactor nitrification, dissolved air flotation, dual media filtration, chlorine disinfection and dechlorination.	Whole Effluent	fipronil desulfinyl	composite	glass		April 22-28, 2013	0.5	1.5	5	

**Fipronil in Wastewater: A Summary of Available Data & Information
December 2013**

1	Confidential CA POTW	Advanced secondary with fixed-film reactor nitrification, dissolved air flotation, dual media filtration, chlorine disinfection and dechlorination.	Whole Effluent	fipronil desulfinyl	composite	glass		May 6-12, 2013	0.5	1.5	4.4	
1	Confidential CA POTW	Advanced secondary with fixed-film reactor nitrification, dissolved air flotation, dual media filtration, chlorine disinfection and dechlorination.	Whole Effluent	fipronil desulfinyl	composite	glass		May 20-26, 2013	0.5	1.5	1.4	J
1	Confidential CA POTW	Advanced secondary with fixed-film reactor nitrification, dissolved air flotation, dual media filtration, chlorine disinfection and dechlorination.	Whole Effluent	fipronil desulfinyl	composite	glass		July 15-21, 2013	0.5	1.5	2.3	
1	Confidential CA POTW	Advanced secondary with fixed-film reactor nitrification, dissolved air flotation, dual media filtration, chlorine disinfection and dechlorination.	Whole Effluent	fipronil desulfinyl	composite	glass		Sept 9 - 15, 2013	0.5	1.5	1.8	
1	Confidential CA POTW	Advanced secondary with fixed-film reactor nitrification, dissolved air flotation, dual media filtration, chlorine disinfection and dechlorination.	Whole Effluent	fipronil sulfide	composite	glass		April 9-15, 2013	0.5	1.5	5.1	
1	Confidential CA POTW	Advanced secondary with fixed-film reactor nitrification, dissolved air flotation, dual media filtration, chlorine disinfection and dechlorination.	Whole Effluent	fipronil sulfide	composite	glass		April 22-28, 2013	0.5	1.5	6.3	
1	Confidential CA POTW	Advanced secondary with fixed-film reactor nitrification, dissolved air flotation, dual media filtration, chlorine disinfection and dechlorination.	Whole Effluent	fipronil sulfide	composite	glass		May 6-12, 2013	0.5	1.5	6.1	
1	Confidential CA POTW	Advanced secondary with fixed-film reactor nitrification, dissolved air flotation, dual media filtration, chlorine disinfection and dechlorination.	Whole Effluent	fipronil sulfide	composite	glass		May 20-26, 2013	0.5	1.5	1.6	
1	Confidential CA POTW	Advanced secondary with fixed-film reactor nitrification, dissolved air flotation, dual media filtration, chlorine disinfection and dechlorination.	Whole Effluent	fipronil sulfide	composite	glass		July 15-21, 2013	0.5	1.5	3.5	

**Fipronil in Wastewater: A Summary of Available Data & Information
December 2013**

1	Confidential CA POTW	Advanced secondary with fixed-film reactor nitrification, dissolved air flotation, dual media filtration, chlorine disinfection and dechlorination.	Whole Effluent	fipronil sulfide	composite	glass		Sept 9 - 15, 2013	0.5	1.5	3.3	
1	Confidential CA POTW	Advanced secondary with fixed-film reactor nitrification, dissolved air flotation, dual media filtration, chlorine disinfection and dechlorination.	Whole Effluent	fipronil sulfone	composite	glass		April 9-15, 2013	0.5	1.5	2.2	
1	Confidential CA POTW	Advanced secondary with fixed-film reactor nitrification, dissolved air flotation, dual media filtration, chlorine disinfection and dechlorination.	Whole Effluent	fipronil sulfone	composite	glass		April 22-28, 2013	0.5	1.5	3.1	
1	Confidential CA POTW	Advanced secondary with fixed-film reactor nitrification, dissolved air flotation, dual media filtration, chlorine disinfection and dechlorination.	Whole Effluent	fipronil sulfone	composite	glass		May 6-12, 2013	0.5	1.5	3.5	
1	Confidential CA POTW	Advanced secondary with fixed-film reactor nitrification, dissolved air flotation, dual media filtration, chlorine disinfection and dechlorination.	Whole Effluent	fipronil sulfone	composite	glass		May 20-26, 2013	0.5	1.5	0.9	J
1	Confidential CA POTW	Advanced secondary with fixed-film reactor nitrification, dissolved air flotation, dual media filtration, chlorine disinfection and dechlorination.	Whole Effluent	fipronil sulfone	composite	glass		July 15-21, 2013	0.5	1.5	2.3	
1	Confidential CA POTW	Advanced secondary with fixed-film reactor nitrification, dissolved air flotation, dual media filtration, chlorine disinfection and dechlorination.	Whole Effluent	fipronil sulfone	composite	glass		Sept 9 - 15, 2013	0.5	1.5	1.7	
2	Wenatchee, WA	Activated sludge plant, secondary treatment, UV disinfection	Filtered Effluent	fipronil	grab			12/1/2009	9	18	ND	
2	Wenatchee, WA	Activated sludge plant, secondary treatment, UV disinfection	Filtered Effluent	fipronil desulfinyl	grab			12/1/2009	6	12	ND	
2	Wenatchee, WA	Activated sludge plant, secondary treatment, UV disinfection	Filtered Effluent	fipronil sulfide	grab			12/1/2009	6	13	ND	
2	Wenatchee, WA	Activated sludge plant, secondary treatment, UV disinfection	Filtered Effluent	fipronil sulfone	grab			12/1/2009	12	24	ND	

**Fipronil in Wastewater: A Summary of Available Data & Information
December 2013**

2	Wenatchee, WA	Activated sludge plant, secondary treatment, UV disinfection	Solids filtered from effluent	fipronil	grab		21	12/1/2009		0.005	ND	
2	Wenatchee, WA	Activated sludge plant, secondary treatment, UV disinfection	Solids filtered from effluent	fipronil desulfinyl	grab		21	12/1/2009		0.005	ND	
2	Wenatchee, WA	Activated sludge plant, secondary treatment, UV disinfection	Solids filtered from effluent	fipronil sulfide	grab		21	12/1/2009		0.005	Present	
3	Richland, WA	Semi-plug flow conventional activated sludge, secondary clarification, chlorination	Filtered Effluent	fipronil	grab			12/2/2009	9	18	27	
3	Richland, WA	Semi-plug flow conventional activated sludge, secondary clarification, chlorination	Filtered Effluent	fipronil desulfinyl	grab			12/2/2009	6	12	Present	
3	Richland, WA	Semi-plug flow conventional activated sludge, secondary clarification, chlorination	Filtered Effluent	fipronil sulfide	grab			12/2/2009	6	13	Present	
3	Richland, WA	Semi-plug flow conventional activated sludge, secondary clarification, chlorination	Filtered Effluent	fipronil sulfone	grab			12/2/2009	12	24	Present	
3	Richland, WA	Semi-plug flow conventional activated sludge, secondary clarification, chlorination	Solids filtered from effluent	fipronil	grab		19	12/2/2009		0.005	0.22	
3	Richland, WA	Semi-plug flow conventional activated sludge, secondary clarification, chlorination	Solids filtered from effluent	fipronil desulfinyl	grab		19	12/2/2009		0.005	Present	
3	Richland, WA	Semi-plug flow conventional activated sludge, secondary clarification, chlorination	Solids filtered from effluent	fipronil sulfide	grab		19	12/2/2009		0.005	0.03	
4	Umatilla, OR	Oxidation ditch, UV disinfection	Filtered Effluent	fipronil	grab			12/2/2009	9	18	Present	
4	Umatilla, OR	Oxidation ditch, UV disinfection	Filtered Effluent	fipronil desulfinyl	grab			12/2/2009	6	12	Present	
4	Umatilla, OR	Oxidation ditch, UV disinfection	Filtered Effluent	fipronil sulfide	grab			12/2/2009	6	13	Present	
4	Umatilla, OR	Oxidation ditch, UV disinfection	Filtered Effluent	fipronil sulfone	grab			12/2/2009	12	24	ND	
4	Umatilla, OR	Oxidation ditch, UV disinfection	Solids filtered from	fipronil	grab		18	12/2/2009		0.006	0.06	

**Fipronil in Wastewater: A Summary of Available Data & Information
December 2013**

			effluent					9				
4	Umatilla, OR	Oxidation ditch, UV disinfection	Solids filtered from effluent	fipronil desulfinyl	grab		18	12/2/2009		0.006	Present	
4	Umatilla, OR	Oxidation ditch, UV disinfection	Solids filtered from effluent	fipronil sulfide	grab		18	12/2/2009		0.006	0.02	
5	The Dalles, OR	Activated sludge plant, UV disinfection	Filtered Effluent	fipronil	grab			12/2/2009	9	18	42	E
5	The Dalles, OR	Activated sludge plant, UV disinfection	Filtered Effluent	fipronil desulfinyl	grab			12/2/2009	6	12	Present	
5	The Dalles, OR	Activated sludge plant, UV disinfection	Filtered Effluent	fipronil sulfide	grab			12/2/2009	6	13	Present	
5	The Dalles, OR	Activated sludge plant, UV disinfection	Filtered Effluent	fipronil sulfone	grab			12/2/2009	12	24	ND	
5	The Dalles, OR	Activated sludge plant, UV disinfection	Solids filtered from effluent	fipronil	grab		18	12/2/2009		0.006	0.17	
5	The Dalles, OR	Activated sludge plant, UV disinfection	Solids filtered from effluent	fipronil desulfinyl	grab		18	12/2/2009		0.006	0.02	
5	The Dalles, OR	Activated sludge plant, UV disinfection	Solids filtered from effluent	fipronil sulfide	grab		18	12/2/2009		0.006	Present	
6	Hood River, OR	Activated sludge plant, UV disinfection	Filtered Effluent	fipronil	grab			12/2/2009	9	18	56	E
6	Hood River, OR	Activated sludge plant, UV disinfection	Filtered Effluent	fipronil desulfinyl	grab			12/2/2009	6	12	Present	
6	Hood River, OR	Activated sludge plant, UV disinfection	Filtered Effluent	fipronil sulfide	grab			12/2/2009	6	13	Present	
6	Hood River, OR	Activated sludge plant, UV disinfection	Filtered Effluent	fipronil sulfone	grab			12/2/2009	12	24	Present	
6	Hood River, OR	Activated sludge plant, UV disinfection	Solids filtered from effluent	fipronil	grab		19	12/2/2009		0.005	0.2	
6	Hood River, OR	Activated sludge plant, UV disinfection	Solids filtered from effluent	fipronil desulfinyl	grab		19	12/2/2009		0.005	0.02	
6	Hood River, OR	Activated sludge plant, UV disinfection	Solids filtered from effluent	fipronil sulfide	grab		19	12/2/2009		0.005	0.02	
7	Portland, OR	Conventional activated sludge, secondary clarification, chlorine disinfection	Filtered Effluent	fipronil	grab			12/10/2009	9	18	78	E
7	Portland, OR	Conventional activated sludge, secondary	Filtered Effluent	fipronil	grab			12/10/20	6	12	ND	

**Fipronil in Wastewater: A Summary of Available Data & Information
December 2013**

		clarification, chlorine disinfection		desulfinyl				09				
7	Portland, OR	Conventional activated sludge, secondary clarification, chlorine disinfection	Filtered Effluent	fipronil sulfide	grab			12/10/2009	6	13	Present	
7	Portland, OR	Conventional activated sludge, secondary clarification, chlorine disinfection	Filtered Effluent	fipronil sulfone	grab			12/10/2009	12	24	ND	
7	Portland, OR	Conventional activated sludge, secondary clarification, chlorine disinfection	Solids filtered from effluent	fipronil	grab		20	12/10/2009		0.005	0.99	
7	Portland, OR	Conventional activated sludge, secondary clarification, chlorine disinfection	Solids filtered from effluent	fipronil	grab		10	12/10/2009		0.01	0.35	
7	Portland, OR	Conventional activated sludge, secondary clarification, chlorine disinfection	Solids filtered from effluent	fipronil	grab		9	12/10/2009		0.01	0.77	
7	Portland, OR	Conventional activated sludge, secondary clarification, chlorine disinfection	Solids filtered from effluent	fipronil desulfinyl	grab		20	12/10/2009		0.005	Present	
7	Portland, OR	Conventional activated sludge, secondary clarification, chlorine disinfection	Solids filtered from effluent	fipronil desulfinyl	grab		10	12/10/2009		0.01	ND	
7	Portland, OR	Conventional activated sludge, secondary clarification, chlorine disinfection	Solids filtered from effluent	fipronil desulfinyl	grab		9	12/10/2009		0.01	Present	
7	Portland, OR	Conventional activated sludge, secondary clarification, chlorine disinfection	Solids filtered from effluent	fipronil sulfide	grab		20	12/10/2009		0.005	0.06	
7	Portland, OR	Conventional activated sludge, secondary clarification, chlorine disinfection	Solids filtered from effluent	fipronil sulfide	grab		10	12/10/2009		0.01	0.02	
7	Portland, OR	Conventional activated sludge, secondary clarification, chlorine disinfection	Solids filtered from effluent	fipronil sulfide	grab		9	12/10/2009		0.01	0.06	
8	Vancouver, WA	Industrial pretreatment lagoon, secondary activated sludge, UV disinfection, sludge incineration	Filtered Effluent	fipronil	grab			12/2/2009	9	18	87	E
8	Vancouver, WA	Industrial pretreatment lagoon, secondary activated sludge, UV disinfection, sludge incineration	Filtered Effluent	fipronil desulfinyl	grab			12/2/2009	6	12	Present	
8	Vancouver, WA	Industrial pretreatment lagoon, secondary activated sludge, UV disinfection, sludge incineration	Filtered Effluent	fipronil sulfide	grab			12/2/2009	6	13	Present	
8	Vancouver, WA	Industrial pretreatment lagoon, secondary activated sludge, UV disinfection, sludge incineration	Filtered Effluent	fipronil sulfone	grab			12/2/2009	12	24	Present	
8	Vancouver, WA	Industrial pretreatment lagoon, secondary	Solids filtered from	fipronil	grab		18	12/2/200		0.006	1.4	

**Fipronil in Wastewater: A Summary of Available Data & Information
December 2013**

		activated sludge, UV disinfection, sludge incineration	effluent					9				
8	Vancouver, WA	Industrial pretreatment lagoon, secondary activated sludge, UV disinfection, sludge incineration	Solids filtered from effluent	fipronil desulfinyl	grab		18	12/2/2009		0.006	0.07	
8	Vancouver, WA	Industrial pretreatment lagoon, secondary activated sludge, UV disinfection, sludge incineration	Solids filtered from effluent	fipronil sulfide	grab		18	12/2/2009		0.006	0.08	
9	St. Helens, OR	Combined municipal and kraft mill aerated stabilization basin	Filtered Effluent	fipronil	grab			12/3/2009	9	18	< 47	
9	St. Helens, OR	Combined municipal and kraft mill aerated stabilization basin	Filtered Effluent	fipronil desulfinyl	grab			12/3/2009	6	12	Present	
9	St. Helens, OR	Combined municipal and kraft mill aerated stabilization basin	Filtered Effluent	fipronil sulfide	grab			12/3/2009	6	13	22	
9	St. Helens, OR	Combined municipal and kraft mill aerated stabilization basin	Filtered Effluent	fipronil sulfone	grab			12/3/2009	12	24	ND	
9	St. Helens, OR	Combined municipal and kraft mill aerated stabilization basin	Solids filtered from effluent	fipronil	grab		12	12/3/2009		0.008	Not determined	Not determined due to poor compound recoveries
9	St. Helens, OR	Combined municipal and kraft mill aerated stabilization basin	Solids filtered from effluent	fipronil desulfinyl	grab		12	12/3/2009		0.008	ND	
9	St. Helens, OR	Combined municipal and kraft mill aerated stabilization basin	Solids filtered from effluent	fipronil sulfide	grab		12	12/3/2009		0.008	0.04	
10	Longview, WA	Conventional activated sludge, secondary clarification, chlorine disinfection, dechlorination	Filtered Effluent	fipronil	grab			12/8/2009	9	18	130	E
10	Longview, WA	Conventional activated sludge, secondary clarification, chlorine disinfection, dechlorination	Filtered Effluent	fipronil desulfinyl	grab			12/8/2009	6	12	Present	
10	Longview, WA	Conventional activated sludge, secondary clarification, chlorine disinfection, dechlorination	Filtered Effluent	fipronil sulfide	grab			12/8/2009	6	13	6	E
10	Longview, WA	Conventional activated sludge, secondary clarification, chlorine disinfection, dechlorination	Filtered Effluent	fipronil sulfone	grab			12/8/2009	12	24	24	

***Fipronil in Wastewater: A Summary of Available Data & Information
December 2013***

10	Longview, WA	Conventional activated sludge, secondary clarification, chlorine disinfection, dechlorination	Solids filtered from effluent	fipronil	grab		20	12/8/2009		0.005	0.05	
10	Longview, WA	Conventional activated sludge, secondary clarification, chlorine disinfection, dechlorination	Solids filtered from effluent	fipronil desulfinyl	grab		20	12/8/2009		0.005	ND	
10	Longview, WA	Conventional activated sludge, secondary clarification, chlorine disinfection, dechlorination	Solids filtered from effluent	fipronil sulfide	grab		20	12/8/2009		0.005	0.01	

Qualifiers

J - reflects estimated analytical result value detected below the Reporting Limit and above the Method Detection Limit. The J flag is also referred to as DNQ (Detected but not quantified).

E – Estimated

ND - Not Detected

Present - Presence is verified, but concentration is not quantified

(<) - Sometimes the reporting limit for an individual sample is raised because of matrix interference, these instances of non-detection are shown as less than (<) the raised reporting limit.