

October 31, 2002

To: Water Resources Discipline

From: Peter F. Rogerson, Senior Chemist  
Office of Water Quality

For: LeRoy Schroder, Chief  
Branch of Quality Systems

Subject: Approval of a Water Quality Analytical Method Supplement for the Determination of Fipronil and Degradates in Water by the National Water Quality Laboratory

The Office of Water Quality (OWQ) has approved a Water Quality Analytical Method Supplement developed by the National Water Quality Laboratory (NWQL) for the determination of the insecticide fipronil and 4 degradates in filtered water samples. This method supplement adds fipronil and 4 degradates to U.S. Geological Survey O-1126-95 which is available from the National Water Quality Laboratory (NWQL) as Schedules 2001 (laboratory extraction) and 2010 (field extraction). Information about Schedules 2001, 2010, along with the new analytes, are available from the NWQL by following the LIMS Catalog link shown below. The method is based on solid-phase extraction of a 1 liter filtered water sample, followed by gas chromatography/mass spectrometric analysis of the extract. The 5 new analytes are:

Fipronil and Degradates Method Supplement (O-1126-02)

Parameter Code (NWIS)	Method Code (lab extract)	Method Code (field extract)	Analyte Name
62166	B	C	Fipronil, water, filtered, ug/L
62167	B	C	Fipronil sulfide, water, filtered, ug/L
62168	B	C	Fipronil sulfone, water, filtered, ug/L
62170	B	C	Desulfinylfipronil, water, filtered, ug/L
62169	B	C	Desulfinylfipronil amide, water, filtered, ug/L

Estimated method detection limits for the 5 analytes are between 0.002 and 0.004 ug/L, with estimated reporting limits between 0.004 and 0.009 ug/L. Please note that two of the analytes, fipronil and desulfinylfipronil amide, have enhanced recoveries due to a surface-water matrix effect. Therefore, results for these two compounds are qualified with an estimated "E" remark code.

Although these compounds are included in laboratory spike samples, they are not now included in the field spike solution available from the NWQL. The NWQL will attempt to include fipronil in future field-spike lots.

This method approval process follows the technical procedure specified in OWQ Tech Memo 98.05. The method performance is described in:

Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory – A Method Supplement for the Determination of Fipronil and Degradates in Water by Gas Chromatography/Mass Spectrometry, by James E. Madsen, Mark W. Sandstrom, and Steven D. Zaugg. U.S. Geological Survey Open File Report 02-XXXX (number to be assigned upon Director's approval).

When approved by the Director, the report will be made available through the NWQL web site at: <http://wwwnwql.cr.usgs.gov/USGS/pubs.html> .

Information about this method supplement is available through the NWQL web site <http://wwwnwql.cr.usgs.gov/USGS> . Please click on LIMS Catalog and request Schedule 2001 or 2010.

If you have questions about the new analytical method, or would like a copy of the report, when it is available, please contact Mike Schroeder ([schroede@usgs.gov](mailto:schroede@usgs.gov) , (303) 236-3270) at the NWQL.

If you have questions about the method approval process, please contact Pete Rogerson ([rogerson@usgs.gov](mailto:rogerson@usgs.gov) , (303) 236-1836).