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Mail Stop 412

July 24, 2009

To: GS-W-ALL

From: Terry L. Schertz
Acting Chief, Office of Water Quality

Subject: Approval of Method for the Determination of Pyrethroid Insecticides in Water and Sediment Using Gas Chromatography/Mass Spectrometry (USGS Method Numbers O-2143-09 for water and O-6143-09 for sediment)

The Office of Water Quality has approved a new analytical method developed by the Pesticide Fate Research Group at the California Water Science Center for the determination of fourteen pyrethroid insecticides by Gas Chromatography/Mass Spectrometry (Table 1) in filtered water (USGS Method Number O-2143-09) and in soils and bed and suspended sediments (USGS Method Number O-6143-09).

The pyrethroids included in this method are applied to many agricultural and urban areas. The method was developed by the U.S. Geological Survey (USGS) in response to increasing concern about the potential effects of these insecticides on aquatic organisms.

Filtered water samples are extracted using solid-phase extraction (SPE) with no additional cleanup steps. Sediment and soil samples are extracted using a microwave-assisted extraction system. The pyrethroids of interest then are separated from co-extracted matrix interferences by passing the extracts through stacked graphitized carbon and alumina SPE cartridges, along with the use of high-performance liquid chromatography and gel-permeation chromatography (HPLC/GPC). Gas chromatography with mass spectrometry (GC/MS) or gas chromatography with tandem mass spectrometry (GC/MS/MS) is used to quantify the pyrethroids from the extracted water and sediment samples.

Method detection limits (MDLs) in water ranged from 2.0 to 6.0 nanograms per liter (ng/L) using GC/MS and 0.5 to 1.0 ng/L using GC/MS/MS. For sediment, the MDLs ranged from 1.0 to 2.6 micrograms per kilogram ($\mu\text{g/kg}$) dry weight using GC/MS and 0.2 to 0.5 $\mu\text{g/kg}$ dry weight using GC/MS/MS.

This method is described in USGS Techniques and Methods Report 5-C2:
Hladik, M.L., Smalling, K.L., and Kuivila, K.M., 2009, Methods of analysis—determination of pyrethroid insecticides in water and sediment using gas chromatography/mass spectrometry: U.S. Geological Survey Techniques and Methods 5-C2.

When published, the report will be made available through the USGS Publications Warehouse.

If you have any questions about the new analytical method, or would like a copy of the report, please contact Michelle Hladik (mhladik@usgs.gov, 916-278-3183) or Kathryn Kuivila (kkuivila@usgs.gov, 916-278-3054). For questions about the method approval process, please contact Terry Schertz (tschertz@usgs.gov, 703-648-6864)

Table 1. CAS number, molecular weight, and USGS parameter codes for each pyrethroid.

This report contains Chemical Abstracts Service Registry Numbers (CASRN), which is a Registered Trademark of [CAS, Chemical Abstracts Service; amu, atomic mass unit]

Pyrethroid	CAS number ¹	Molecular weight (amu)	Water parameter code ²	Bed-sediment parameter code	Suspended-sediment parameter code	Soil Parameter Code
Allethrin	584-79-2	302.41	66586	66588	66587	67541
Bifenthrin	82657-04-3	422.87	65067	64151	63415	67545
Cyfluthrin	68359-37-5	434.27	65074	65109	65122	67569
λ-Cyhalothrin	91456-08-6	449.86	65086	64162	65134	67674
Cypermethrin	52315-07-8	416.30	65075	64156	65123	67571
Deltamethrin	52918-63-5	505.24	65077	65110	65125	67581
Esfenvalerate	66230-04-4	419.91	65081	64159	65129	67601
Fenpropathrin	39515-41-8	349.42	65083	65111	65131	67631
τ-Fluvalinate	102851-06-9	502.93	65106	65114	65148	67727
Permethrin	52645-53-1	391.29	65099	64168	65143	67695
Resmethrin	10453-86-8	338.45	65104	65113	65147	67723
Sumithrin (phenothrin)	26002-80-2	350.46	65100	65112	65144	67697
Tefluthrin	79538-32-2	418.74	67731	67733	67732	67734
Tetramethrin	7696-12-0	331.41	66657	66659	66658	67738

¹ The American Chemical Society Chemical Abstracts Service (CAS) recommends the verification of the CASRNs through CAS Client Services.

² The five-digit parameter codes shown are used by the U.S. Geological Survey (USGS) to uniquely identify a specific constituent or property in the National Water Information System (NWIS) database.