

In Reply Refer To:
Mail Stop 412

September 19, 2012

To: GS-W-ALL

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

Subject: Approval of Methods of Analysis-Determination of Pesticides in Sediment Using Gas Chromatography/Mass Spectrometry (USGS Method Numbers O-6144-12 and O-7144-12)

The Office of Water Quality has approved a new analytical method developed by the Organic Chemistry Laboratory at the California Water Science Center for the determination of 119 pesticides in sediment by Gas Chromatography/Mass Spectrometry (Table 1) (USGS Method Numbers O-6144-12 (bed sediment) and O-7144-12 (suspended sediment)).

The pesticides included in this method are applied to many agricultural and urban areas. The method was developed by the U.S. Geological Survey (USGS) in support of the National Water Quality Assessment (NAWQA), the Toxic Substances Hydrology Program (Toxics) and Federal/State Cooperative Projects.

Sediment samples are extracted using an accelerated solvent extraction (ASE) system. The pesticides of interest then are separated from co-extracted matrix interferences with the use of high-performance liquid chromatography and gel-permeation chromatography (HPLC/GPC) along with passing the extracts through either stacked graphitized carbon and alumina solid-phase extraction (SPE) cartridges, or packed Florisil columns. Gas chromatography with mass spectrometry (GC/MS) is used to quantify the extracted sediment samples. Method detection limits (MDLs) ranged from 0.6 to 3.4 micrograms per kilogram ($\mu\text{g/kg}$) dry weight.

This method is described in USGS Techniques and Methods Report 5-C3:
Hladik, M.L., and McWayne, M.M., 2012, Methods of Analysis-Determination of Pesticides in Sediment Using Gas Chromatography/Mass Spectrometry.

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). For questions about the method approval process, please contact Terry Schertz (tschertz@usgs.gov, 703-648-6864).

Table 1. CAS Registry number and USGS parameter codes for each pesticide.

[This report contains Chemical Abstracts Service Registry Numbers (CASRN), which is a Registered Trademark of the American Chemical Society. Chemical Abstracts Service (CAS) recommends the verification of the CASRNs through CAS Client Services. The five-digit parameter codes are used by the U.S. Geological Survey to uniquely identify a specific constituent or property in the National Water Information System (NWIS) database. NA, not available]

Compound	CASRN	Bed-sediment parameter code	Suspended-sediment parameter code
2-Chloro-2,6-Diethylacetanilide	6967-29-9	68876	68875
3,4-Dichloroaniline	95-76-1	66585	63400
3,5-Dichloroaniline	626-43-7	67538	67537
Alachlor	15972-60-8	04006	04021
Allethrin	584-79-2	66588	66587
Atrazine	1912-24-9	39631	04017
Azinphos-methyl	86-50-0	64150	65115
Azoxystrobin	131860-33-8	66591	66590
Benfluralin (Benefin)	1861-40-1	68878	68877
Bifenthrin	82657-04-3	64151	63415
Boscalid	188425-85-6	67552	67551
Butralin	33629-47-9	68880	68879
Butylate	2008-41-5	64152	65116
Captan	133-06-2	68324	68323
Carbaryl	63-25-2	64153	65117
Carbofuran	1563-66-2	64154	65118
Chlorothalonil	1897-45-6	62904	65119
Chlorpyrifos	2921-88-2	81404	65120
Clomazone	81777-89-1	67564	67563
Coumaphos	56-72-4	68882	68881
Cyhalofop-butyl	122008-85-9	68884	68883
Cycloate	1134-23-2	64155	65121
Cyfluthrin	68359-37-5	65109	65122
Cyhalothrin	68085-85-8	68356	68355
Cypermethrin	52315-07-8	64156	65123
Cyproconazole	94361-06-5	66595	66594
Cyprodinil	121522-61-2	67576	67575
DCPA (Dacthal)	1861-32-1	62905	65124
Deltamethrin	52918-63-5	65110	65125
Diazinon	333-41-5	39571	65126
Difenoconazole	119446-68-3	67584	67853
Dimethomorph	110488-70-5	68375	68374
Dithiopyr	97886-45-8	68886	68885
EPTC	759-94-4	64158	65128
Esfenvalerate	66230-04-4	64159	65129
Ethalfluralin	55283-68-6	64160	65130
Etofenprox	80844-07-1	67606	67605
Famoxadone	131807-57-3	67611	67610
Fenarimol	60168-88-9	67615	67614
Fenbuconazole	114369-43-6	67620	67619

Fenhexamide	126833-17-8	67624	67622
Fenpropathrin	39515-41-8	65111	65131
Fenpyroximate	134098-61-6	68888	68887
Fenthion	55-38-9	62046	68889
Fipronil	120068-37-3	66606	66605
Fipronil desulfinyl	NA	66609	66608
Fipronil desulfinyl amide	NA	68891	68890
Fipronil sulfide	120067-83-6	66612	66611
Fipronil sulfone	120068-36-2	66615	66614
Fluazinam	79622-59-6	67638	67637
Fludioxinil	131341-86-1	67642	67641
Flufenacet	142459-58-3	68893	68892
Flumetralin	62924-70-3	68895	68894
Fluoxastrobin	361377-29-9	67647	67646
Flusilazole	85509-19-9	67651	67650
Flutolanil	66332-96-5	68897	68896
Flutriafol	76674-21-0	67655	67654
Hexazinone	51235-04-2	64161	65133
Imazalil	35554-44-0	67664	67663
Indoxacarb	173584-44-6	68899	68898
Iprodione	36734-19-7	66618	63457
Kresoxim-methyl	143390-89-0	67672	67671
Malathion	121-75-5	35931	65135
Metalaxyl	57837-19-1	68439	68438
Metconazole	125116-23-6	66622	66621
Methidathion	950-37-8	62047	65136
Methoprene	40596-69-8	66625	66624
Methyl parathion	298-00-0	39601	65137
Metolachlor	51218-45-2	04005	04020
Molinate	2212-67-1	64163	65138
Myclobutanil	88671-89-0	66634	66633
Napropamide	15299-99-7	64164	65139
Novaluron	116714-46-6	68901	68900
Oxadiazon	19666-30-9	68903	68902
Oxyfluorfen	42874-03-3	64165	63468
<i>p,p'</i> -DDD	72-54-8	39311	63124
<i>p,p'</i> -DDE	72-55-9	39321	63125
<i>p,p'</i> -DDT	50-29-3	39301	63126
Pebulate	1114-71-2	64166	65141
Pendimethalin	40487-42-1	64167	65142
Pentachloroanisole	1825-21-4	49460	66638
Pentachloronitrobenzene	82-68-8	49446	66640
Permethrin	52645-53-1	64168	65143
Phenothrin	26002-80-2	65112	65144
Phosmet	732-11-6	64169	65145
Piperonyl butoxide	51-03-6	64170	65146
Prodiamine	29091-21-2	68905	68904
Prometon	1610-18-0	82402	04011
Prometryn	7287-19-6	78688	04010
Pronamide (Propyzamide)	23950-58-5	67708	67707

Propanil	218.08	66642	63481
Propargite	2312-35-8	68907	68906
Propiconazole	60207-90-1	66645	66644
Propyzamide	23950-58-5	67708	67707
Pyraclostrobin	175013-18-0	66648	66647
Pyridaben	96489-71-3	68909	68908
Pyrimethanil	53112-28-0	67719	67718
Resemethrin	10453-86-8	65113	65147
Simazine	122-34-9	39046	04008
tau-Fluvalinate	69409-94-5	65114	65148
Tebuconazole	107534-96-3	66650	67728
Tebupirimfos oxon	NA	68911	68910
Tebupirimfos	96182-53-5	68913	68912
Tefluthrin	79538-32-2	67733	67732
Tetraconazole	112281-77-3	66656	66655
Tetradifon	116-29-0	68915	68914
Tetramethrin	7696-12-0	66659	66658
Thiazopyr	117718-60-2	68917	68916
Thiobencarb	28249-77-6	64171	65149
Triadimefon	43121-43-3	67743	67742
Triadimenol	55219-65-3	67748	67746
Triallate	2303-17-5	68919	68918
Tribuphos	78-48-8	39050	68920
Trifloxystrobin	141517-21-7	66662	66661
Triflumizole	68694-11-1	67755	67754
Trifluralin	1582-09-8	62902	04019
Triticonazole	131983-72-7	67760	67759
Vinclozolin	50471-44-8	67765	67764
Zoxamide	156052-68-5	67770	67769
