October 17, 2003

To: Water Resources Discipline

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

Subject: Approval of a Water Quality Analytical Method for the Determination of Organochlorine Pesticides and PCBs in Sediment and Suspended Sediment by the National Water Quality Laboratory

The Office of Water Quality (OWQ) has approved a new Water Quality Analytical Method developed by the National Water Quality Laboratory (NWQL) for the determination of organochlorine pesticides and polychlorinated biphenyls (PCBs) in bed material and suspended sediment. This new method uses Soxhlet extraction, gel permeation chromatography, and adsorption chromatography for sample preparation, with analysis by dual-column capillary gas chromatography with electron-capture detection (ECD) of 19 pesticides and 3 PCB Aroclor mixtures. The new U.S. Geological Survey method number is O-5504-03 for bed material and O-7504-03 for suspended sediment. The NWQL schedule numbers are 5504 for bed material and 7504 for suspended sediment. The 19 organochlorine pesticides and 3 PCB Aroclor mixtures are listed in the attached spreadsheet, along with their Parameter and Method Codes for both bed material and suspended sediment.

Interim method reporting limits in bottom material range from about 0.4 to about 3 ug/Kg for the 18 single-constituent organochlorine pesticides, 200 ug/Kg for technical Toxaphene, and between 4 and 5 ug/Kg for the PCB Aroclor mixtures. For suspended sediment, concentrations are reported on a mass per unit volume of water filtered basis (ug/L). Reporting limits are between 0.06 and 0.5 ng/L for the single-constituent pesticides, 30 ng/L for technical Toxaphene, and 0.6 to 0.7 ng/L for the PCB Aroclor mixtures. Most of the compounds were determined with good precision and accuracy, but *p,p'*-Methoxychlor demonstrated high bias which was due to an interferent from the spike solution. Additionally, Toxaphene is difficult to identify and quantify, particularly in weathered samples. Results from these 2 analytes will be routinely qualified with an 'E' data qualifier indicating that precision and accuracy are not as good as other method analytes, and are so indicated in the attached spreadsheet.

Data users are cautioned that both PCBs and Toxaphene are complex mixtures that contain many compounds that create many peaks in the ECD chromatogram. In particular, there are peaks in Aroclor 1254 and/or Aroclor 1260 that may interfere with *p,p'*-DDE when the PCBs are at high concentration. Likewise, high concentrations of *p,p'*-DDE may interfere with PCB determinations. Under these circumstances, results for these analytes may also be given the 'E' data qualifier.

This method approval process follows the technical procedure specified in OWQ Tech Memo 98.05, except that this method is described in a Water Resources Investigations Report instead of an Open File Report. The method performance is described in:

> Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory – Determination of Organochlorine Pesticides and Polychlorinated Biphenyls in Bottom and Suspended Sediment by Gas Chromatography with Electron-Capture Detection, by Mary C. Noriega, Duane S. Wydoski, and William T. Foreman. U.S. Geological Survey Water Resources Investigations Report 03-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 new method will be available through the NWQL web site http://wwwnwql.cr.usgs.gov/USGS. Please click on LIMS Catalog and request Schedule 5504 (bed material) or Schedule 7504 (suspended sediment).

If you have questions about the new analytical method, or would like a copy of the report, when it is available, please contact Mary Noriega (mnoriega@usgs.gov (303) 236-3282) or Duane Wydoski (dwydoski@usgs.gov, (303) 236-3240) at the NWQL.

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

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Compounds in Schedules 5504 or 7504

NWQL Schedule 5504 - Bed Material (ug/Kg) OC Pesticides & PCBs by GC/ECD in Sed Method O-5504-03 Compounds	CAS Number	NWIS Parameter Code	NWQL Method Code	"E" for Estimated Data Flag
Aldrin	309-00-2	39333	B	
cis-(alpha-)Chlordane	5103-71-9	62802	A	
trans-(beta-)Chlordane	5103-74-2	62803	A	
4,4'-Dichlorodiphenyldichloroethane (*p,p'*-DDD)	72-54-8	39363	B	
4,4'-Dichlorodiphenyldichloroethene (*p,p'*-DDE)	72-55-9	39368	B	
4,4'-Dichlorodiphenyltrichloroethane (*p,p'*-DDT)	50-29-3	39373	B	
Dieldrin	60-57-1	39383	B	
Endosulfan I	959-98-8	39389	B	
Endrin	72-20-8	39393	B	
Heptachlor	76-44-8	39413	B	
Heptachlor epoxide (Isomer B)	1024-57-3	39423	B	
Hexachlorobenzene (HCB)	118-74-1	39701	B	
alpha-Hexachlorocyclohexane	319-84-6	39076	B	
beta-Hexachlorocyclohexane	319-85-7	34257	B	
gamma-Hexachlorocyclohexane (Lindane)	58-89-9	39343	B	
p,p'-Methoxychlor	72-43-5	39481	B	**E**
Mirex	2385-85-5	39758	B	
trans-Nonachlor	39765-80-5	62804	A	
PCB Aroclor 1016/1242	12674-11-2/53469-21-9	46343	A	
PCB Aroclor 1254	11097-69-1	39507	A	
PCB Aroclor 1260	11096-82-5	39511	A	
Toxaphene (technical)	8001-35-2	39403	B	**E**
Surrogates				
alpha-Hexachlorocyclohexane-d6	- -	62837	A	
Isodrin	465-73-6	62836	A	
2,2'.3,3',4,4',5,6,6'-Nonachlorobiphenyl (PCB-207)	53742-07-7	62838	A	