November 25, 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 Polynuclear Aromatic Hydrocarbons in 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 polynuclear aromatic hydrocarbons (PAHs) in bed material. This new method uses Soxhlet extraction and gel permeation chromatography for sample preparation, with analysis by capillary column gas chromatography/mass spectrometry. The analyte list contains 28 individual PAH compounds and 25 alkyl PAH homolog groups listed in the attached spreadsheet, along with their Parameter and Method Codes. The new U.S. Geological Survey method number is O-5505-03 and the NWQL schedule number is 5505.

The 28 individual PAHs are all individual compounds, including 18 unsubstituted PAHs and 10 with alkyl (carbon chain) substituents at specified locations of their aromatic rings. These compounds are identified and quantified in the usual manner with retention times and spectral matches, along with standard calibration curves, and generally have good analytical results.

The 25 groups of alkyl PAHs are homologs of 5 groups of isomeric parent PAHs that have alkyl substituents on multiple places of several isomeric PAH ring systems. The groups of alkyl PAHs are identified first by the number of carbon atom substituents attached to the aromatic rings (C1, 1 methyl group to C5, any combination of alkyl groups that add up to 5 carbon atoms) and, second, by examples of the isomeric aromatic ring structures on which these alkyl substituents are placed (naphthalene through benzopyrene/perylene.) Each isomer within a group shares the same molecular weight. They are isomeric homolog groups of similar compounds for which there are no defined standards. They are identified by similar spectral features and quantified by using the closest available compound for calibration. These 25 alkyl PAH homolog groups are reported with an 'E' code because their calculated concentration integrates multiple individual compounds for which there are no standards available. Therefore, the accuracy of these groups cannot be assessed and the uncertainty in the calculated concentration of each alkyl PAH homolog group requires qualification.

Interim method detection limits for the 28 individual PAHs in bottom material range from about 1.3 to about 5.1 ug/Kg. For the 25 alkyl PAH homolog groups, method detection limits cannot be determined because there are no standards. For both the individual PAHs and the alkyl PAH homolog groups, the method reporting limit is set at 10 ug/Kg.

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 Polycyclic Aromatic Hydrocarbons in Sediment by Gas Chromatography/Mass Spectrometry, by Mary C. Olson, Jana L. Iverson, Edward T. Furlong, and Michael P. Schroeder. 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 is available through the NWQL web site http://wwwnwql.cr.usgs.gov/USGS. Please click on LIMS Catalog and request Schedule 5505.

If you have questions about the new analytical method, or would like a copy of the report, when it is available, please contact Mary Olson (mcolson@usgs.gov , (303) 236-3274) or Mike Schroeder (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, (303) 236-1836).

03.10 Compounds in Schedule 5505

NWQL Schedule 5505 - Bed Material (ug/Kg) Polynuclear Aromatic Hydrocarbons (PAHs) by GC/MS in Sed Method O-5505-03 Compounds	CAS Number	NWIS Parameter Code	NWQL Method Code	"E" for Estimated Data Flag
Acenaphthlene	83-32-9	62549	A	
Acenaphthylene	208-96-8	62550	A	
Anthracene	120-12-7	62551	A	
Benz[a]anthracene	56-55-3	62552	A	
Benzo[b]fluoranthene	205-99-2	62554	A	
Benzo[k]fluoranthene	207-8-9	62557	A	
Benzo[g,k,i]perylene	191-24-2	62556	A	
Benzo[a]pyrene	50-32-8	62553	A	
Benzo[e]pyrene	192-97-2	62555	A	
Chrysene	218-01-9	62558	A	
Dibenz[a,h]anthracene	53-70-3	62560	A	
1,2-Dimethylnaphthalene	573-98-8	62538	A	
1,6-Dimethylnaphthalene	575-43-9	62539	A	
2,6-Dimethylnaphthalene	581-42-0	62544	A	
2-Ethylnaphthalene	939-27-5	62545	A	
Fluoranthene	206-44-0	62561	A	
9H-Fluorene	86-73-7	62548	A	
Indeno[1,2,3-cd]pyrene	193-39-5	62562	A	
2-Methylanthracene	613-12-7	62546	A	
1-Methyl-9-H-fluorene	1730-37-6	62540	A	
1-Methylphenanthrene	832-69-9	62541	A	
1-Methylpyrene	2381-21-7	62542	A	
4,5-Methylenephenanthrene	203-64-5	62547	A	
Naphthalene	91-20-3	62563	A	
Perylene	198-55-0	62565	A	
Phenanthrene	85-01-8	62566	A	
Pyrene	129-00-0	62568	A	
2,3,6-Trimethylnaphthalene	829-26-5	62543	A	
C1-alkylated naphthalene	- -	62569	A	E
C2-alkylated naphthalene	- -	62574	A	E
C3-alkylated naphthalene	- -	62579	A	E
C4-alkylated naphthalene	- -	62584	A	E

C5-alkylated naphthalene	- -	62589	A	E
C1-Alkylated phenanthracene/anthracene	- -	62570	A	E
C2-Alkylated phenanthracene/anthracene	- -	62575	A	E
C3-Alkylated phenanthracene/anthracene	- -	62580	A	E
C4-Alkylated phenanthracene/anthracene	- -	62585	A	E
C5-Alkylated phenanthracene/anthracene	- -	62590	A	E
C1-Alkylated fluoranthene/pyrene	- -	62571	A	E
C2-Alkylated fluoranthene/pyrene	- -	62576	A	E
C3-Alkylated fluoranthene/pyrene	- -	62581	A	E
C4-Alkylated fluoranthene/pyrene	- -	62586	A	E
C5-Alkylated fluoranthene/pyrene	- -	62591	A	E
C1-Alkylated benz[a]anthracene/chrysene	- -	62572	A	E
C2-Alkylated benz[a]anthracene/chrysene	- -	62577	A	E
C3-Alkylated benz[a]anthracene/chrysene	- -	62582	A	E
C4-Alkylated benz[a]anthracene/chrysene	- -	62587	A	E
C5-Alkylated benz[a]anthracene/chrysene	- -	62592	A	E
C1-Alkylated benzopyrene/perylene	- -	62573	A	E
C2-Alkylated benzopyrene/perylene	- -	62578	A	E
C3-Alkylated benzopyrene/perylene	- -	62583	A	E
C4-Alkylated benzopyrene/perylene	- -	62588	A	E
C5-Alkylated benzopyrene/perylene	- -	62593	A	E

Surrogates				
2-Fluorobiphenyl	321-60-8	62594	A	
Nitrobenzene-d5	4165-60-0	62595	A	
Terphenyl-d14	1718-51-0	62596	A	