April 10, 2006
(replaces memo issued April 3, 2006)

To: Water Resources Discipline

From: Janice R. Ward, Senior Hydrologist
Office of Water Quality

Subject: Approval of the new USGS National Water Quality Laboratory Analytical Method O-5506-06 for the Determination of Semivolatile Organic Compounds and Polycyclic Aromatic Hydrocarbons in Solids by Gas Chromatography/Mass Spectrometry by the Office of Water Quality

The Office of Water Quality has approved a new water-quality analytical method, O-5506-06 (NWQL Schedules 5506 and 5507), developed for the determination of semivolatile organic compounds and polycyclic aromatic hydrocarbons in solids by gas chromatography/mass spectrometry. The NWQL will issue a separate Rapi-Note when the method is available on a routine basis with additional details and a link to the schedules in the NWQL catalog.

This new USGS method uses a pressurized liquid extraction system and disposable solid-phase extraction cartridges for extract cleanup. Coupling these two steps allows for complex matrices to be extracted, matrix interferences minimized, and a full-scan GC/MS analysis to be performed. It uses less solvent compared to conventional Soxhlet extraction, such as with NWQL schedules 2502 and 2505. Comparisons of PAH data for 28 sediment samples extracted by the conventional Soxhlet method and this method produced similar results, as did comparisons from standard reference material. Because of the inherent complexity of sediment matrices, it is strongly suggested that the sampling analysis plan include matrix spike quality-control samples for appropriate interpretation of the data.

There are two schedules (NWQL schedule 5506 and 5507) associated with this method. Schedule 5506 (see Table 1 below) contains 38 semivolatile organic compounds (SVOCs) and polycyclic aromatic hydrocarbons (PAHs) in solids. The performance of the method was determined using Ottawa sand. Initially, 1,2,4-trichlorobenzene will always be reported as an estimated concentration because of low recovery. Interim method detection limits for the remaining 37 compounds range from about 7 to 28 ug/kg. The initial method reporting levels for all compounds is 50 ug/kg, assuming a 25-g sample size.

The second schedule (NWQL schedule 5507) contains all the compounds in schedule 5506 plus the analysis of 25 alkylated PAH homolog groups associated with the parent compounds in schedule 5506 (see Table 2 below). The PAH homolog groups are determined simply by reprocessing the GC/MS data, without requiring additional sample preparation or analysis. However, because of the lack of authentic reference standard compounds, these results are considered semiquantitative, and are always reported as

estimated concentrations. The method reporting level for all of the alkylated PAH homolog groups is 50 ug/kg.

This method approval process follows the technical procedures specified in OWQ Tech Memo 98.05, except that this method is described in a USGS Techniques and Methods Report instead of an Open-File Report. The reference for this method is:

> Zaugg, S.D., Burkhardt, M.R., Burbank, Terry, Olson, M.C., Iverson, J.L., and Schroeder, M.P., in production, Determination of semivolatile organic compounds and polycyclic aromatic hydrocarbons in solids by gas chromatography/mass spectrometry: U.S. Geological Survey Techniques and Methods, book 5, sec. B, chap. X. (number to be assigned upon Director's approval)

When approved by the Director, the report will be made available through the USGS Publications Warehouse.

If you have questions about the new analytical method, or would like a copy of the report, when it is available, please contact Steve Smith (sgsmith@usgs.gov , 303-236-3274) or Duane Wydoski (dwydoski@usgs.gov , 303-236-3270).

If you have questions about the method approval process, please contact Janice Ward (jward@usgs.gov, 303-236-1871.

Table 1. Semivolatile and polycyclic aromatic hydrocarbon compounds determined using this method**.**

[NWIS, National Water Information System]

Compound name	NWIS parameter code	Compound name	NWIS parameter code
Acenaphthene	64108	Fluoranthene	63208
Acenaphthylene	64109	9H-Fluorene	64107
Anthracene	63180	Hexachlorobenzene	63631
Anthraquinone	63181	Indeno[1,2,3-*cd*]pyrene	64118
Benz[*a*]anthracene	63610	2-Methylanthracene	64105
Benzo[*b*]fluoranthene	64111	1-Methyl-9H-fluorene	64100
Benzo[*k*]fluoranthene	64114	1-Methylphenanthrene	64101
Benzo[*g,h,i*]perylene	64113	1-Methylpyrene	64102
Benzo[*a*]pyrene	63183	4,5-Methylenephenanthrene	64106
Benzo[*e*]pyrene	64112	Naphthalene	63220
Bis(2-ethylhexyl)phthalate	63187	Pentachloroanisol	64119
9H-Carbazole	63194	Pentachloronitrobenzene	63650
Chrysene	64115	Perylene	64120
Dibenz[*a,h*]anthracene	64116	Phenanthrene	63224
Dibenzothiophene	64117	Phenanthridine	64121
Diethyl phthalate	63202	Pyrene	63227
1,2-Dimethylnaphthalene	64097	1,2,4-Trichlorobenzene	64095
1,6-Dimethylnaphthalene	64099	2,3,6-Trimethylnaphthalene	64103
2,6-Dimethylnaphthalene	63167	2-Fluorobiphenyl (method surrogate)	90754
2-Ethylnaphthalene	64104	Nitrobenzene-d_5 (method surrogate)	90755
		Terphenyl-d_{14} (method surrogate)	90756

Table 2. Alkylated polycyclic aromatic hydrocarbon homolog groups determined using this method and reported with estimated concentrations.

[NWIS, National Water Information System]

Compound name	NWIS parameter code
C_1-alkylated naphthalene	64122
C_2-alkylated naphthalene	64123
C_3-alkylated naphthalene	64124
C_4-alkylated naphthalene	64125
C_5-alkylated naphthalene	64126
C_1-alkylated phenanthrene/anthracene	64127
C_2-alkylated phenanthrene/anthracene	64128
C_3-alkylated phenanthrene/anthracene	64129
C_4-alkylated phenanthrene/anthracene	64130
C_5-alkylated phenanthrene/anthracene	64131
C_1-alkylated fluoranthene/pyrene	64132
C_2-alkylated fluoranthene/pyrene	64133
C_3-alkylated fluoranthene/pyrene	64134
C_4-alkylated fluoranthene/pyrene	64135
C_5-alkylated fluoranthene/pyrene	64136
C_1-alkylated benz[*a*]anthracene/chrysene	64137
C_2-alkylated benz[*a*]anthracene/chrysene	64138
C_3-alkylated benz[*a*]anthracene/chrysene	64139
C_4-alkylated benz[*a*]anthracene/chrysene	64140
C_5-alkylated benz[*a*]anthracene/chrysene	64141
C_1-alkylated benzopyrene/perylene	64142
C_2-alkylated benzopyrene/perylene	64143
C_3-alkylated benzopyrene/perylene	64144
C_4-alkylated benzopyrene/perylene	64145
C_5-alkylated benzopyrene/perylene	64146