February 28, 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-5433-06 for the Determination of Wastewater Indicators in Sediment by the Office of Water Quality

The Office of Water Quality (OWQ) has approved a new water-quality analytical method, O-5433-06 (NWQL schedule number 5433) developed for the determination of wastewater indicator compounds in solids and bed material. The NWQL will issue a separate Rapi-Note when the method is available with additional details and a link to the schedule in the NWQL catalog.

This new USGS method uses accelerated solvent extraction (ASE), Soxhlet extraction, and solid-phase extraction (SPE) for sample preparation, with analysis by capillary-column gas chromatography/mass spectrometry. The method was developed in response to increasing concern over the effects of endocrine-disrupting chemicals in wastewater and wastewater-impacted sediment on aquatic organisms. The method also may be used to evaluate the effects of combined sanitary and storm-sewer overflow on the sediment quality of urban streams. The method identifies and quantifies individual compounds using retention times and spectral matches, along with standard calibration curves. Compounds included are: alkylphenol ethoxylate nonionic surfactants and their degradates, food additives, fragrances, antioxidants, flame retardants, plasticizers, industrial solvents, disinfectants, fecal sterols, polycyclic aromatic hydrocarbons, and high-use domestic pesticides. These compounds were chosen because they are wastewater indicators and/or they have endocrine-disrupting potential or toxicity.

The analyte list contains 61 individual compounds (see list below) that complements, but does not exactly match NWQL Schedule 1433, wastewater indicators in water. Initially, 20 compounds will always be reported as estimated concentrations because of recovery, precision, blank contamination, or reference standard issues. Interim method detection limits for the 61 individual compounds range from about 12 to 850 ug/kg. Many of the individual compounds have an interim method reporting level of 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:

> Burkhardt, M.R., Zaugg, S.D., Smith, S.G., and ReVello, R.C., in press, Determination of wastewater compounds in sediment and soil by pressurized solvent extraction, solid-phase extraction, and capillary-column gas chromatography/mass spectrometry: U.S. Geological Survey Techniques and Methods Report, 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 NWQL web site at: http://wwwnwql.cr.usgs.gov/USGS/pubs.html .

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.

NWQL Schedule 5433 Compounds, in micrograms per kilogram - Wastewater Indicators in Sediment

[EDP, endocrine-disrupting potential; S, suspected; K, known; E remark code, estimated concentration reported; F, fungicide; H, herbicide; I, insecticide; GUP, general use pesticide; FR, flame retardant; WW, wastewater; Manuf, manufacturing; %, percent; >, greater than; CP, combustion product; PAH, polycyclic aromatic hydrocarbon; UV, ultraviolet; NA, not applicable; --, no data]

Compound name	EDP	E remark code	Possible compound uses or sources
1,4-Dichlorobenzene[1]	S	--	Moth repellant, fumigant, deodorant
1-Methylnaphthalene	--	--	2-5% of gasoline, diesel fuel, or crude oil
2,6-Dimethylnaphthalene[1]	--	--	Present in diesel/kerosene (trace in gasoline)
2-Methylnaphthalene	--	--	2-5% of gasoline, diesel fuel, or crude oil
3,4-Dichlorophenyl isocyanate	--	E	Degradate of diuron, a noncrop herbicide
3-*beta*-Coprostanol	--	--	Carnivore fecal indicator
3-Methyl-1H-indole (skatol)	--	--	Fragrance, stench in feces and coal tar
3-*tert*-Butyl-4-hydroxyanisole (BHA)	K	--	Antioxidant, general preservative
4-Cumylphenol	K	--	Nonionic detergent metabolite
4-*n*-Octylphenol	K	--	Nonionic detergent metabolite
4-*tert*-Octylphenol	K	--	Nonionic detergent metabolite
Acetophenone	--	E	Fragrance in detergent and tobacco, flavor in beverages
Acetyl-hexamethyl-tetrahydro-naphthalene (AHTN)	--	--	Musk fragrance (widespread use) persistent in ground water
Anthracene[1]	--	--	Wood preservative, component of tar, diesel, or crude oil, CP
Anthraquinone[1]	--	--	Manuf dye/textiles, seed treatment, bird repellant
Atrazine	--	--	Selective triazine herbicide
Benzo[*a*]pyrene[1]	K	--	Regulated PAH, used in cancer research, CP
Benzophenone	S	--	Fixative for perfumes and soaps
beta-Sitosterol	--	--	Plant sterol
beta-Stigmastanol	--	--	Plant sterol
Bisphenol A	K	E	Manuf polycarbonate resins, antioxidant, FR
Bromacil[1]	--	E	H (GUP), >80% noncrop usage on grass/brush
Camphor	--	--	Flavor, odorant, ointments
Carbazole	--	--	I, Manuf dyes, explosives, and lubricants
Chlorpyrifos[1]	K	--	I, domestic pest and termite control (domestic use restricted as of 2001)
Cholesterol	--	--	Often a fecal indicator
Diazinon[1]	K	--	I, > 40% nonagricultural usage, ants, flies

Compound name	EDP	E remark code	Possible compound uses or sources
Diethyl phthalate	--	--	Plasticizer for polymers and resins
Diethylhexyl phthalate	--	--	Plasticizer for polymers and resins, pesticides
d-Limonene	--	E	F, antimicrobial, antiviral, fragrance in aerosols
Fluoranthene[1]	--	--	Component of coal tar and asphalt (only traces in gasoline or diesel fuel), CP
Hexahydrohexamethyl-cyclopentabenzopyran (HHCB)	--	--	Musk fragrance (widespread use) persistent in ground water
Indole	--	--	Pesticide inert ingredient, fragrance in coffee
Isoborneol	--	--	Fragrance in perfumery, in disinfectants
Isophorone[1]	--	E	Solvent for lacquer, plastic, oil, silicon, resin
Isopropylbenzene (cumene)	--	E	Manuf phenol/acetone, fuels and paint thinner
Isoquinoline[1]	--	E	Flavors and fragrances
Menthol	--	--	Cigarettes, cough drops, liniment, mouthwash
Metalaxyl[1]	--	--	H, F (GUP), mildew, blight, pathogens, golf/turf
Methyl salicylate	--	E	Liniment, food, beverage, UV-absorbing lotion
Metolachlor[1]	--	--	H (GUP), indicator of agricultural drainage
N,N-Diethyl-meta-toluamide (Deet)	--	E	I, urban uses, mosquito repellent
Naphthalene[1]	--	--	Fumigant, moth repellent, major component (about 10%) of gasoline
Nonylphenol, diethoxy- (total, NPEO2)	K	E	Nonionic detergent metabolite
Nonylphenol, monoethoxy- (total, NPOE1)	--	E	Nonionic detergent metabolite
Octylphenol, diethoxy- (OPEO2)	K	E	Nonionic detergent metabolite
Octylphenol, monoethoxy- (OPEO1)	K	E	Nonionic detergent metabolite
para-Cresol[1]	S	--	Wood preservative
para-Nonylphenol (total)	K	E	Nonionic detergent metabolite
Pentachlorophenol[1]	S	E	H, F, wood preservative, termite control
Phenanthrene[1]	--	--	Manuf explosives, component of tar, diesel fuel, or crude oil, CP
Phenol[1]	--	E	Disinfectant, manuf several products, leachate
Prometon[1]	--	--	H (noncrop only), applied prior to blacktop

Compound name	EDP	E remark code	Possible compound uses or sources
Pyrene[1]	--	--	Component of coal tar and asphalt (only traces in gasoline or diesel fuel), CP
Tetrabromodiphenyl ether	--	--	Fire retardant
Tri(2-butoxyethyl) phosphate	--	--	Flame retardant
Tri(2-chloroethyl) phosphate	S	E	Plasticizer, flame retardant
Tri(dichloroisopropyl) phosphate	S	E	Flame retardant
Tributyl phosphate	--	--	Antifoaming agent, flame retardant
Triclosan	S	--	Disinfectant, antimicrobial (concern for acquired microbial resistance)
Triphenyl phosphate	--	E	Plasticizer, resin, wax, finish, roofing paper, FR

[1]Compound determined by at least one other method at the National Water Quality Laboratory.