APPENDIX A

SUMMARY OF LABORATORY PROCEDURES

APPENDIX A.1

Summary of Fish Tissue Extraction and Analysis Procedures for Pesticides and PCBs

A fish tissue homogenate sample is extracted with acetonitrile in an 8oz. glass Qorpak wide mouth bottle. The extracted sample is filtered and the filtrate is partitioned with petroleum ether. The petroleum ether extract is eluted through a Florisil column to remove lipids and fractionate the sample extract into four fractions to simplify the chromatography. The Florisil columns are eluted with petroleum ether (Fraction 1), 6% ethyl ether (Fraction 2), 15% ethyl ether (Fraction 3), and 50% ethyl ether (Fraction 4). The fractions are concentrated to an appropriate volume using a Zymark Turbovap II concentrator prior to analysis by gas chromatography. A mixture of synthetic organic standards is eluted through the florisil column to determine the recovery and separation characteristics of the column. Method quantitation limits for target compounds are reported on the following page.

Distribution of Synthetic Organic Compounds among Four Fractions of Standard Florisil® Column

(0%) Fraction 1	(6%) Fraction 2	(15%) Fraction 3	(50%) Fraction 4
HCH, alpha*	HCH, alpha*	Dacthal	Endosulfan II
aldrin	HCH, beta	Diazinon	Endosulfan Sulfate
Chlordene, alpha	HCH, gamma	Dichlorobenzophenone, p,p'	
Chlordene, gamma	HCH, delta	dieldrin	
DDE, o,p'	chlorbenside	endosulfan I	
DDE p,p'	cis-chlordane	endrin	
DDMU, p,p'*	trans-chlordane	malathion	
DDT, o,p'	chlorpyrifos	oxadiazon	
DDT, p,p'*	DDD, o,p'*	Parathion, ethyl	
heptachlor	DDD, p,p'	Parathion, methyl	
hexachorobenzene	DDMU p,p'*	Tetradifon (tedion)	
trans-nonachlor	DDT, p,p'*		
PCB 1248	dicofol (kelthane)		
PCB 1254	ethion		
PCB 1260	heptachlor epoxide		
	methoxychlor		
	cis-nonachlor		
	oxychlordane		
	toxaphene		

APPENDIX A.1 (continued)

Quantitation Limits for Synthetic Organic Compounds and Trace Elements Analyzed in Flesh

Compound	Quantitation Limit (µg/kg, ppb wet weight)
aldrin	5
chlorbenside	50
cis-chlordane	5
trans-chlordane	5
chlordene, alpha	5
chlordene, gamma	5
chlorpyrifos	10
dacthal	5
DDD, o,'p	10
DDD, p,p'	10
DDE, o,p'	10
DDE, p,p'	5
DDMU,p,p'	15
DDT, o,p'	10
DDT, p,p'	10
diazinon	50
dichlorobenzophenone-p,p'	30
dicofol (Kelthane)	100
dieldrin	5
endosulfan I	5
endosulfan II	70
endosulfan sulfate	85
endrin	15
ethion	20
HCH, alpha	2
HCH, beta	10
HCH, gamma	2
HCH, delta	5
heptachlor	5
heptachlor epoxide	5
HCB	2
methoxychlor	15
cis-nonachlor	5
trans-nonachlor	5
oxadiazon	5
oxychlordane	5
parathion, ethyl	10
parathion, methyl	10
PCB 1248	50
PCB 1254	50
PCB 1260	50
tetradifon (Tedion)	10
toxaphene	100

APPENDIX A.2

Trace Element Digestion Techniques and Quantitation Limits in Fish Tissue

Element	Digestion Techniques	Instrumental Analysis	Quantitation Limits (µg/g, ppm wet weight)
Arsenic	Dry Ash w/$Mg(NO_3)_2 6H_2 0$	$NaBH_4$ Reduction A.A.	0.05
Mercury	HNO_3 reflux	Cold Vapor A.A.	0.02
Copper	HNO_3 reflux	Flame A.A. or Graphite Furnace	0.02
Zinc	HNO_3 reflux	Flame A.A.	0.05
Cadmium	HNO_3 reflux	Graphite Furnace (Ammonium phosphate/magnesium nitrate)	0.01
Chromium	HNO_3 reflux	Graphite Furnace	0.02
Lead	HNO_3 reflux	Graphite Furnace (Ammonium phosphate/magnesium nitrate)	0.1
Nickel	HNO_3 reflux	Graphite Furnace	0.1
Selenium	Dry Ash w/$Mg(NO_3)_2 6H_2O$	$NaBH_4$ Reduction A.A.	0.05
Silver	HNO_3 reflux	Graphite Furnace	0.02

APPENDIX A.3

SCHEDULES AND METHOD REPORTING LIMITS (MRLs) FOR U.S. LABORATORIES

WATER

SCHEDULE 2703
Trace elements in water filtered through a 0.45 µm-filter

COMPOUND NAME, ANALYTICAL TECHNIQUE	MRL (µg/L)
Arsenic, DIS	1
Aluminum, DIS, ICP/MS	1
Antimony. DIS, ICP/MS	1
Barium, DIS, ICP/MS	1
Beryllium, DIS, ICP/MS	1
Cadmium, DIS, ICP/MS	1
Chromium, DIS, ICP/MS	1
Cobalt, DIS, ICP/MS	1
Cooper, DIS, ICP/MS	1
Lead, DIS, ICP/MS	1
Manganese, DIS, ICP/MS	1
Molybdenum, DIS, ICP/MS	1
Nickel, DIS, ICP/MS	1
Selenium, DIS	1
Silver, DIS, ICP/MS	1
Uranium, DIS, ICP/MS	1
Zinc, DIS, ICP/MS	1

SCHEDULE 2750
Major inorganics in water

COMPOUND NAME, ANALYTICAL TECHNIQUE	MRL
Chloride, DIS	0.1 mg/L
ROE DIS @ 180°C	1 mg/L
Potassium, DIS	0.1 mg/L
pH laboratory	
Specific conductance, lab	1 µS/cm
Alkalinity, as $CaCO_3$, lab	1 mg/L
Iron, DIS	3 µg/L
Manganese, DIS	1 µg/L
Calcium, DIS	.02 mg/L
Magnesium, DIS	.01
Silica, DIS	.01
Sodium, DIS	.20

Bromide, DIS	.01
Sulfate, DIS	.10
Fluoride, DIS	.10

SCHEDULE 2702
Nutrients in water

COMPOUND NAME, ANALYTICAL TECHNIQUE	MRL (mg/L)
Nitrogen, nitrite as N,DIS	0.01
Phosphorus, orthophosphate, as P, DIS	0.01
Nitrogen, nitrate +nitrite, as N, DIS	0.05
Nitrogen, ammonia, as N, DIS	0.01
Phosphorus, as P, DIS	0.01
Nitrogen, ammonia +organic, as N. DIS	0.20
Nitrogen, ammonia +organic, as N total	0.20
Phosphorus, as P total	0.01

SCHEDULE 2075
Gross Organics in water

Carbon, organic, dissolved (DOC)	0.10 µg/L
Carbon, organic, suspended (SOC)	0.10

SCHEDULE 2090
Volatile organic compounds at 0.20µg/L by purge and trap GC/MS plus library search of nontarget constituents

COMPOUND NAME	MRL (µg/L)
Benzene	0.2
Benzene,1,2,3 trichloro-	0.2
Benzene,1,2,4 trichloro-	0.2
Benzene, 1,2,4 trimethyl-	0.2
Benzene,1,2 Dichloro-	0.2
Benzene,1,3,5 trimethyl-	0.2
Benzene,1,3 Dichloro	0.2
Benzene,1,4 Dichloro-	0.2
Benzene, 1-chloro-2-methyl-	0.2
Benzene, 1- chloro-4 methyl-	0.2
Benzene, Isopropyl-	0.2
Benzene, Bromo-	0.2
Benzene, Chloro-	0.2
Benzene, dimethyl-(Xylene)	0.2
Benzene, Ethyl-	0.2
Benzene, 1-Methyl-4 isopropyl-	0.2
Benzene, Methyl (toluene)	0.2
Benzene, n-butyl-	0.2

Benzene, n-propyl-	0.2
Benzene, sec-butyl-	0.2
Benzene, ter-butyl-	0.2
Ethane, 1,1,1,2-tetrachloro	0.2
Ethane 1,1,1,1-trichloro-	0.2
Ethane 1,1,2,2 Tetrachloro-	0.2
Ethane, 1,1,2- Trichloro	0.2
Ethane, 1,1-Dichloro	0.2
Ethane, 1,2-Dibromo (EDB)	0.2
Ethane 1,2, Dichloro	0.2
Ethane, Chloro	0.2
Ethane, Trichlorotrifluoro-	0.2
Ethylene, 1,1-Dichloro	0.2
Ethylene, Chloro (vinyl Chloride)	0.2
Ethylene, cis-1,2-Dichloro	0.2
Ethylene, tetrachloro-	0.2
Ethylene, trans-1,2 dichloro	0.2
Ethylene, trichloro	0.2
Hexachlorobutadiene	0.2
Methane, bromo	0.2
Methane, bromochloro-	0.2
Methane, chloro	0.2
Methane, dibromo	0.2
Methane, Dibromochloro	0.2
Methane, Dichloro	0.2
Methane dichlorobromo	0.2
Methane, dichlrodifluoro-	0.2
Methane, tetrachloro-	0.2
Methane, tribromo- (Bromoform)	0.2
Methane, Trichloro- (Chloroform)	0.2
Methane, trichlorofluoro-	0.2
Propene,2 methoxy-2methyl	0.2
Naphthalene	0.2
Propane,1,2,3 Trichloro	0.2
Propane 1,2, Dibromo-3-chloro (DBCP)	0.2
Propane, 1,2 Dichloro	0.2
Propane, 1,3 Dichloro-	0.2
Propane 2,2, Dichloro-	0.2
Propene 1,1, Dichloro-	0.2
Propene, cis-1,2-Dichloro-	0.2
Propene, trans-1,3-dichloro-	0.2
Styrene	0.2

SCHEDULE 1385

Extractable Priority Pollutants. Organic compounds, acid and base/neutral, total recoverable. GC/MS

COMPOUND NAME	MRL (µg/L)
4-Chloro-3methylphenol	30
2-Chlorophenol	5
2,4 Dichlorophenol	5
2,4,6 Trichlorophenol	20
2,4-Dimethylphenol	5
4,6-Dinitro-2-methylphenol	30
2,4 Dinitrophenol	20
2-Nitrophenol	5
4-Nitrophenol	30
Pentachlorophenol	30
Phenol	5
Acenaphthene	5
Acenphtylene	5
Anthracene	5
Benzidine	40
Benzo[a]anthracene	10
Benzo[b]fluoranthene	10
Benzo[k]fluoranthene	10
Benzo[a]pyrene	10
Benzo[g,h,i]perylene	10
Butyl benzyl phtalate	5
Bis(2-chloroethoxy) methane	5
Bis (2-chloroethyl) ether	5
Bis (2-chloropropyl) ether	5
4-Bromophenylphenyether	5
2-Chloronaphthalene	5
4-Chloropehylphenyether	5
Chrysene	10
1,2,5,6-Dibenz[a,h]anthracene	10
Di-n-butyl phtalate	5
1,2, Dichlorobenzene	5
1,3 Dichlorobenzene	5
1,4 Dichlorobenzene	5
3,3,Dichlorobenzidine	20
Diethyl phtalate	5
Dimethyl phtalate	5
2,4, Dinitrotoluene	5
2,6-Dinitrotoluene	5
Di-n-octyl phtalate	10
Bis (2-ethylhexyl) phtalate	5
Fluorene	5

Fluoranthene	5
Hexachlorobenzene	5
Hexachlorobutadiene	5
Hexachlorocyclopentadiene	5
Hexachloroethane	5
Indeno(1,2,3-cd) pyrene	10
Isophorone	5
Naphthalene	5
Nitrobenzene	5
n- Nitrosodimethylyamine	5
n-Nitrosodiphenylamine	5
n- Nitrosodi-n-propylamine	5
Phenanthrene	5
Pyrene	5
1,2,4 Trichlorobenzene	5
1,2-Diphenyhydrazine	5

SCHEDULE 2001

Pesticides in filtered water extract on C-18 SPE cartridge and analyzed by GC/MS.

COMPOUND NAME	MRL (µg/L)
Alachlor	0.009
Atrazine-desethyl-	0.007
Atrazine	0.017
Azinphos-Methyl-	0.038
Benfluralin	0.013
Butylate	0.008
Carbaryl	0.046
Carbofuran	0.013
chlorpyrifos	0.005
Cyanazine	0.013
DCPA	0.004
DDE-p.p'	0.010
Diazinon	0.008
Dieldrin	0.008
Diethylaniline	0.006
Dimethoate	0.024
Disulfoton	0.028
EPTC	0.005
Ethafluralin	0.013
Ethoprop	0.012
Fonofos	0.008
HCH-alfa	0.007
HCH-gama	0.011
Linuron	0.039

Malathion	0.010
Metolachlor	0.009
Metribuzin	0.012
Molinate	0.007
Napropamide	0.010
Parathion-ethyl-	0.022
Parathion, methyl-	0.035
Pebulate	0.009
Pendimethalin	0.018
Permethrin-cis	0.019
Phorate	0.011
Pronamide	0.009
Prometon	0.008
Propachlor	0.015
Propanil	0.016
Propargite	0.006
Simazine	0.008
Thiobenacarb	0.008
Tebuthiuron	0.015
Terbacil	0.030
Terbufos	0.012
Triallate	0.008
Trifluralin	0.012

SCHEDULE 2050

Pesticides in filtered water extract on Carbopack B SPE and analyzed by HPLC

COMPOUND NAME	MRL (µg/L)
2,4,5-T	0.05
2,4 D	0.05
2,4-DB	0.05
Acifluorfen	0.05
Aldicarb	0.05
Aldicarb sulfone	0.05
Aldicarb sulfoxide	0.05
Bentazon	0.05
Bromacil	0.05
Bromoxynil	0.05
Carbaryl	0.05
Carbofuran	0.05
Carbofuran 3-hydroxy	0.05
Chloramben	0.05
Chlorothalonil	0.05
Clopyralid	0.05
Dacthal, Mono acid	0.05

Dicamba	0.05
Dichllobenil	0.05
Dichlorprop	0.05
Dinoseb	0.05
Diuron	0.05
DNOC	0.05
Esfenvalerate	0.05
Fenuron	0.05
Fluometuron	0.05
Linuron	0.05
MCPA	0.05
MCPB	0.05
Methiocarb	0.05
Methomyl	0.05
1-Naphthol	0.05
Neburon	0.05
Norflurazon	0.05
Oryzalin	0.05
Oxamyl	0.05
Picloram	0.05
Propham	0.05
Propoxur	0.05
Silvex	0.05
Triclopyr	0.05

SEDIMENTS

SCHEDULE 2400
Trace elements in bed sediments

COMPOUND NAME, ANALYTICAL TECHNIQUE	LLD (dry wt)
Aluminum, ICP	0.05%
Calcium, ICP	0.05
Iron, ICP	0.05
Potassium, ICP	0.05
Magnesium, ICP	0.005
Sodium, ICP	0.005
Phosphorous, ICP	0.005
Titanium, ICP	0.005
Gold, ICP	8 µg/g
Barium, ICP	1
Beryllium, ICP	1
Bismuth, ICP	10
Cerium, ICP	4
Cobalt, ICP	1

Chromium, ICP	1
Copper, ICP	1
Europium, ICP	2
Gallium, ICP	4
Holmium, ICP	4 µg/g
Lanthanum, ICP	2
Lithium, ICP	2
Manganese, ICP	4
Molybdenum, ICP	2
Niobium, ICP	4
Neodymium, ICP	4
Nickel, ICP	2
Lead, ICP	4
Scandium, ICP	2
Tin, ICP	10
Strontium, ICP	2
Tantalum, ICP	40
Thorium, ICP	4
Vanadium, ICP	2
Yttrium, ICP	2
Ytterbium, ICP	1
Zinc, ICP	4
Silver, GFAA	0 .1
Cadmium, GFAA	0 .1
Mercury, CVAA	0 .02
Arsenic, HA	0 .1
Antimony, HA	0.1
Selenium, HA	0.1
Uranium, DNAA	0.05
Thorium, DNAA	1
Sulfur, IR	0.05
Carbon, total	0.01
Carbon, organic	0.01

SCHEDULE 1386

Organic compounds acid and base/neutral recoverable from bottom material

	MRL (µg/kg)
4-Chloro-3methylphenol	600
2-Chlorophenol	200
2,4Dichlorophenol	200
2,4 Dimethylphenol	200
2-Methyl-4,6 dinitrophenol	600
2,4 Dinitrophenol	600
2-Nitrophenol	200

4-Nitrophenol	600
Pentachlorophenol	600
Phenol	200
2,4,6 Trichlophenol	600
Acenaphtalene	200
Acenaphthylene	200
Anthracene	200
Benzo[a]anthracene	400
Benzo[b]fluoranthene	400
Benzo[k]fluoranthene	400
Benzo[a]pyrene	400
Benzo[g,h,i]perylene	400
Butyl benzyl phtalate	200
Bis(2-chloroethoxy) methane	200
Bis (2-chloroethyl) ether	200
Bis (2-chloropropyl) ether	200
4-Bromophenylphenyether	200
2-Chloronaphthalene	200
4-Chloropehylphenyether	200
Chrysene	400
1,2,5,6-Dibenz[a,h]anthracene	400
Di-n-butyl phtalate	200
1,2, Dichlorobenzene	200
1,3 Dichlorobenzene	200
1,4 Dichlorobenzene	200
Diethyl phthalate	200
Dimethyl phthalate	200
2,4, Dinitrotoluene	200
2,6-Dinitrotoluene	200
Di-n-octyl phtalate	200
Bis (2-ethylhexyl) phtalate	200
Fluorene	200
Fluoranthene	200
Hexachlorobenzene	200
Hexachlorobutadiene	200
Hexachlorocyclopentadiene	200
Hexachloroethane	200
Indeno(1,2,3-cd) pyrene	400
Isophorone	200
Naphthalene	200
Nitrobenzene	200
n- Nitrosodimethylyamine	200
n-Nitrosodiphenylamine	200
n- Nitrosodi-n-propylamine	200
Phenanthrene	200

Pyrene	200
1,2,4 Trichlorobenzene	200

SCHEDULE 2501
Chlorinated organic compounds

	MRL (µg/kg)
Aldrin	1
Chlordane, cis	1
Chlordane, trans	1
Chloroneb	5
DCPA	5
DDD,o.p'	1
DDD, p,p'	1
DDE, o,p'	1
DDT,o,p'	2
DDT,p,p'	2
Dieldrin	1
Endosulfan I	1
Endrin	2
HCB, alpha,	1
HCH, beta-	1
HCH, gamma	1
Heptachlor	1
Heptachlor epoxide	1
Hexachlorobenzene	1
Isodrin	1
Methoxychlor, o,p'	5
Methoxychlor, pp	5
Mirex	1
Nonachlor, cis	1
Nonachlor, trans	1
Oxychlordane	1
PCB's total	50
Pentachloroanisole	1
Permetrin, cis-	5
Permetrin, trans	5
Toxaphene	200

SCHEDULE 2502
Base-neutral Acid (BNA) semivolatile organic compounds in bed sediments

	MRL (µg/kg)
Acenaphthylene	50
Acenaphthene	50
Acridine	50
Amine, n-Nitroso-Di-n-Propyl	50

Amine, n-Nitroso-diphenyl-		50
Anthracene		50
Anthracene, 3- Methyl	50	
Benzo[a]anthracene		50
Dibenzo[a,h]anthracene		50
Anthraquinone	50	
Benzene,1,2,4 trichloro-		50
Benzene, 1,2 dichloro-		50
Benzene, 1,3 dichloro		50
Benzene, 1,4 dichloro	50	
Azobenzene		50
Nitrobenzene		50
Pentachlorobenzene		50
Pentachloronitrobenzene		50
Hexachlorocyclobutadiene		50
9H-Carbazole		50
Chrysene		50
p-cresol		50
Dibenzothiophene		50
Hexachloroethane		M-del
4-Bromophenylphenyl-ether		50
4- Chlorophenylphenyl-ether		50
bis (2Chloroethyl)-ether		50
bis (2Chloroisopropyl)ether		50
Fluoranthene		50
Benzo[b]fluoranthene		50
Benzo[k]fluoranthene		50
9H-Fluorene,1 Methyl-		50
9H Fluorene		50
Hexchlorocyclopentadiene		M-Del
Isophorone		50
Methane, bis (2-chloroethoxy)-		50
Naphthalene		50
Naphthalene, 2 Chloro		50
Naphthalene, 1,2 dimethyl		50
Naphthalene, 1,6 dimethyl		50
Naphthalene, 2,6 dimethyl		50
Naphthalene, 2 ethyl		50
Naphthalene,2,3,6 trimethyl		50
Pentachloroanisole		50
Benzo[g,h,I] perylene		50
Phenanthrene		50
Phenanthrene, 1 methyl		50
Phenanthrene,4,5 Methylene		50
Phenantridine		50

Phenol	50
2,4 dinitrophenol	M-Del
2 methyl-4,6 dinitrophenol	M-Del
2,3,5,6 tetramethylphenol	M-Del
2-Nitrophenol	M-Del
4-Nitrophenol	M-Del
2,4,6 trichlorophenol	M-Del
3,5 Dimethyl phenol	50
Phenol, 4-Chloro-3Mthyl	M-Del
2,4 Dichlorophenol	M-Del
2,4,6 trimethyl phenol	50
C8 Alkyl- phenol	50
Pentachlorphenol	M-Del
Phthalate, bis (2Ethylhexyl)	50
Phthalate, buylbenzyl	50
Phtahalate, Di-n-butyl	50
Phthalate, Di-n-octyl	50
Phthalate, Diethyl	50
Phthalate, Dimethyl	50
Pyrene	50
Pyrene, 1-Methyl	50
Benzo[a]pyrene	50
Indeno [1,2,3-cd] pyrene	50
2,2, Biquinoline	50
Benzo[c]quinoline	50
Isoquinoline	50
2,4 Dinitrotoluene	50
2,6 Dinitrotoluene	50