SCOPE OF WORK FOR CHEMICAL ANALYSES IN SUPPORT OF GRAYS HARBOR SALMON SURVIVAL STUDIES IN FY 88 AND 89

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EPA and Ecology are contributing laboratory analyses to support studies being conducted by Fisheries, the U.S. Fish and Wildlife Service (USFWS), the National Marine Fisheries Service (NMFS), and the University of Washington (UW) on survival of Chehalis River coho salmon. This document outlines the scope of work for toxic chemical and conventional water quality analyses to be done in conjunction with the following study elements:

FY 88
- Smolt bioassays (study element V)
- Barging and holding studies (study element IV)
- Field collections of smolts (study elements I, II, VI)

FY 89
- Smolt bioassays (study element V)
- Barging and holding studies (study element IV)

The objectives of the EPA/Ecology analytical program will be to characterize the industrial effluents, municipal effluents, and Chehalis River water being evaluated through bioassay and obtain water and sediment quality data on the Chehalis River, inner Grays Harbor, and Humptulips River during the period of smolt migration. Emphasis will be placed on identification of potential toxicants. These data will be furnished to Fisheries and other project investigators and documented by EPA and Ecology in the final project report.

SAMPLING AND ANALYSIS DESIGN

Smolt bioassays (FY 88) -- USFWS plans to conduct coho smolt bioassays in May using final effluents from the Weyerhaeuser and ITT Rayonier pulp mills, and the Aberdeen and Hoquiam sewage treatment plants. Chehalis River water and a reference toxicant (50 ug/L copper) will also be assayed. Exposure to pulp mill effluents will be at concentrations of 30 percent, 10 percent, and 1 percent; sewage treatment plant effluents will be tested as a single mixture of 0.5 percent. Each exposure will be replicated. Dechlorinated Aberdeen municipal water (Wishkah reservoir) will be used for dilution water.

The bioassays will be flow-through using 3,000- and 6,000-gallon tanks for storage of effluents and river water. The tanks will be refilled with fresh sample every one to two days. The expected exposure period is between two and seven days, depending on results of smolt migration pattern studies being done by Fisheries, NMFS, and UW (study element III). At the end of the bioassays, fish will be subjected to a series of tests and transported to the USFWS Marrowstone Island field station for nine-month seawater growth and survival evaluations.

Table 1 summarizes the chemical analyses to be done for these bioassays in FY 88. EPA priority pollutants/hazardous substances are analyzed for each bioassay. Toxic constituents reported to be present in pulp mill effluents--dioxins, furans, fatty acids, resin acids, guaiacols and catechols--are analyzed in pulp mill effluents only. Herbicides,

organophosphorus and carbamate pesticides are analyzed in Chehalis River water. Appendix I lists individual analytes.

The sampling frequency for the above chemicals is twice over the course of each bioassay (once for dioxin/furan). All samples will be grabs[1] collected directly from the storage tanks mid-way through the one- to two-day period over which each batch of effluents/river water is used. Within-batch variability will be assessed by replicating one sample collection each for the Weyerhaeuser and combined sewage treatment plants effluents. Between-batch variability will be evaluated through more frequent analyses for sulfite/bisulfite, ammonia, total chlorine residual, total phenolics, total organic halogen, and ancillary water quality variables (conductivity, hardness, total organic carbon, and total suspended solids). USFWS will monitor temperature, pH, and D.O. frequently. Sampling and analysis protocols are provided in the quality assurance plan (Appendix II).

In addition to the above analyses, it is anticipated that NOAA Northwest and Alaska Fisheries Center will analyze fish bile for resin acid and phenolics metabolites. A yet-to-be-determined number of smolt tissue samples will also be stored frozen at the EPA/Ecology Manchester laboratory for possible future toxics analyses.

Smolt bioassays (FY 89) -- USFWS tentatively plans to do a second round of bioassays in FY 89 using effluent mixtures. The level of effort is expected to be similar to FY 88. The chemical analyses done in FY 88 should allow the number of effluents and chemicals of concern to be substantially narrowed in FY 89. For planning purposes, analytical costs for the FY 89 bioassays was estimated to be half that in FY 88.

Barging and holding studies (FY 88) -- Fisheries will barge hatchery smolts through the lower Chehalis River and inner Grays Harbor, and through the Humptulips estuary. There will be separate trips for the north and south channels of the Chehalis. One trip is tentatively planned for each migration route, probably in late May. The barge path will mimic the patterns found in the smolt migration studies. Smolts will be sampled periodically and subjected to a series of tests. The remaining fish will be transferred to NMFS pens at Manchester for nine-month evaluations.

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[1] If a suitable continuous centrifuge or large-volume filter can be obtained, a portion of the Table 1 analyses will be done on particulate matter samples of the effluents. This will afford lower detection limits.

The water quality analyses for the barging portion of these studies are summarized in Table 2. Automatic samplers[2] mounted on the barge or tow vessel will be used to collect composite samples for analysis of priority pollutants/hazardous substances and other selected toxicants. The nature of the compounds included in the volatiles analysis requires these samples be grabs. Ancillary variables will be conductivity, salinity, hardness, total organic carbon, total suspended solids and turbidity. Fisheries will measure temperature, pH, D.O., and salinity hourly. Total phenolics and total organic halogen (Table 1) will not be analyzed for the barging study because method detection limits are not sufficiently low for the concentration levels expected.

The number of composite and grab samples taken will depend on the duration of each barge trip through the Chehalis River/inner Grays Harbor. Table 2 assumes each trip lasts about three days. The first composite will begin at the point of origin (Montesano); two composite samples will be taken during the period the barge is in the lower river/inner harbor. If the trip last longer than three days, either the compositing period will be increased or the three composites staggered over the course of the trip. Sampling for the barge trip through the Humptulips estuary will be limited to one composite sample in the Campbell Slough/North Bay area.

Bile analysis is also anticipated for the barging study. Smolt tissue samples for possible additional analyses will be collected at various points along each barge trip and frozen.

Ecology has scheduled Class II/biomonitoring inspections at the Weyerhaeuser and ITT mills to coincide with the FY 88 barging study. Table 3 summarizes the toxics analyses to be done on the final effluents and receiving environment. A suite of acute toxicity bioassays will be done for both inspections. The types of tests have not been selected.

Sediment samples are routinely collected at industrial outfalls during Ecology Class II inspections. The number of samples to be collected during the Grays Harbor Class II's will be expanded to give greater coverage of the inner harbor and will also include a sample in the Humptulips estuary. Tentative station locations are shown in Figure 1. Physical/chemical analyses will be as shown in Table 3. Acute toxicity bioassays also will be done on sediments[3].

Barging and holding studies (FY 89) -- Two trips per route are planned for FY 89. For planning purposes analytical support was estimated to

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[2]Continuous centrifuge or large-volume filter is a possible alternative sampling method.

[3]EPA tentatively plans to do preliminary analyses on sediment samples collected at both pulp mill outfalls in April 1988.

be the same as for the FY 88 barging, again assuming the list of chemicals of potential concern will have been narrowed.

Field collections of smolts (FY 88) -- Fisheries will collect smolts from both drainages between March and May to determine if there are differences in terms of smoltification and health. The occurrence of toxicants in the Chehalis River, inner Grays Harbor, and Humptulips estuary will be assessed through the above-mentioned sampling program for the bioassays and barging studies. If barging is not done in FY 88, those samples will be taken in conjunction with the smolt collections, probably during the peak of the outmigration. A yet-to-be-determined number of smolt tissue samples will be saved frozen from among Fisheries' field collections.

Other pertinent data-gathering efforts -- Water quality of the Chehalis and Humptulips basins is monitored monthly as part of Ecology's ongoing ambient monitoring program; Grays Harbor is monitored once a month between April and November. Table 4 describes station locations and variables measured. Weyerhaeuser and ITT monitor the estuary stations weekly throughout the year for the same variables.

EPA collected 11 coho smolt samples in the Chehalis drainage in 1987. These are currently being analyzed at the Ecology/EPA Manchester laboratory for semivolatiles, PCBs, chlorinated pesticides, and metals.

EPA also collected flounder, crab, clams, and sediment below both the Weyerhaeuser and ITT mills in June 1987 as part of the National Bioaccumulation Study. These samples are currently being analyzed at the EPA Duluth laboratory for mercury and a wide range of organic chemicals (Appendix III).

TIME TABLE

Table 5. Time table for FY 88 analytical support.

	--FY 88 --		- - - - - -FY 89- - - - - -		
Activity	May	June	July	August	September
Bioassays	X			0	0
Barging/smolt collections	X			0	0
Class II inspections	X			0	0

X = samples collected
0 = analyses completed
0 = data reported to other investigators

COST ESTIMATE

Table 6 shows estimated analytical costs. Cost of organics and metals analyses includes 20 percent for quality assurance samples. It should be recognized that findings from the first year may push the FY 89 studies in a new direction requiring a different type and level of analytical support.

Table 6. Cost estimate for analytical support.

Activity	FY 88	FY 89
Bioassays*	$28,813	$14,407
Class II inspections	30,000	--
Barging/smolt collections	18,276	18,276
Unanticipated analytical needs (tissue, sediment, other)	--	10,000
Total	$77,089	$42,683

*Assumes 7-day bioassay

Table 1. Sampling frequency and analyses for smolt bioassays in FY 88.

Analyses	Sampling Frequency [a] Weyco Effluent	ITT Effluent	Aberdeen and Hoquiam STP Effluents	Chehalis River Water	Control Water	Total No. of Samples [b]
EPA Priority Pollutants/Hazardous Substances						
volatiles	2	2	2	2	1	9
semi-volatiles	2	2	2	2	1	9
metals	2	2	2	2	1	9
PCB's/OC pesticides	2	2	2	2	1	9
cyanide	2	2	2	2	1	9
dioxins/furans	1	1	--	--	--	2
Other Toxics Analyses						
fatty acids, resin acids, guaiacols, catechols	2[c]	2[c]	--	--	[c]	4
herbicides, OP pesticides, carbamates	--	--	--	2	1	3
sulfite/bisulfite	2-7[d]	2-7	--	--	1	4-14
ammonia	2-7	2-7	2-7	2-7	1	9-29
total chlorine residual	2-7	2-7	2-7	2-7	1	9-29
total phenolics	2-7	2-7	2-7	2-7	1	9-29
total organic halogen	2-7	2-7	2-7	2-7	1	9-29
Ancillary Variables[e]						
specific conductivity	2-7	2-7	2-7	2-7	2-7	9-29
turbidity	2-7	2-7	2-7	2-7	2-7	9-29
total hardness	2-7	2-7	2-7	2-7	2-7	9-29
total organic carbon	2-7	2-7	2-7	2-7	2-7	9-29
total suspended solids	2-7	2-7	2-7	2-7	2-7	9-29

[a] grab samples

[b] QA samples (blanks, replicates, duplicates, matrix spikes) not included

[c] fish bile also analyzed for metabolites

[d] 2-7 exposure, 1-day renewal period

[e] temperature, pH, and D.O. monitored 4-12 times/day by USFWS

Table 2. Sampling frequency and analyses for barging studies in FY 88.

Analyses	Sampling Frequency: Upper Chehalis River	North Chehalis Channel	South Chehalis Channel	Humptulips Estuary	Total Number of Samples[a]
EPA Priority Pollutants/Hazardous Substances					
volatiles[b]	2	4	4	2	12
semi-volatiles[c]	2	2	2	1	7
metals[c]	2	2	2	1	7
PCB's/OC pesticides[c]	2	2	2	1	7
cyanide[c]	2	2	2	1	7
Other Toxics Analyses					
fatty acids, resin acids, guaiacols, catechols[c]	--	2[d]	2[d]	--	4
herbicides, OP pesticides carbamates[c]	2	--	--	1	3
sulfite/bisulfite[b]	2	4	4	1	11
ammonia[b,c]	4	6	6	1	17
total chlorine residual[b]	2	4	4	1	11
Ancillary Variables[e]					
specific conductivity[b,c]	4	6	6	1	17
salinity[b,c]	4	6	6	1	17
turbidity[b,c]	4	6	6	1	17
total hardness[b,c]	4	6	6	1	17
total organic carbon[b,c]	4	6	6	1	17
total suspended solids[b,c]	4	6	6	1	17

[a] QA samples (blanks, replicates, duplicates, matrix spikes) not included

[b] grab samples

[c] composite samples

[d] fish bile also analyzed for metabolites

[e] temperature, pH, D.O. and salinity monitored frequently by Fisheries

Table 3. Sampling frequency and analyses for Class II/biomonitoring surveys at Grays Harbor pulp mills in FY 88.

Analyses	Sampling Frequency: Weyco Whole Effluent	Weyco Estuary Sediment	ITT Whole Effluent	ITT Estuary Sediment	Total No. of Samples[a]
EPA Priority Pollutants/Hazardous Substances					
volatiles[b]	2	5	2	5	14
semi-volatiles[c]	1	5	1	5	12
PCB's/OC pesticides[c]	1	5	1	5	12
metals[c]	1	5	1	5	12
cyanide[c]	1	5	1	5	12
dioxins/furans[c]	1	2	1	2	6
Other Toxics Analyses					
fatty acids, resin acids,[c] guaiacols, catechols	1	5	1	5	12
ammonia[c]	1	--	1	--	2
sulfite/bisulfite[b]	2	--	2	--	4
total chlorine residual[b]	2	--	2	--	4
total phenolics[c]	1	--	1	--	2
total organic halogen[c]	1	--	1	--	2
Ancillary Variables[d]					
specific conductivity[c]	1	--	1	--	2
total hardness[c]	1	--	1	--	2
total suspended solids[c]	1	--	1	--	2
total organic carbon[c]	1	5	1	5	12
grain size	--	5	--	5	10
percent solids	--	5	--	5	10

[a] QA samples (blanks, replicates, duplicates, matrix spikes) not included

[b] grab samples

[c] 24-hour composite samples

[d] temperature, pH, and D.O. measured in field

Table 4. Ecology's ambient water quality monitoring program in the Chehalis and Humptulips drainages and Grays harbor (monthly samples).

Station Number	Description	Flow	D.O.	Temp	pH	Cond.	Fecal Coliform	Turb.	Color
A. River Basins									
22A070	Humptulips R. near Humptulips	X	X	X	X	X	X	X	X
22C050	Chehalis R. near Montesano	X	X	X	X	X	X	X	X
22G070	Satsop R. near Satsop[a]	X	X	X	X	X	X	X	X
23A070	Chehalis R. at Porter[a]	X	X	X	X	X	X	X	X
23A120	Chehalis R. at Centralia	X	X	X	X	X	X	X	X
23A160	Chehalis R. at Dryad	X	X	X	X	X	X	X	X

Station Number	Description	Depth (m)	D.O.	Temp	pH	Cond.	Salinity	Fecal Coliform	Turb.	Nutrients
B. Estuary[b]										
GYS 004	Grays Hbr at Standard Oil Dock	0, 10	X	X	X	X	X	X	X	X
GYS 007	Grays Hbr N. Channel near ITT	0, 10	X	X	X	X	X	X	X	X
GYS 009	Grays Hbr at Moon Island Reach	0, 10	X	X	X	X	X	X	X	X
GYS 008	Grays Hbr at Mid S. Channel	0, 10	X	X	X	X	X	X	X	X
GYS 015	Grays Hbr near Whitcomb Flats	0, 10	X	X	X	X	X	X	X	X
GYS 016	Grays Hbr near Damon Point	0, 10	X	X	X	X	X	X	X	X

[a] USGS NASQAN station, expanded analysis (anions, cations, etc.)

[b] collected April through November only

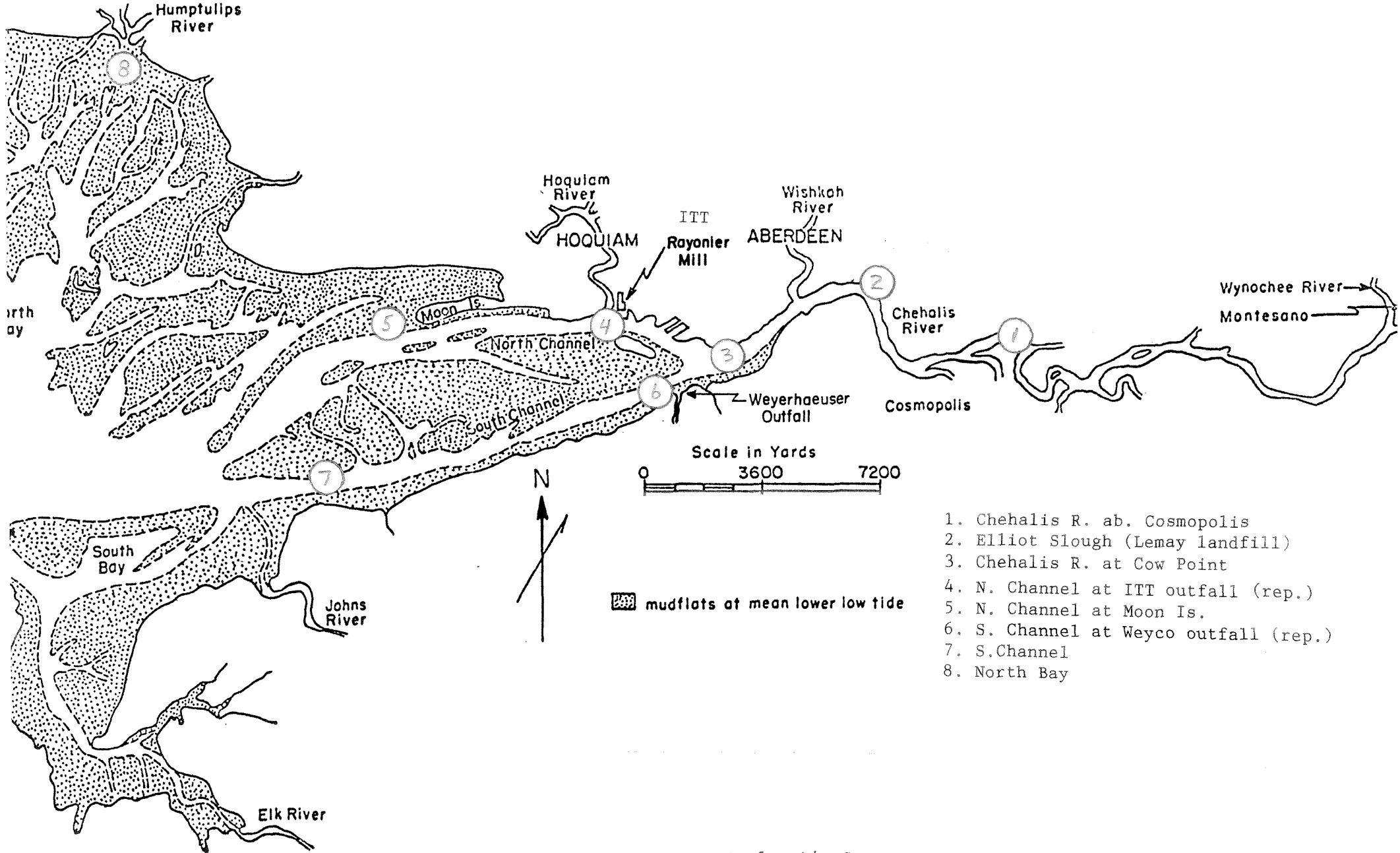

Figure 1. Sediment sample locations

APPENDIX I. List of chemicals to be analyzed.

VOLATILES

Acrolein
Acrylonitrile
Benzene
Carbontetrachloride
Chlorobenzene
1,2-Dichloroethane
1,1,1-Trichloroethane
1,1-Dichloroethane
1,1,2,2-Tetrachloroethane
Chloroethane
2-Chloroethylvinyl ether
Chloroform
1,1-Dichloroethylene
1,2-trans-Dichloroethylene
1,2-dichloropropane
cis-1,3-Dichloropropylene
trans-1,3-Dichloropropylene
Ethylbenzene
Methane,dichloro
Methane, chloro
Methane, bromo-
Methane, tribromo-
Methane, dichlorobromo-
Chlorodibromomethane
Tetrachloroethylene
Toluene
Trichloroethylene
Vinylchloride
Acetone
2-Butanone
Carbondisulfide
2-Hexanone
4-Methyl 2-pentanone
Styrene
Vinyl acetate
o-Xylene
Total Xylenes
"Library search" (10-compound tentative identification)
*Chloroacetone
*Dichloroacetone
*Methylmercaptan
*Hydrogen sulfide

*Special analytical request.

APPENDIX I. List of chemicals to be analyzed (page 2).

SEMI-VOLATILES

Phenol, 2,4,6-trichloro-
m-cresol, p- chloro-
Phenol 2-chloro-
Phenol, 2, 4-dichloro-
Phenol, 2, 4-dimethyl-
Phenol, 2-nitro-
Phenol, 4-nitro-
Phenol, 2,4-dinitro-
o-cresol, 4,6-dinitro
Phenol, pentachloro-
Phenol
Benzoic acid
Phenol, 2-methyl
Phenol, 4-methyl
Phenol, 2,4,5 trichloro-
Acenaphthene
Benzene, 1,2,4-trichloro-
Benzene, hexachloro-
Ethane, hexachloro-
Ether, bis(2-chloroethyl)
Naphthalene, 2-chloro-
Benzene, 1,2-dichloro-
Benzene, 1,3-dichloro-
Benzene, 1,4-dichloro-
Benzidine, 3,3'-dichloro-
Toluene, 2,4-dinitro-
Toluene, 2,6-dinitro-
Hydrazine, 1,2-diphenyl-
Flouranthene
Ether, 4-chlorophenyl phenyl
Ether, 4-bromophenyl phenyl
Ether, bis(2-chloroisopropyl)
Methane, bis(2-chloroethoxy)
Butadiene, hexachloro-
Cyclopentadiene, hexachloro-
Isophorone
Naphthalene
Benzene, nitro-
Nitrosamine, dimethyl-
Nitrosamine, diphenyl-
Nitrosamine, di-n-propyl
Phthalate, bis(2-ethylhexyl)
Pthalate, n-butyl benzyl
Phthalate, di-n-butyl
Phthalate, di-n-octyl
Phthalate, diethyl
Phthalate, dimethyl
Anthracene, benzo(a)-
Pyrene, benzo(a)-
Flouranthene, benzo(b)-
Flouranthene, benzo(k)-
Chrysene
Ancenaphthylene
Anthracene
Perylene, benzo(g,h,i)-
Flourene
Phenanthrene
Anthracene, dibenzo(a,h)
Pyrene, idenol(1,2,3-c,d)
Pyrene
"Library search" (10-compound) tentative identification)
*Alkylphenanthrenes
*Perylene
*Retene
*2-Chloropropenal
*Tetrachloropropene
*Pentachloropropene

*Special analytical request.

APPENDIX I. List of chemicals to be analyzed (page 3).

Polychlorinated biphenyls

PCB-1016
PCB-1221
PCB-1232
PCB-1242
PCB-1248
PCB-1254
PCB-1260

Organochlorine Pesticides

Aldrin
Chlordane
Dieldrin
DDT, 4, 4'
DDE, 4, 4'
DDD, 4,4'
Endosulfan, alpha
Endosulfan, beta
Endosulfan, sulfate
Endrin
Endrin aldehyde
Heptachlor
Heptachlor epoxide
BHC, alpha
BHC, gamma
BHC, delta
Toxaphene

Dioxins/Furans

chlorinated dibenzodioxins (dichloro-heptachloro)
chlorinated dibenzofurans (dichloro-heptachloro)

Fatty acids, resin acids, chlorinated phenolics

linoleic acid
linolenic acid
oleic acid
palustric acid
abietic acid
neoabietic acid
dehydroabietic acid
monochlorodehydroabietic acid
dichlorodehydroabietic acid
isopimeric acid
pimaric acid
levopimaric acid
sandaracopimaric acid
9,10-epoxystearic acid
9,10-dichlorostearic acid
3,4,5-trichlorosyringol
4,5-dichloroguaiacol
3,4,5-trichloroguaiacol
4,5,6-trichloroguaiacol
3,4,5,6-tetrachloroguaiacol
3,4,5-trichlorocatechol
3,4,5,6-tetrachlorocatechol
terpineol
eugenol
isoeugenol
dimethylcyclopentenones
trimethylcyclopentenones

APPENDIX I. List of chemicals to be analyzed (page 4).

Metals

Sb
As
Be
Cd
Cr
Cu
Pb
Hg
Ni
Se
Ag
Tl
Zn
Sn

Organophosphorus Pesticides

Mevinphos
Phorate
Dimethoate
Diazinon
Methylparathion
Ethylparathion
Malathion
Ethion
Carbophenothion
Azinphos (methyl or ethyl)
Dichlorvos
Dioxathion
Ronnel
Fenthion
Phosphamidon
Folex
DEF
Phencapton
Monocrotophos
EPN
Imidan
Coumaphos
Disulfoton

Other

nonylphenol ethoxylate

Herbicides

2,4-D
2,4-DB
2,4,5-T
2,4,5-TB
Silvex 4,5-TP
MCPA
MCPP
MCPB
Pentachlorophenol
Tetrachlorophenol
Dicamba
Bromoxynil
Ioxynil
Dinoseb
Picloram

Carbamate Pesticides

to be determined

Appendix II. Quality Assurance for Analysis of Priority Pollutants and Other Toxicants in Water

Sampling Procedures -- Table IIA gives the details of handling and preservation of water samples. ISCO automatic samplers (model 2710) modified with all teflon tubing and glass sample bottles, will be used for collecting composite samples during the barging studies and Class II inspections. Other samples will be grabs.

Analytical Protocols -- Analysis methods and associated detection limits for water samples are shown in Table IIB. The analyses will be done at the Ecology/EPA Manchester laboratory or by a contractor selected by Manchester. QA review of the data will be done by Manchester.

Accuracy and Precision -- The following routine procedures will be used to assess the accuracy and precision of the measurement data:

Laboratory -- Routine within-lab QA at Manchester includes analysis of duplicates, surrogate standards, matrix spikes, and methods blanks.

Field -- QA samples for field collections will consist of replicate samples (as described in the sampling plan) and field blanks. Both transport blanks (sample bottles filled with blank water and carried throughout the survey) and transfer blanks (blank water pumped through automatic samplers or transferred to empty sample bottles in the field) will be included in each collection.

Appendix Table IIA. Sampling procedures (selected toxicants in water).

Analysis	Sample Container[a]	Cleaning Procedure[b]	Preservation and Handling	Maximum Holding Time
volatiles	40 mL glass vial w/teflon septum	no. 3[c]	cool, 4°C	14 days
semi-volatiles	1 gallon glass w/teflon lid liner	no. 2[c]	cool, 4°C	7 days until extraction; 40 days until analysis
pesticides	1 gallon glass w/teflon lid liner	no. 2[c]	cool, 4°C	7 days until extraction; 40 days until analysis
dioxins/furans	1 gallon glass w/teflon lid liner	no. 2	cool, 4°C	7 days until extraction; 40 days until analysis
fatty acids, resin acids, guaiacols, catechols	1/2 gallon glass w/teflon lid liner	no. 2	cool, 4°C	7 days until extraction; 40 days until analysis
mercury	250 mL glass w/ teflon lid liner	no. 4	HNO_3 to pH < 2 cool, 4°C	28 days
other metals	1 liter polyethylene cubitainer w/teflon lid liner	no. 4[c]	HNO_3 to pH < 2 cool, 4°C	6 months
cyanide	250 mL polyethylene	no. 1	NaOH to pH > 12 (0.6 g ascorbic acid where residual chlorine) cool, 4°C	14 days
total phenolics	1 liter glass w/ teflon lid liner	new	phosphoric acid, copper sulfate, ferrous sulfate, cool, 4°C	28 days
total organic halogen	1 liter amber glass w/teflon lid liner	no. 2	cool, 4°C	28 days
sulfite/ bisulfite	to be determined			

[a] ICHEM or equivalent

[b] see Appendix Table IIC

[c] QC checked by ICHEM

Appendix Table IIB. Analytical protocols (selected toxicants in water).

Analysis	Lower Limit of Detection (ug/L)[a]	Method	Reference
volatiles	1-5	GC/MS (method 624)	EPA (1984)
semi-volatiles	1-5	GC/MS (method 625)	EPA (1984)
PCB's	0.1 - 1	GC/ECD (method 608)	EPA (1984)
OC pesticides	0.01 - 0.1	GC/ECD (method 608)	EPA (1984)
dioxins/furans	0.0001	GC/MS	EPA Duluth Laboratory Procedure
fatty acids, resin acids, guaiacols, catechols	1-5	GC/MS	NCASI (1986)
herbicides	0.01 - 0.	GC/ECD	EPA Region 10 procedure
mercury	0.05	cold vapor/AA (method 245.1)	EPA (1979)
arsenic, chromium, copper, nickel, lead, antimony, zinc	1	graphite or flame AA (methods 206.2, 218.1, 220.2, 249.1, 239.2, 204.2, 289.2)	EPA (1979)
silver, selenium, thallium	0.05	graphite or flame AA (methods 272.2, 270.2, 279.2)	EPA (1979)
cadmium	0.1	graphite AA (method 213.2)	EPA (1979)
total phenolics	5	automated colorimetric (method 420.2)	EPA (1979)
total organic halogen	5	carbon absorption/ microcoulometric-titration (method 450.1)	EPA (1979)

[a] 1 ug/L = 1 part per billion

References:

EPA 1979. Methods for Chemical Analysis of Water and Wastes. EMSL Cincinnati, OH EPA-600/4-79-020.

EPA 1984. Guidelines establishing test procedures for the analysis of pollutants under the Clean Water Act. Fed. Reg. Vol. 19 no. 209.

NCASI 1986. Procedures for the Analysis of Resin and Fatty Acids in Pulp Mill Effluents. Tech. Bull. 501.

APPENDIX TABLE IIC. Cleaning protocols.

Protocol 1

1. Wash containers and caps with hot tap water.
2. Wash with non-phosphate detergent and hot tap water.
3. Rinse with hot tap water.
4. Rinse with distilled water.
5. Air dry and place closures on containers.

Protocol 2

1. Wash containers, closures, and teflon liners in hot tap water with non-phosphate detergent.
2. Rinse three times with tap water.
3. Rinse with 1:1 nitric acid.
4. Rinse three times with deionized water.
5. Rinse with pesticide grade methylene chloride.
6. Oven Dry.
7. Remove containers, closures, and teflon liners from oven.
8. Place teflon liners in closures and place closures on containers. Attendant to wear gloves and containers not to be removed from preparation room until sealed.

Protocol 3

1. Wash vials, septa, and closures in hot tap water with non-phosphate detergent.
2. Rinse three times with tap water.
3. Rinse three times with deionized water.
4. Oven dry vials, septa, and closures.
5. Remove vials, septa, and closures from oven.
6. Place septa in closures, teflon side down and place on vials. Attendant to wear gloves and vials not to be removed from preparation room until sealed.

Protocol 4

1. Wash bottles, closures, and teflon liners with hot tap water with non-phosphate detergent.
2. Rinse three times with tap water.
3. Rinse with 1:1 nitric acid.
4. Rinse three times with deionized water.
5. Air dry in contaminant-free environment.
6. Place liners in closures and place closures on bottles. Attendant to wear gloves and bottles not to be removed from preparation room until sealed.

APPENDIX III. Target analytes for the EPA National Bioaccumulation Study.

Biphenyl
Butachlor
Chlorbenzilate
Chlordane, cis
Chlordane, trans

Chlorinated Dioxins & Furans:
- 2378-TCDD
- 12378-PeDD
- 123678-HxDD
- 123789-HxDD
- 123478-HxDD
- 1234678-HpDD
- 2378-TCDF
- 12378-PeDF
- 23478-PeDF
- 123678-HxDF
- 123789-HxDF
- 123478-HxDF
- 234678-HxDF
- 1234678-HpDF
- 1234789-HpDF

Chlorpyrifos
p,p'-DDE
Dicofol (Kelthane)
Dieldrin
Diethylhexylphthalate (DEHP)
Diphenyldisulfide
Endrin
Heptachlor
Heptachlor epoxide
Hexachlorobenzene (HCB)
Hexachlorocyclohexanes (BHC) including Lindane

Isopropalin
Kepone
Mercury
Methoxychlor
Mirex
n-alkanes (C_{20-32})
Nitrofen
Nonachlor, cis
Nonachlor, trans
Octachlorostyrene
Oxychlordane
Pentachloroanisole
Pentachlorobenzene
Pentachloronitrobenzene (PCNB)
Pentachlorophenol
Perthane
Polychlorinated Biphenyls:
- Mono-Decachlorinated Biphenyls
- 3,4,3',4'-Tetrachlorobiphenyl
- 3,4,5,3',4'-Pentachlorobiphenyl
- 3,4,5,3',4',5'-Hexachlorobiphenyl
- Total PCBs

1,2,4,5-Tetrachlorobenzene
1,2,3,4-Tetrachlorobenzene
1,2,3,5-Tetrachlorobenzene
1,2,3-Trichlorobenzene
1,2,4-Trichlorobenzene
1,3,5-Trichlorobenzene
Trichloronate
Trifluralin
Triphenyl phosphate