89-e21

Segment No. 10-22-04
10-22-12

WA-22-0030

WEYERHAEUSER, COSMOPOLIS CLASS II INSPECTION

by
Pat Hallinan

Washington State Department of Ecology
Environmental Investigations and Laboratory Services Program
Compliance Monitoring Section
Olympia, Washington 98504

May 1989

ABSTRACT

Ecology conducted a Class II inspection at the Weyerhaeuser pulp mill at Cosmopolis on May 23-25, 1988. The mill was meeting all effluent permit limits during the inspection. However, effluent toxicity was observed in pacific oyster (EC_{50} of 0.3 percent effluent) and mysid shrimp bioassays (LC_{50} of 58 percent effluent). Flouranthene concentrations in the effluent could account for the mysid mortalities. All other effluent organic concentrations were below acute water quality criteria. 4-Methylphenol, lead, nickel, and polyaromatic hydrocarbons (PAHs) in the sediment around the Weyerhaeuser outfall were mildly elevated compared to control sediments. Sediment samples showed no toxicity to the amphipod *Rhepoxynius abronius.*

INTRODUCTION

Ecology conducted a Class II inspection at the Weyerhaeuser pulp mill at Cosmopolis on May 23-25, 1988. Pat Hallinan and Carlos Ruiz from the Ecology Compliance Monitoring Section conducted the inspection. Steve Avery, the mill's water quality analyst, provided assistance.

This inspection was part of a larger study on the low survival of Chehalis River coho salmon smolts directed by the Washington State Department of Fisheries with cooperation from the U.S. Fish and Wildlife Service, U.S. EPA, the National Marine Fisheries Service and the University of Washington. Elements of the study included field collection of smolts, coho barging and holding studies, and bioassays using effluent from Weyerhaeuser, ITT Rayonier's pulp mill at Hoquiam, and the Aberdeen and Hoquiam wastewater treatment plants. Ecology and EPA supported the study with toxic chemical analyses of effluent and inner Grays Harbor estuary water column and bottom sediment samples. A similar Class II inspection at the ITT mill was also conducted. Results from this other Ecology work will be forthcoming.

The Weyerhaeuser mill uses the sulfite process to produce specialty grades of bleached pulp. Plant wastewater is routed through a series of aerated lagoons before being discharged into inner Grays Harbor. Non-contact cooling water discharges separately into the Chehalis River. NPDES (National Pollutant Discharge Elimination System) permit No. WA000080-9 regulates both discharges.

Objectives of this survey included:

1. Verify effluent compliance with NPDES limits.

2. Collect effluent samples for toxic pollutant analyses.

3. Collect sediments near the outfall for toxic pollutant analyses.

4. Evaluate effluent and sediment samples for acute and chronic toxicity using bioassays.

5. Review laboratory procedures at the mill to determine conformance with standard techniques and verify the accuracy of laboratory results by splitting samples with the permittee.

SITE DESCRIPTION

Process wastewater from the mill is treated by an activated sludge process and then a series of four lagoons located about six miles downstream on inner Grays Harbor. ITT's mill discharges treated effluent on the opposite side of the harbor from the lagoons (see Figures 1 and 2). The Weyerhaeuser activated sludge system treats about 8 MGD of high-strength plant wastewater. About 12 MGD of lower strength plant wastewater and the activated sludge effluent is pumped to the lagoons, designated ponds A, B, C, and D. Wastewater enters at pond A and discharges from pond D (outfall-001). Ponds B, C, and D are aerated. To control coliform growth in the lagoons, the pH is lowered to 2.5 to 3.0 by adding sulfuric acid at the outlet of pond C. Wasted sludge from the activated sludge treatment system is also disposed at the outlet of pond C. Non-contact cooling water discharges via a slough to the Chehalis River (outfall-002).

PROCEDURES

Ecology collected both cooling water discharge and pond D effluent composite samples. An ISCO automatic sampler collected about 220 mLs of cooling water discharge every 30 minutes for 24 hours. The compositor was located where the discharge enters the slough. Grab samples for field and laboratory analyses were also collected at this site and at the confluence of the slough and the Chehalis River (see Figure 2). Pond D effluent discharges twice a day on outgoing tides for 2 to 2 1/2 hour periods. ISCO automatic samplers collected about 800 mLs of sample every 10 minutes during these discharge periods. Grab samples were also collected at Pond D for field and laboratory analyses. Samples for bioassays were collected as grab composites; half the volume was collected during the a.m. discharge and the final volume was collected during the p.m. discharge.

Weyerhaeuser also collected a pond D effluent composite sample using a time-proportional sampler. Weyerhaeuser usually collects a composite cooling water discharge sample; however, their compositor was not working at the time of the inspection. Effluent composite samples were split for analysis between Ecology and the permittee for permit parameter analyses. In addition, the Ecology aerated lagoon effluent composite sample was split for priority pollutant analyses. Effluent sampling times and parameters analyzed are listed in Table 1. Analytical methods are given in Appendix 1.

The composite sampler used to collect pond D effluent for priority pollutant analyses was fitted with teflon tubing and a glass sampling bottle. This equipment was cleaned before use by washing with non-phosphate detergent and rinsing successively with tap water, 10 percent nitric acid, then three times with deionized water, pesticide grade methylene chloride, and with pesticide-grade acetone. Collection equipment was air-dried then wrapped in aluminum foil until used.

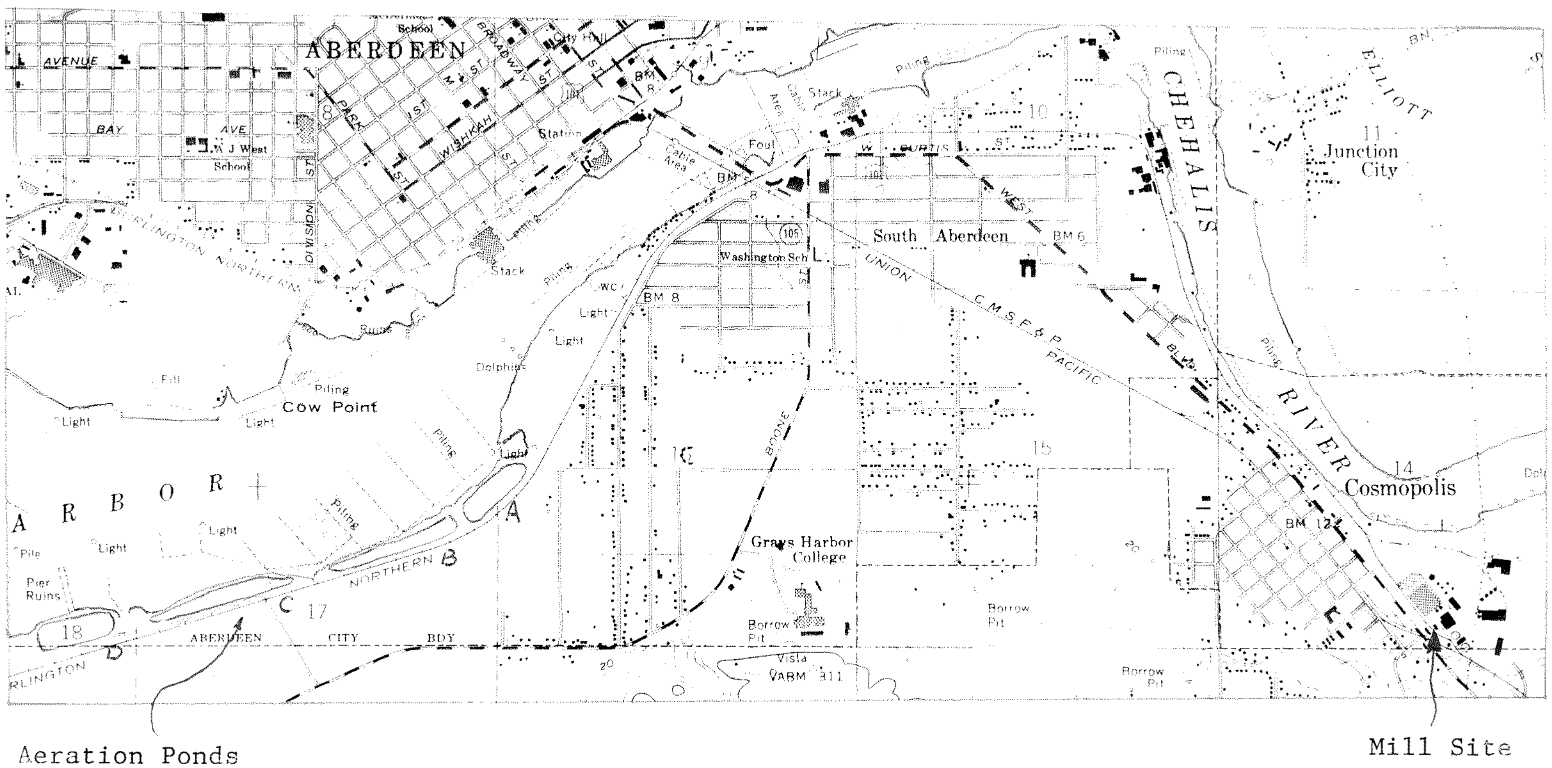

Figure 1. Site Location - Weyco, 5/88

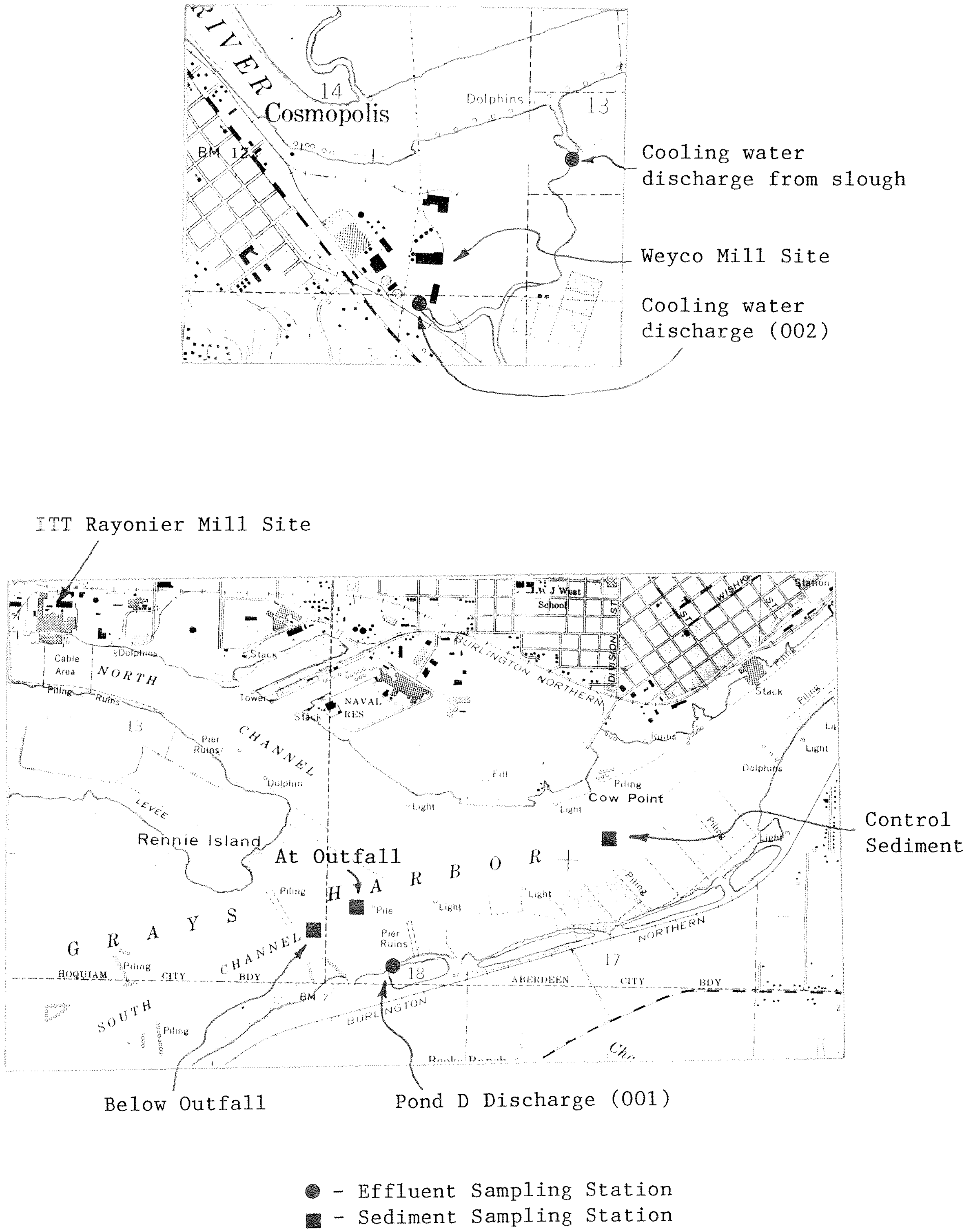

Figure 2. Sampling Locations - Weyco, 5/88

Table 1 - Sampling times and parameters analyzed - WEYCO 5/88.

Station	Date	Time	Sampler	Laboratory	Field Analyses: Temp. (°C)	Field Analyses: pH (S.U.)	Field Analyses: Cond. (umhos/cm)	Laboratory Analyses: D.O. (mg/L)	Fecal Coliform (#100/mL)	Oil & Grease (mg/L)	BOD_5	COD (mg/L)	Solids (mg/L): TS	Solids (mg/L): TNVS	Solids (mg/L): TSS	Solids (mg/L): TNVSS	Turb. (NTU)	Nutrients (mg/L): NH_3-N	Nutrients (mg/L): NO2+NO3+N	Nutrients (mg/L): Total-P	pH (S.U.)	Cond. (umhos/cm)	Alkalinity (Mg/L as $CaCO_3$)	Acidity (mg/L as $CaCO_3$)	Sulfite	Hardness (mg/L)	Sludge metals	% Solids	Grain size	Resid Acids	TOC (mg/L)	TOX (mg/L)	Cyanide (mg/L)	Priority Pollutants: BNA's	Priority Pollutants: Pest/PCB's	Priority Pollutants: VOA's	Priority Pollutants: Metals	Total Phenolics	Dioxins	Rhepoxinius	Bioassays: Ames Test	Bioassays: Trout	Bioassays: Mysid Shrimp	Bioassays: Pacific Oyster	Bioassays: Daphnia pulex
Outfall-001	May 24	0935			X	X	X		X	X		X	X	X	X	X		X	X	X																									
(Final Eff.)		2300			X	X	X		X	X		X	X	X	X	X		X	X	X					X											X									
		Comp	Ecol	Ecol							X	X	X	X	X	X	X	X	X	X	X	X	X	X		X				X	X	X	X	X	X		X	X			X	X	X	X	X
		(0845-1045)		Weyco							X				X																														
		(2130-2430)	Weyco	Ecol							X	X	X	X	X	X	X	X	X	X	X	X	X	X																					
				Weyco							X				X																														
Outfall-002	24	1055			X	X	X		X	X		X	X	X	X	X		X	X	X																									
(Cooling Water)		1541			X	X	X		X	X		X	X	X	X	X		X	X	X																									
	25	Comp	Ecol	Ecol							X	X	X	X	X	X	X	X	X	X	X	X	X																						
		(0600-0600)		Weyco							X				X																														
Discharge-002	24	1140			X	X	X		X	X		X	X	X	X	X		X	X	X																									
(Cooling Water)		1604			X	X	X	X	X	X		X	X	X	X	X		X	X	X																									
Waste Sludge	24																										X	X																	
Sediments	23																																												
Field Control		1230																										X	X	X	X		X	X	X	X	X		X	X					
At Outfall		1645																										X	X	X	X		X	X	X	X	X		X	X					
Below Outfall		1800																										X	X	X	X		X	X	X	X	X			X					

Two sites were sampled for sediments in the vicinity of the Weyerhaeuser outfall (see Figure 2): at the outfall line diffuser section ("at outfall") and about 100 yards downstream of the outfall ("below outfall"). Sediments off Cow Point were collected as a background reference sample ("field control"). The site at the diffuser section served as a quality control station for the sediment samples collected throughout the inner Grays Harbor estuary. Laboratory analysis variability was checked by duplicating chemical analyses on a single sediment composite sample collected at the outfall (designated "duplicates 1 and 2"; lab ID Nos. 228133 and 228135). Sampling variability was checked by collecting an additional composite sample at the outfall ("replicate"; lab ID No. 228134).

Sediment sampling followed recommended Puget Sound protocols (Tetra Tech, 1986). Composite sediment samples were taken using a 0.1 meter square van Veen grab. Composite samples consisted of three to four individual grabs in which the top 2 cm of sediment from each grab was collected. Composites were homogenized using stainless steel spoons and beakers, then divided for separate analyses. The stainless steel utensils were cleaned by the same procedures as the effluent composite sampler. Sediment sampling times and parameters analyzed are also listed in Table 1.

The toxicity of the sediment samples was assessed using the amphipod *Rhepoxynius abronius*. Pond D effluent toxicity was measured using Pacific oyster (*Crassostrea gigas*), *Daphnia magna*, and mysid shrimp (*Mysidopsis bahia*) bioassays and the Ames test. In addition, both Ecology and Weyerhaeuser conducted an effluent rainbow trout (*Salmo gairdneri*) bioassay. Bioassay test methods are listed in Appendix 1.

RESULTS

Flow

Flow data collected during the inspection are summarized in Table 2. Effluent flow is measured at pond D by ultrasonic meters attached to two submerged pipes. Effluent is then discharged through a single line to the outfall. Because the pipes were inaccessible, Ecology could not verify the accuracy of the meter. An independent means of verifying flow at this outfall is needed. Weyerhaeuser does not measure flow at outfall-002 (non-contact cooling water discharge). Plant personnel reported that the cooling water flow has been measured in the past and has always been 0.8 MGD. A weir should be constructed at outfall-002 so continuous readings can be made.

Comparison of Inspection Results to NPDES Permit Limits

Conventional pollutant data collected during the inspection are summarized in Table 3.

A comparison of effluent parameters to NPDES permit limits is given in Table 4. At outfall 001, BOD_5, TSS, and fecal coliform were well below the daily average and daily maximum permit limits. At outfall-002, all permit limits were met. Plant pulp production on the day of the inspection was 293 ADMT (air dried metric tons) per day of pH grade pulp.

Table 2. Flow measurement at outfall-001 - WEYCO, 5/88.

Date	Time	Totalizer Reading	Flow for Time Increment (MGD)	
5/24	0845	0		
			180	
	0933	6003		
			122	
	1100	13384		-End of discharge cycle 1-
	2130	13384		-Start of discharge cycle 2-
			124	
	2249	20204		
			122	
	2330	23665		

Total flow during inspection = 23.7 MGD

Table 3 - Ecology conventional analytical results - WEYCO, 5/88.

Station	Date	Time	Sampler	Field Analyses: Temperature (°C)	Field: pH (S.U.)	Field: Cond. (umhos/cn)	Field: D.O. (mg/L)	Laboratory Analyses: Fecal Coliform (#/100 mL)	Oil & Grease (mg/L)	BOD_5	COD (mg/L)	Solids (mg/L): TS	Solids: TNVS	Solids: TSS	Solids: TNVSS	Turbidity (NTU)	Nutrients (mg/L): NH_3-N	Nutrients: NH_2+NO_3-N	Nutrients: Total-P	pH (S.U.)	Cond. (umhos/cm)	Alkalinity (mg/L as $CaCO_3$)	Acidity (mg/L as $CaCO_3$)	Sulfite (mg/L)	Hardness (mg/L)	Cyanide (mg/L)	TOC (mg/L)	TOX (ug/L)	Total phenolics (mg/L)
	May																												
Outfall-001	24	0935		19.8	2.75	>1000		<3	1		840	2200	1100	110	37		0.06	0.03+	1.3**										
(Final Eff.)		2300		21.2	2.74	>1000			10		860	1900	920	120	<1		0.08	0.04+	1.4**					1.5					
		Comp	Ecol							87	890	2000	940	140	32	25	0.05	0.04+	1.6**	2.7	2380	0	300		370	<0.005	271	173	10
	(0845-1045)																												
	(2130-2430)		Weyco							84	830	1900	930	140	30	24	0.05	0.04+	1.5**	2.7	2380	0	290						
Outfall-002	24	1055		27.7	7.18	>1000		220*	<1		32	1000	840	4	16		2.0	0.09+											
(Cooling		1541		26.4	7.20	>1000		220*	7		39	790	610	<1	8		1.7	0.10+											
Water)	25	Comp	Ecol							<5	20	650	490	5	1	2	1.6	0.06+	0.29**	7.2	1170	160							
	(0600-0600)																												
Discharge-002	24	1140		16.8	7.13	390		470	8		15	300	230	4	19		0.28	0.06+	0.09**										
(Cooling Water)		1604		19.2	6.80	320	7.0	540	13		19	250	180	8	14		0.22	0.16+	0.15**										

* Many background organisms
\+ Questionable result due to an interfering substance
** Questionable result, exceeded analytical holding time

Table 4. Comparison of Class II inspection results to NPDES permit limits - WEYCO, 5/88.

	NPDES Permit Limits		Inspection Data	
	Daily Average*	Daily Maximum	Ecology Composite	Grab Samples
Outfall-001 (Final effluent)				
BOD5				
(mg/L)			87	
(lbs/D)	25,900	47,900	17,196	
TSS				
(mg/L)			140	
(lbs/D)	38,000	70,700	27,672	
Fecal coliform (#/100 mL)	5,000	20,000		<3
Flow (MGD)			23.7	
Rainbow Trout Bioassay	80 % survival in 65 % effluent		100 % survival	
Outfall-002 (Cooling Water)				
BOD5				
(mg/L)			<5	
(lbs/D)	--	500	<33	
D.O. (mg/l)	exceed 6.0			7.0
pH (S.U.)	6.5 to 8.5			6.8, 7.2
Flow (MGD)			0.8 (est)	

* - defined as the average over one month's time

The rainbow trout bioassay showed no mortality at a 65 percent effluent concentration. The mill has had past difficulties passing the bioassay limit (80 percent survival in 65 percent effluent). Weyerhaeuser failed the bioassay six of 15 times in 1985, nine of 20 in 1986, four of 14 in 1987, and five of 23 in 1988.

In 1986, Weyerhaeuser did a toxicity identification study as required by Ecology Order 86-663 (Campbell, 1987). The study found no specific cause of toxicity and concluded resin acids, chlorinated organics, bisulfite ion, and surfactant all contribute. Since 1986, control strategies have been introduced at the mill in an effort to reduce effluent toxicity.

Effluent Bioassays

Effluent bioassay results are given in Table 5. Some acute toxicity was observed in the Daphnia and the mysid shrimp bioassays. Of the two test organisms, the mysid shrimp was more sensitive: 90 percent mortality in 100 percent effluent (LC_{50} of 58 percent effluent) compared to 25 percent mortality for Daphnia.

The Pacific oyster bioassay indicated a high level of sub-lethal toxicity. The EC_{50} (effluent concentration resulting in 50 percent of larvae developing abnormal shells) was 0.3 percent. Pacific oyster larvae are particularly sensitive to pulp mill effluents. Biologically treated kraft mill effluent has been shown to cause impaired growth and development of oyster larvae at 1.3 percent effluent (EPA, 1979a). Pacific oyster results from the Ecology Class II inspection of the ITT pulp mill (also a chlorine bleach sulfite mill) showed similar sensitivity with an EC_{50} of 0.2 percent. An EC_{50} of 3.0 percent was observed from an earlier Ecology Class II inspection at an unbleached kraft mill in Port Townsend (Reif, 1987).

The Ames test measures a substance's potential to cause genetic damage (based on histidine reversion) to *Salmonella typhimurium* bacteria strains. Spent water from the chlorine bleaching of pulp is known to contain compounds that are mutagenic in the Ames assay (Kringstad, *et al.*, 1981). However, dilution of the spent bleaching water with other mill wastewater followed by biological treatment reduces the mutagenic effect (Voss, 1983; Sulkinoja-Salonen, *et al.*, 1981). The Weyerhaeuser effluent showed no mutagenic activity.

Effluent Chemistry

Appendix 1 gives the complete results of pond D effluent analyses for volatiles, semivolatiles, PCBs, pesticides and resin acids and other compounds. Table 6 lists the organic chemicals detected. Two volatile compounds were identified in small amounts: chloroform at 15 ug/L (parts per billion) and 2-hexanone at an estimated concentration of 0.6 ug/L. Another volatile, toluene, was found at 2 ug/L but was also detected in the method blank. Several semivolatile compounds were also identified, including numerous polyaromatic hydrocarbons (PAHs) up to 16 ug/L, benzoic acid at 7 ug/L, 2,4,6-trichlorophenol at 5 ug/L, and 4-methylphenol (para-cresol) at 37 ug/L. No chlorinated pesticides, herbicides, organophosphorus pesticides, or PCBs were detected.

Table 5 - Final effluent bioassay results - WEYCO, 5/88.

96 hour Rainbow trout (Salmo gairdneri) - 65 percent concentration

	# of live test organisms Initial	Final	Percent Mortality
Effluent	30	30	0
Control	30	30	0

48 hour Daphnia pulex - 100 percent concentration

	# of live test organisms Initial	Final	Percent Mortality
Effluent	20	15	25
Control	20	19	5

96 hour Mysid shrimp (Mysidopsis bahia)

Effluent	# of live test organisms Initial	Final	Percent Mortality
100%	20	2	90
30%	20	19	5
10%	20	20	0
3%	20	20	0
1%	20	20	0
Control	20	20	0

LC_{50} = 58% effluent (95% confidence limits 47% - 71%)

48 hour Pacific oyster (Crassostrea gigas)

EC_{50} = 0.3%

Ames test - Effluent showed no mutagenic activity

Table 6 - Organic chemicals detected in effluent and sediments - WEYCO, 5/88.

		Sediments (ug/kg dry wt.)				
	Effluent (ug/L)	@ Cow Point (Control Station)	@ Outfall Duplicate 1	Duplicate 2	Replicate	Below Outfall
Lab ID #:	228085	228132	228133	228135	228134	228136
% Fines*		50.2	21.0	20.1	37.0	57.5
% Sand		49.8	79.0	78.9	63.0	42.5
% Gravel		<2.0	<2.0	5.0	<2.0	<2.0
% Total Organic Carbon		1.3	0.6	0.8	0.9	1.6
% Dry Weight		53.5	71.8	67.5	58.6	53/0
Volatile organics:						
Chloroform	**15**	8 U	6 U	6 U	8 U	9 U
Toluene	**2 BJ**	8 U	6 U	0.1 U	0.2 U	0.3 U
2-Hexanone	**0.6 J**	16 U	12 U	13 U	16 U	17 U
Low molecular weight PAH's:						
Anthracene	**1 J**	100 U	76 U	77 U	**53 J**	**15 J**
Acenaphthene	**2**	100 U	**31 J**	**30 J**	**71 J**	**24 J**
Phenanthrene	**11**	**51 J**	**110**	**120**	**530**	**120**
Fluorene	**2**	**10 J**	**23 J**	**22 J**	**79 J**	**27 J**
1-Methylnaphthalene	1 U	100 U	**20** J	77 U	91 U	100 U
Naphthalene	**1 J**	**34 BJ**	**45 BJ**	**44 BJ**	**110 B**	**87 BJ**
2-Methylnaphthalene	**0.1 J**	**9 J**	**11 J**	**11 J**	**13 J**	**19 J**
Acenaphthylene	**0.3 J**	**7 J**	**4 J**	**4 J**	**8 J**	**13 J**
High molecular weight PAH's:						
Benzo(a)Anthracene	**4**	**16 J**	**19 J**	**23 J**	**91 J**	**23 J**
Chrysene	**11**	100 U	76 U	77 U	**57 J**	18 U
Benzo(k)Fluoranthene	**3**	100 U	76 U	77 U	91 U	100 U
Benzo(b)Fluoranthene	1 U	100 U	76 U	**20 J**	**39 J**	100 U
Pyrene	**11**	**76 J**	**110**	**120**	**640**	**120**
Fluoranthene	**16**	**57 J**	**95**	**100**	**840**	**88 J**
Phenols:						
4-Methylphenol	**37**	**51 J**	**46 J**	77 U	**130**	**520**
Phenol	**0.8 J**	**16 BJ**	76 U	77 U	91 U	**24 BJ**
2,4,6-Trichlorophenol	**5.0**	100 U	76 U	77 U	91 U	100 U
Phthalates:						
Diethylphthalate	**0.6 J**	100 BU	76 BU	77 U	91 BU	100 BU
Di-n-Butylphthalate	1 U	**29 BJ**	**25 BJ**	**33 BJ**	91 BU	**35 BJ**
bis(2-Ethylhexyl)Phthalate	1 U	**97 BJ**	**320 B**	**270 B**	**82 BJ**	**46 BJ**
Di-n-Octyl Phthalate	1 U	**15 BJ**	76 BU	**46 BJ**	91 BU	100 BU
Miscellaneous:						
N-Nitrosodiphenylamine	1 U	100 U	76 U	77 U	91 U	**12 J**
Retene	1 U	**110**	76 U	**64 J**	**120**	**150**
Dibenzofuran	1 J	100 U	**19 J**	**19 J**	**31 J**	**21 J**
Benzoic Acid	**7**	480 U	370 U	370 U	440 U	480 U
Resin and Fatty Acids/ Guaiacols:						
Tetrachloroguaicol	**3**	100 U	76 U	77 U	91 U	100 U
Trichlorosyringol	**13**	100 U	76 U	77 U	91 U	100 U
Linoleic acid	0.6 U	**630 J**	76 U	**170 J**	**440 J**	**540 J**
Oleic acid	**5 B**	**1500**	**1400 J**	**510 J**	**740 J**	**800 J**
Dehydroabietic acid	**0.6 BU**	**250 BJ**	**140 B**	**170 JB**	**260 JB**	**400 JB**
Abietic acid	0.6 U	100 U	76 U	**29 J**	**66 J**	**130 J**
Dichlorodehydroabeitic acid	**4**	100 U	76 U	77 U	91 U	100 U

* - Silt + Clay (<4um-62um)
U - Not detected at detection limit shown
J - Estimated Concentration
B - Also detected in method blank

Volatile organics found in the effluent were well within Washington State water quality criteria (EPA, 1986) for protection of aquatic life (Table 7). Concentrations of 4-methylphenol (37 ug/L) were also below acute levels. The toxic threshold of 4-methylphenol to Daphnia is 12,000 ug/L (McKee and Wolf, 1963).

Flouranthene concentrations could account for the mortality in the mysid bioassay. This mysid species is very sensitive to fluoranthene, detected at 16 ug/L in the effluent. Fluoranthene causes acute and chronic effects to the mysid at 40 and 16 ug/L, respectively (EPA, 1980). No toxicity data are available for the other high molecular weight PAHs found: pyrene at 11 ug/L, chrysene at 11 ug/L, benzo(a)-anthracene at 4 ug/L, and benzo(b)-fluoranthene at 3 ug/L. PAHs may be typically found in pulp and paper mill effluents (EPA, 1981).

The resin acid scan identified four compounds in the effluent: terachloroguaciacol at 3 ug/L, trichlorosyringol at 13 ug/L, oleic acid at 5 ug/L, and dichlorodehydroabietic acid at 4 ug/L. Ninety-six-hour juvenile rainbow trout LC_{50} (lethal concentration to 50 percent of the bioassay test organisms) for tetrachloroguaiacol and dichlorodehydroabietic acid are 320 and 600 ug/L, respectively (EPA, 1979a). Rainbow trout LC_{50} for other resin/fatty acids and guaiacols vary from 600 to 2,500 ug/L (EPA 1979a)

Metals found in the effluent are listed in Table 8. Silver and selenium were detected in the transfer blank; therefore, effluent results for these two metals are questionable. Copper and nickel were above criteria set by EPA (EPA, 1986) for protection of saltwater aquatic life (Table 7). All other metals were below freshwater and saltwater criteria. However, the effluent was analyzed for total metals which may overestimate concentrations bioavailable to aquatic life. EPA recommends criteria values be compared to total recoverable metals. Given this possible overestimation and the dilution of the effluent, copper and nickel would not be expected to have receiving water impacts. Metals analyses of waste activated sludge samples are also listed in Table 8. Chromium, copper, nickel, and zinc were found in the largest amounts. These metals, except chromium, were also detected in the effluent in the largest amounts.

Both the resin acid scan and semivolatile scan tentatively identified numerous organic compounds in the effluent. These are listed in Table 9. Only one chlorinated organic compound was identified, 1,3,5-trichloro-2-methoxy-benzene, at 3.3 ug/L. An attempt to obtain aquatic toxicity information on this compound using the Chemical Information System (CIS) data base was unsuccessful.

Sediment Bioassay

Results of the amphipod bioassay are given in Table 10. No sediment sample showed any significant toxicity. Mortality and avoidance among samples were similar to the laboratory control. The percentage of amphipods able to rebury after the ten-day exposure period, a measure of sub-lethal effects, was near or at 100 percent for all samples.

Table 7. Comparsion of organics and metals to EPA/Ecology water quality criteria - WEYCO 5/88.

		EPA Water Quality Criteria+			
		Freshwater		Saltwater	
	Effluent* (ug/L)	Acute (ug/L)	Chronic (ug/L)	Acute (ug/L)	Chronic (ug/L)
Volatile organics					
Chloroform	15	28,900	1,240	--	--
2-Hexanone	0.6J	- No Criteria -			
Toluene	2 BJ	17,500	---	6,300	5,000
Phenols					
4-Methylphenol	37	- No Criteria -			
2,4,6 Trichlonphenol	5.0	--	970	--	--
Phenol	6.8J	10,200	2,560	5,800	--
Total PAHs	60	--	--	300	--
Miscellaneous					
Benzoic Acid	7	- No Criteria -			
Diethl phthalate	0.6J	- No Criteria -			
Metals (total)					
Arsenic	3	360	190	69	36
Copper	16	61	36	2.9	--
Nickel	110	4,290	480	--	8.3
Zinc	14	355	320	95	86

* Hardness = 370 mg/L as $CaCO_3$
+ Criteria for metals based on total recoverable method

Table 8 - Metal concentrations in effluent and sediments - WEYCO, 5/88.

			Sediments (mg/kg dry)				
			@ Cow Point (Control Station)	@ Outfall			Below Outfall
Lab ID #:	Effluent (ug/L) 228085	Waste Sludge** (mg/kg dry) 228093	228132	Duplicate 1 228133	Duplicate 2 228135	Replicate 228134	228136
% Fines*			50.2	21.0	20.1	37.0	57.5
% Sand			49.8	79.0	78.9	63.0	42.5
% Gravel			<2.0	<2.0	5.0	<2.0	<2.0
% Total Organic Carbon			1.3	0.6	0.8	0.9	1.6
% Dry Weight			53.5	71.8	67.5	58.6	53.0
Arsenic	**3**	0.18 U	**3.9**	**3.5**	**3.4**	**3.6**	**3.8**
Lead	1 U	**0.82**	0.5 U	**0.7**	**1.8**	**1.2**	**3.2**
Thallium	1 U	0.06 U	0.1 U	0.1 U	0.1 U	0.1 U	0.1 U
Silver	**4.2**	**0.27**	**0.05**	0.02 U	0.02 U	0.02 U	0.02 U
Antimony	1 U	**0.35**	0.1 U	0.1 U	0.1 U	0.1 U	0.1 U
Selenium	**10**	0.06 U	**0.8**	**1.0**	**1.3**	**0.9**	**0.9**
Mercury	0.034 U	**0.01**	**0.037**	**0.033**	**0.046**	**0.053**	**0.091**
Beryllium	1 U	**0.12**	**1**	**1**	**0.9**	**1.0**	**1.1**
Cadmium	5 U	0.29 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U
Chromium	10 U	**6.9**	**30**	**29**	**27.9**	**27**	**28.7**
Copper	**16**	**11.4**	**44**	**44**	**40**	**45**	**51.5**
Nickel	**110**	**36.0**	**56**	**65**	**62.5**	**61**	**57.5**
Zinc	**14**	**41.9**	**71**	**74**	**73**	**75**	**77.1**
Tin	NA	NA	**96**	**109**	**105**	**103**	**101**

NA - not analyzed
U - Not detected at detection limit shown
* - Silt + Clay (<4um - 6um)
** - % Solids = 1.7%

Table 9 - Tentatively identified compounds in effluent and sediment - WEYCO, 5/88.

		Sediments (ug/kg dry wt.)				
		@ Cow Point	@ Outfall			
	Effluent	(Control				Below
	(ugL/)	Station)	Duplicate 1	Duplicate 2	Replicate	Outfall
Lab ID #:	228085	228132	228133	228135	228134	228136
Chlorinated Organics:						
Benzene, 1,3,5-trichloro-2-methoxy-	3.3 J					
2-Propanol, 1,1,1-trichloro-2-methyl-		130 J				60 J
1,1,2,2-Terachloroethane		310 J	72 J			
Other Oorganics:						
2-Butanoic acid, 2-methyl-, methyl ester	250 J					
2-Butenoic acid, 3-methyl-, methyl ester	1.8 J					
Pentanoic acid, 2-methyl-, methyl ester	62 J					
Ethanone, 1-(2-furanyl)-	4.4 J					
Methyl 2-furoate	41 J					
Benzene, 1-methoxy-4-methyl-	2.3 J					
Heptanoic acid, 2-methyl-, methyl ester	2.2 J					
Ethanol, 1-(2-butoxyethoxy)-	15 J					
Benzenepropanoic acid, methyl ester	6.6 J					
Ethanol, 2-(2-butoxyethoxy)-, acetate	63 J					
Cyclohexanecarboxylic acid, 4-(1,5-dimethyl-3-oxohexyl)	7.6 J					
Heptadecanoic acid, 16-methyl-, methyl ester	13 J					
Tetracosanoic acid, methyl ester	21 J	12000 J	11000 J	6600 J	9900 J	1400 J
Pentadecanoic acid, 14-methyl-, methyl ester	7.4 J			2300 J		
9-Octadecenoic acid, 12-(acetyloxy)-, methyl ester	9.4 J					
2-Hexanone, 5-methyl-	34 J	3000 J	710 J		2100 J	2500 J
Heptadecane	16 J	650 J				
Heptadecane, 2,6-dimethyl-	14 J					
Hexadecanoic acid	46 J					
Benzo[b]naphtho[2,1-d]thiophene	11 J					
Tetradecanoic acid, 12-methyl-, methyl ester (S)-				790 J		
Hexadecanoic acid		2300 J		1400 J	3400 J	1800 J
Pentacosane		1800 J				
2-cyclohehexen-1-one		160 J				
Benzaldehyde (acn)(dot)		100 J				
1H-pyrrole-2,5-dione, 3-ethyl-4-methyl-		190 J				
Tetradecanoic acid		700 J		350 J	850 J	330 J
Tetradecanoic acid, 12-methyl-, (S)-	6.8 J	570 J	1300 J	400 J	650 J	
Pentadecanoic acid		1100 J	480 J	160 J	150 J	740 J
1H-naphtho[2,1-B]pyran, 4A,5,6,6A,7,8,9,10,10A-deca		300 J				
Heptadecenoic acid		790 J	360 J	580 J		
9-Octadecenoic acid (Z)-, methyl ester		1600 J		1000 J		
Pentatriacontane		1300 J				
Hexatriacontane		860 J				
10-Octadecenoic acid, methyl ester		3600 J		2100 J		3100 J
Eicosanoic acid, methyl ester		3300 J		1100 J	2800 J	
Hexanedioic acid, mono(2-ethylhexyl)ester				4500 J		
Hexacosanoic acid, methyl ester		5600 J	3100 J	2500 J		7500 J
Octacosanoic acid, methyl ester				2400 J		
2-Heptanol acetate				140 J		
Octanoic acid, methyl ester		87 J		64 J		170 J
Tetradecanoic acid, methyl ester		1100 J	630 J	410 J	810 J	1700 J
Tetradecanoic acid, 12-methyl-, methyl ester		1900 J	1400 J	630 J	1700 J	2800 J
Pentadecanoic acid, methyl ester		590 J	360 J	230 J		920 J
9-Hexadecenoic acid, methyl ester, (Z)-		4100 J	2500 J	760 J	690 J	2600 J
Tridecane, 5-propyl-						160 J
Hexathiepane					130 J	120 J

J - estimated concentration

Table 9. Continued.

		Sediments (ug/kg dry wt)				
		@ Cow Point	@ Outfall			
Lab ID #:	Effluent (ugL/) 228085	(Control Station) 228132	Duplicate 1 228133	Duplicate 2 228135	Replicate 228134	Below Outfall 228136
Pentadecanoic acid, 14-methyl-, methyl ester			800 J		1300 J	960 J
9-Octadecenoic acid, methyl ester, (E)-						900 J
Triacontanoic acid, methyl ester				400 J		340 J
3-Hexen-2-one, 5-methyl-		200 J	84 J			82 J
1,2-Benzenedicarboxylic acid, butyl 2-methylpropyl ester			1300 J			
9-Hexadecenoic acid			1100 J		2800 J	
Heptadecanoic acid			360 J			
Stigmast-4-en-3-one			570 J			
1-Naphthalenecarboxylic acid					82 J	
9-Octadecenoic acid, 12-(acetyloxy)-, methyl ester, [R]		6200 J		760 J		
11-Hexadecenoic acid, methyl ester				1200 J		
Hexadecanoic acid, 15-methyl-, methyl ester				370 J	260 J	
Hexadecanoic acid, 14-methyl-, methyl ester				150 J		230 J
Heptadecanoic acid, 15-methyl-, methyl ester				1500 J		3300 J
1-Eicosyne				300 J		
Hexadecanedioic acid, dimethyl ester				350 J		
1-Phenanthrenecaroxylic acid, 1,2,3,4,4A,9,10,10A-octa		530 J		210 J		1100 J
Docosanoic acid, methyl ester				5400 J		
Tricosanoic acid, methyl ester		1800 J		700 J	1400 J	
Pentacosanoic acid, methyl ester			610 J	570 J		
Heptacosanoic acid, methyl ester			430 J	260 J		
3-Penten-2-one, 4-methyl-			390 J		490 J	1100 J
Hexanoic acid, methyl ester		74 J	52 J		62 J	120 J
1,3-Dithiolane		110 J	260 J		660 J	190 J
Heptanoic acid, methyl ester		69 J				130 J
9-Octadecenoic acid, 12-(acetyloxy)-, methyl ester,[R]		6200 J	3800 J		5100 J	5700 J
Tetradecanoic acid, 5,9,13-trimethyl-, methyl ester						560 J
Heptadecanoic acid, 16-methyl-, methyl ester		3300 J	2300 J		3200 J	3200 J
1-Phenanthrenecarboxylic acid, 7-ethyltetradecahydro-1		560 J				910 J
1H-Imidazole, 2,4-dimethyl-					170 J	
2-Cyclohexen-1-one			56 J		79 J	
Trisulfide, dimethyl					66 J	
Disulfide, methyl (methylththio)methyl					130 J	
9H-Fluorene-9-carboxylic acid					95 J	
Pyrene					370 J	
1-Phenanthrenecarboxylic acid, tetradecahydro-1,4A-dimethyl					350 J	
Triacontanoic acid, methyl ester			7800 J		6400 J	
5-Hexen-2-one, 5-methyl-			84 J			
Hexadecanoic acid, 14-methyl-, methyl ester			390 J			
2-Cyclohexen-1-one, 3,5-dimethyl-, o-methyloxime		910 J				
1,3-Dioxolane, 2,2,4-trimethyl-		94 J				
Hexanedioic acid, dioctyl ester		1300 J				440 J

J - estimated concentration

Table 10. Sediment (Rhepoxynius abronius) bioassay results - WEYCO, 5/88.

Sample	Lab ID	Mean values +/- S.D. Survival*	Avoidance+	%Reburial**
Field Control	#228132	18.6+/-1.1	0.1+/-0.3	98.9
At Outfall	#228133	19.4+/-0.9	0.1+/-0.4	100.0
Below Outfall	#228136	18.6+/-0.5	0.3+/-0.7	100.0
Lab Control		18.8+/-1.6	0.7+/-1.1	100.0

* Value of 20.0 = 100%
\+ No. of amphipods on the surface of jar per day
** At end of ten-day exposure, surviving amphipods were transfered to clean sediment. % Reburial indicates the number able to rebury after one hour.

Sediment Chemistry

Appendix 1 lists the complete results of sediment analyses for volatiles, semivolatiles, pesticides, and PCBs and resin acids. Table 6 lists the organic chemicals detected and Table 8 gives the metals detected.

Duplicate analyses on sediment samples collected at the outfall agreed closely. Analyses of the additional independent sample (replicate) at the outfall detected two to five times higher levels of semivolatile organics than the duplicate analyses. Resin acids and metals in the replicate analyses agreed closely with the duplicate analyses.

No volatile organic, pesticides, or PCBs were detected in any sediment samples. However, several semivolatile compounds were found which included polyaromatic hydrocarbons (PAHs), phthalates, and 4-meythlphenol. In most cases, compounds detected in the effluent also appeared in sediments around the outfall.

Table 11 gives the results of analysis for dioxin and furans in the sediments. 2,3,7,8-Tetrachlorodibenzo-p-dioxin (TCDD), the most toxic of the polycholorinated dioxins, was not detected at detection limits of 0.85 ng/kg (parts per trillion) at Cow Point and 0.68 ng/kg at the outfall. However, other dioxin isomers were detected. Octa- CDD (OCDD), total hepta CDD (HpCDD), and 1,2,3,4,6,7,8-HpCDD were found at 140, 42, and 18 ng/kg, respectively, at Cow Point and at 120, 25, and 11 ng/kg at the outfall. In addition, one dibenzofuran, 2,3,7,8-TCDF, was found at 2.8 ng/kg at Cow Point. Dioxin and furan concentrations do not appear elevated compared to Cow Point reference sediments.

Of the dioxins/furans detected, 2,3,7,8-TCDF and 1,2,3,4,6,7,8-HpCDD are the most toxic (Palmer, *et al.*, 1988). Biological effects of these two chlorinated compounds in aquatic sediments are unknown. However, bioassay results suggest they are not at levels high enough to cause acute effects to Rhepoxynius.

Several resin and fatty acids were detected in the sediments. Oleic acid was found in the largest amount: 1500 ug/kg (parts per billion) at Cow Point. Other acids including linoleic, dehydroabietic, and abietic were detected in the sediments at 400 to 800 ug/kg. Acids in the vicinity of the outfall do not appear elevated compared to levels at Cow Point. The resin/fatty acid and dioxin/furan results suggest sediments off Cow Point may not have been far enough away from the influence of the two pulp mills to serve as a true control.

PAH and 4-methylphenol concentrations were elevated around the Weyerhaeuser outfall (by a factor of two to ten times) compared to concentrations at Cow Point. Nickel and lead were mildly elevated around the outfall. All other metal concentrations were uniform at the three sites.

Semi-volatile compounds, except 4-methylphenol and N-nitrosodiphenylamine, found in all sediments were far below the apparent effects threshold (AET) levels being developed for Puget Sound sediments (Table 12). Chemical concentrations above AET levels are predicted to adversely effect sediment benthic infauna and/or sediment bioassay test organisms.

Table 11. Results of sediment dioxin/furan analyses - WEYCO, 5/88.

	@ Cow Pt (ng/kg)*	@ Outfall (ng/kg)*
Lab #:	228144	228142
Dioxins:		
TCDDs (Total	0.85 U	0.68 U
2,3,7,8-TCDD	0.85 U	0.68 U
PeCDDs (total)	8.6 U	4.0 U
1,2,3,7,8-PeCDD	8.6 U	4.0 U
HxCDDs (total)	11 U	3.7 U
1,2,3,4,7,8-HxCDD	11 U	3.7 U
1,2,3,6,7,8-HxCDD	11 U	3.7 U
1,2,3,7,8,9-HxCDD	11 U	3.7 U
HpCDDs (total	**42**	**25**
1,2,3,4,6,7,8-HpCDD	**18**	**11**
OCDD	**140**	**120**
Furans:		
TCDFs (totals)	**2.8**	0.63 U
2,3,7,8,-TCDF	**2.8**	0.63 U
PeCDFs (total)	9.4 U	1.5 U
1,2,3,7,8-PeCDF	9.4 U	1.5 U
2,3,4,7,8-PeCDF	9.4 U	1.5 U
HxCDFs (total)	5.7 U	1.8 U
1,2,3,4,7,8-HxCDF	5.7 U	1.8 U
1,2,3,6,7,8-HxCDF	5.7 U	1.8 U
2,3,4,6,7,8-HxCDF	5.7 U	1.8 U
1,2,3,7,8,9-HxCDF	5.7 U	1.8 U
HpCDFs (totals)	7.7 U	3.5 U
1,2,3,4,6,7,8-HpCDF	7.7 U	3.5 U
1,2,3,4,7,8,9-HpCDF	7.7 U	3.5 U
OCDF	21 U	14 U

* - ng/kg = parts per trillion (ppt)
U - not detected at detection limit shown

Table 12. Comparison of AET values to compounds detected in sediments - WEYCO, 5/88.

	Sediments (ug/kg, dry)				AET Values	
	@ Cow Point (Control Station)	@ Outfall		Below Outfall		
		Duplicate*	Replicate			
Lab ID :	#228132	#228133,-35	#228134	#228136	LAET	ACR NOEC
Low molecular weight PAHs						
Anthracene			53 J	15 J	960	1,300
Acenaphthene		31 J	71 J	24 J	500	200
Phenanthrene	51 J	115	530	120	1,500	690
Fluorene	10 J	23 J	79 J	27 J	540	360
1-Methylnaphthalene		20 J			--	--
Naphthalene	34 BJ	45 BJ	110 B	87 BJ	2,100	270
2-Methylnaphthalene	9 J	11 J	13 J	19 J	670	190
Acenaphthylene	7 J	4	8 J	13 J	--	--
TOTAL LPAH	111	248	864	305	5,200	2,400
High molecular weight PAHs						
Benzo(a)Anthracene	16 J	21 J	91 J	23 J	1,300	510
Chrysene			57 J		1,400	920
Benzo(b)Fluoranthene		20	39 J		--	--
Pyrene	76 J	115	640	120	2,600	1,600
Fluoranthene	57 J	98	840	88 J	2,500	4,170
TOTAL HPAH	149	254	1,667	331	12,000	6,900
Phenols						
4-Methylphenol	51 J	62 J	130	520	670	360
Phenol	16 BJ			24 BJ	420	120
Phthalates						
Diethylphthalate	100 BJ				200	20
Di-n-Butylphthalate		29 BJ		35 BJ	1,400	140
bis(2-Ethylhexyl)Phthalate	97 BJ	295 B	82 BJ	46 BJ	1,300	190
Di-n-Octyl Phthalate	15 BJ	46			1,100	620
Miscellaneous						
N-Nitrosodiphenylamine				12 J	28	13
Retene		64	120	150	--	--
Dibenzofuran		19 J	31 J	21 J	540	170

* - Average of duplicate samples 228133 and 228135
u - Not detected at detection limit shown
J - Estimated concentration
B - Also detected in method blank

Two AET values are shown in Table 12. The lowest apparent effects threshold, LAET, represents a concentration above which acute effects may occur. The acute to chronic ratio no observable effects concentration, ACR NOEC, represents a concentration above which sub-lethal effects may occur (Ecology, 1988). 4-Methylphenol below the outfall (520 ug/kg) were above the ACR NOEC of 360 ug/kg. N-nitrosodiphenylamine below the outfall (estimated concentration of 12 ug/kg) approached the ARC NOEC of 13 ug/kg.

Sediment metal concentrations, except chromium and nickel, were also below AET levels (Table 13). Chromium was only slightly above the ACR NOEC while nickel was about four times greater than the ACR NOEC.

Sediment resin acid and semivolatile scans also tentatively identified several compounds which are listed in Table 9.

Comparison of Lab Results

A comparison of laboratory results is given in Table 14. BOD_5 and TSS results from both labs agreed closely. Ecology's result for acidity was higher than Weyerhaeuser's. Weyerhaeuser is required to monitor acidity; however, no limits are specified in the permit.

A comparison of priority pollutant results is given in Table 15. Weyerhaeuser duplicated Ecology analyses on pond D effluent for total phenolics, volatiles, semivolatiles, and pesticides/PCBs. Detection limits for both labs were similar for the semivolatile scan. For the volatile analyses, Ecology's detection limits were about ten times lower than Weyerhaeuser's. For the pesticide/PCB analyses, Ecology's detection limits were three to five times lower than Weyerhaeuser's.

There was general agreement between the labs that the effluent contained low concentrations of chloroform, phthalates, phenol and trichlorophenol. However, the PAH and total phenolic results varied. Ecology measured 10 mg/L of total phenols, while the Weyerhaeuser result was 0.27 mg/L. The Weyerhaeuser sample was not properly preserved in the field by addition of copper sulfate and ferrous ammonium sulfate, as per EPA method 420 (EPA, 1979b). This may account for the difference.

Total PAHs detected by Ecology were 60 ug/L while Weyerhaeuser detected only 6 ug/L. Weyerhaeuser did not analyze for acenaphthene, 2-methyl-naphthalene, benzo(k)-fluoranthene, 4-methylphenol, dibenzofuran, or benzoic acid. No pesticides or PCBs were detected by either lab.

LABORATORY REVIEW

Laboratory procedures at the mill were in conformance with standard techniques. The mill lab was clean and well organized. The mill's water quality analysts perform the BOD and fecal coliform tests; environmental operators perform the TSS test. No written procedure for the TSS test is used; one should be made to give environmental operators guidance. A complete laboratory review sheet is included in Appendix 2 of this report.

Table 13. Comparsion of metal concentrations in sediments to AET values - WEYCO, 5/88.

Lab ID:	(Control Station) #228132	At Outfall Duplicate* #228133,-35	At Outfall Replicate #228134	Below Outfall #228136	AET Values LAET	AET Values ACR NOEC
Arsenic	3.9	3.5	3.6	3.8	57	57
Lead		1.3	1.2	3.2	450	66
Silver	0.05				5.9	0.59
Selenium	0.8	1.2	0.9	0.9	- No AET -	
Mercury	0.037	0.040	0.053	0.091	0.41	0.21
Beryllium	1	1.0	1.0	1.1	- No AET -	
Chromium	30	29.2	27	28.7	260	27
Copper	44	42	45	51.5	390	130
Nickel	56	63.8	61	57.5	140	14
Zinc	71	73.5	75	77.1	410	160
Tin	96	107	103	101	- No AET -	

* - Average of duplicate samples 228133 and 228135
All units in mg/kg dry wt

Table 14. Comparison of laboratory results - WEYCO, 5/88.

Station	Date	Time	Sampler	Laboratory	BOD_5 (mg/L)	TSS (mg/L)	Oil+ Grease (mg/L)	Alkalinity (mg/L)	Acidity (mg/L as $CaCO_3$)	Rainbow Trout Bioassay (65 % effluent)
Outfall-001	5/24	Comp	Ecology	Ecology	87	140		0	300	100 % survival
		(0845-1045)		WEYCO	104	152				
		(2130-2430)								
			WEYCO	Ecology	84	140		0	290	
				WEYCO	95	132		0	172	100 % survival
		0935	Ecology	Ecology			1			
			WEYCO	WEYCO			3			
		2300	Ecology	Ecology			10			
			WEYCO	WEYCO			4			
Outfall-002	5/24	1055								
		1541								
	5/25	Comp	Ecology	Ecology	<5	5				
		(0600-0600)		WEYCO	3	4				

Table 15. Comparison of effluent priority pollutant results - WEYCO, 5/88.

Compound	Pond D Effluent	
	Ecology (ug/L)	WEYCO (ug/L)
Total phenolics	**10**	**0.27**
Volatile organics:		
Methylene Chloride	2 U	**20 J**
Acetone	42 U	**200 B**
2-Butanone	14 U	**14 BJ**
Chloroform	**15**	**10 J**
Toluene	**2 BJ**	**2 BJ**
2-Hexanone	**0.6 J**	100 U
Semivolatiles:		
Benzo(a)Anthracene	**4**	1 U
Acenaphthylene	**0.3 J**	1 U
Acenaphthene	**2**	NA
Phenanthrene	**11**	**2**
Fluorene	**2**	**2**
Naphthalene	**1 J**	**1 U**
2-Methylnaphthalene	**0.7 J**	NA
Benzo(k)Fluoranthene	**3**	NA
Anthracene	**1 J**	1 U
Pyrene	**11**	1 U
Fluoranthene	**16**	**2**
Chrysene	**11**	1 U
4-Methylphenol	**37**	NA
Phenol	**0.8 J**	**3**
Diethylphthalate	**0.6 J**	1 U
Di-n-Butylphthalate	1 U	**1**
bis(2-Ethylhexyl)Phthalate	1 U	**2 B**
Dibenzofuran	**1 J**	NA
2,4,6-Trichlorophenol	**5**	**11**
2,4-Dimethylphenol	1 U	**3**
Benzoic Acid	**7**	NA
2,4-Dichlorophenol	**0.6 J**	
Pesticides/PCBs:	ND	ND

ND - None detected
NA - Not analyzed
U - Not detected at detection limit shown
J - Estimated concentration
B - Also detected in method blank

RECOMMENDATIONS AND CONCLUSIONS

1. The mill was meeting all NPDES permit limits during the inspection. BOD, TSS, and fecal coliform were all well below daily maximum and monthly average permit limits. A weir should be constructed at outfall-002 so flow can be measured continuously. In addition, some independent capability of verifying flow at outfall-001 is needed.

2. Effluent priority pollutant and resin acid analyses showed no organic pollutants above acute criteria.

3. PAH and 4 methylphenol concentrations were two to ten times higher around the outfall than at Cow Point, an upstream reference site. Nickel and lead were mildly elevated around the outfall. Resin acid and dioxin results suggest sediments off Cow Point may not have been far enough away from the influence of the ITT Rayonier and Weyerhaeuser discharges to serve as a true control.

4. Sediment samples showed no toxicity to the amphipod *Rhepoxynius abronius*. Some acute effluent toxicity was observed in mysid shrimp and Daphnia bioassays. Flouranthene in the effluent could account for the mysid mortality. The effluent was not mutagenic as measured by the Ames test.

5. The Pacific oyster bioassay indicated a high level of chronic effluent toxicity. Future NPDES permit requirements will include effluent limits for chronic toxicity. The Pacific oyster should be used as one of the test species. Unannounced sampling by Ecology has begun to characterize the variability of the biological responses and chemical composition of the effluent.

6. Sample splits for permit parameters agreed closely, except acidity. Laboratory procedures at the mill were in conformance with standard techniques. Minor recommendations are included in the laboratory review section of this report.

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APPENDIX I

Effluent and sediment bioassay methods - WEYCO, 5/88.

Test Organism	Matrix	Method Ref.	Laboratory	Test Duration	Effluent Concentration	Type of Test	Endpoint Measured
Rhepoxynius abronius	Sediment	1	E.V.S. Consultants Seattle, WA	10 days	N/A	Acute and Chronic	Survival and avoidance; % reburial after 10 days
Pacific Oyster (*Crassostrea gigas*)	Effluent	2	E.V.S. Consultants Seattle, WA	48 hrs	0.1,1,2.2,4.6, 10,18%	Chronic	Development of abnormal larvae
Mysid Shrimp (*Mysidopsis bahia*)	Effluent	3	E.V.S. Consultants Seattle, WA	96 hrs	1,3,10,30,100%	Acute	Survival
Daphnia pulex	Effluent	3	Ecology	48 hrs	100%	Acute	Survival
Rainbow Trout (*Salmo gairdneri*)	Effluent	4	Ecology	96 hrs	65%	Acute	Survival
Ames Test	Effluent	5	SRI International Menlo Park, CA	48 hrs	50,100,200, 300,400,500 uL per plate	Mutagenic Activity	Genetic damage to *Salmonella typhimurium* bacteria strains TA1535, TA1537, TA1538, TA98 & TA100

1 - Swartz *et al*. (1985) as amended by Chapman and Becker (1986)
2 - ASTM Method E 724-80, "Standard Practice for Conducting Static Acute Tests with Larvae of Four Species of Bivalve Molluscs."
3 - EPA/600/4-85/013, "Methods for Measuring the Acute Toxicity of Effluents of Freshwater and Marine Organisms."
4 - Department of Ecology procedure "Static Acute Fish Toxicity Test," July 1981 revision. DOE 80-12.
5 - Maron and Ames (1983)

Chemical analytical methods - WEYCO, 5/88.

Analyses	Method Used	Laboratory
TOC (water)	EPA, 1983: #415	Ecology; Manchester, WA
TOC (solids)	APHA, 1985: #505	Laucks Testing Labs; Seattle, WA
TOX (water)	EPA, 1986: #9020	Sound Analytical Services, Inc., Tacoma, WA
% Solids	APHA, 1985: #209F	Laucks Testing Labs; Seattle, WA
Grain Size	Tetra Tech, 1986	Laucks Testing Labs; Seattle, WA
Cyanide (water)	EPA, 1983: #335.2-1	Ecology; Manchester, WA
Total Phenolics	EPA, 1983: #420.2	Ecology; Manchester, WA
Volatiles (water)	EPA, 1984: #624	Ecology; Manchester, WA
Volatiles (solids)	EPA, 1986: #8240	Ecology; Manchester, WA
Semivolatiles (water)	EPA, 1984: #625	Ecology; Manchester, WA
Semivolatiles (solids)	EPA, 1986: #8270	Ecology; Manchester, WA
Pest/PCB (water)	EPA, 1984: #608	Ecology; Manchester, WA
Pest/PCB (solids)	EPA, 1986: #8080	Ecology; Manchester, WA
Metals (water)	EPA, 1983: #200 series	Ecology; Manchester, WA
Metals (solids)	EPA, 1983: #200 series	Ecology; Manchester, WA
Resin acids (water + solids)	NCASI, 1986	Ecology; Manchester, WA
Dioxin (solids)	EPA, 1986: #8280	Enseco Incorportated; West Sacramento, CA

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Results of Resin acid/Guaiacol scan - WEYCO, 5/88.

		Sediments (ug/kg dry wt.)				
		At Cow Point (Control Station)	At Outfall			Below Outfall
Lab ID:	Effluent (ug/L) #228085	#228132	Dupl. 1 #228133	Dupl. 2 #228135	Replicate #228134	#228136
Guaiacol	0.6 U	100 U	76 U	77 U	91 U	100 U
a-Terpeneol	0.6 U	R	R	R	R	R
4-Chloroguaiacol	0.6 U	100 U	76 U	77 U	91 U	100 U
4-Allylguaiacol (eugenol)	0.6 U	100 U	76 U	77 U	91 U	100 U
4,5-Dichloroguaicol	0.6 U	100 U	76 U	77 U	91 U	100 U
4-Propenylguaiacol	0.6 U	R	R	R	R	R
4,5,6-Trichloroguaicol	0.6 U	100 U	76 U	77 U	91 U	100 U
Tetrachloroguaicol	3	100 U	76 U	77 U	91 U	100 U
Trichlorosyringol	13	100 U	76 U	77 U	91 U	100 U
Linoleic acid	0.6 U	630 J	76 U	170 J	440 J	540 J
Oleic acid	5 B	1500	1400 J	510 J	740 J	800 J
Linolenic acid	0.6 U	NA	R	R	R	R
Sandaracopimaric acid	0.6 U	100 U	76 U	77 U	91 U	100 U
Isopimaric acid	0.6 U	100 U	76 U	77 U	91 U	100 U
Palustric acid	0.6 U	R	R	R	R	R
Levopimaric acid	0.6 U	R	R	R	R	R
Dehydroabietic acid	0.6 BU	250 BJ	140 B	170 JB	260 JB	400 JB
Abietic acid	0.6 U	100 U	76 U	29 J	66 J	130 J
Neoabietic acid	0.6 U	R	R	R	R	R
9,10-Dichlorosteric Acid	0.6 U	100 U	76 U	77 U	91 U	100 U
Dichlorodehydroabeitic acid	4	100 U	76 U	77 U	91 U	100 U

NA - Not analyzed
R - No analyses result, compound may or may not be present

Results of VOA priority pollutant scan - WEYCO, 5/88.

		Sediments (ug/kg dry wt.)				
		At Cow Point (Control Station)	At Outfall			Below Outfall
Lab ID:	Effluent (ug/L) #228085	#228132	Dupl. 1 #228133	Dupl. 2 #228135	Repl. #228134	#228136
Carbon Tetrachloride	5 U	8 U	6 U	6 U	8 U	9 U
Acetone	42 U	4 U	11 U	8 U	12 U	8 U
Chloroform	15	8 U	6 U	6 U	8 U	9 U
Benzene	0.8 U	8 U	6 U	6 U	8 U	9 U
1,1,1-Trichloroethane	5 U	8 U	6 U	6 U	8 U	9 U
Bromomethane	10 U	16 U	12 U	13 U	16 U	17 U
Chloromethane	10 U	16 U	12 U	13 U	16 U	17 U
Dibromomethane	5 U	8 U	6 U	6 U	8 U	9 U
Chloroethane	10 U	16 U	12 U	13 U	16 U	17 U
Vinyl Chloride	10 U	16 U	12 U	13 U	16 U	17 U
Methylene Chloride	2 U	77 B	4 U	4 U	6 U	7 U
Carbon Disulfide	0.2 U	8 U	6 U	6 U	8 U	2 U
Bromoform	5 U	8 U	6 U	6 U	8 U	9 U
Bromodichloromethane	5 U	8 U	6 U	6 U	8 U	9 U
1,1-Dichloroethane	5 U	8 U	6 U	6 U	8 U	9 U
1,1-Dichloroethene	5 U	8 U	6 U	6 U	8 U	9 U
Trichlorofluoromethane	5 U	8 U	6 U	6 U	8 U	9 U
Dichlorodifluoromethane	10 U	16 U	12 U	13 U	16 U	17 U
1,2-Dichloropropane	5 U	8 U	6 U	6 U	8 U	9 U
2-Butanone	14 U	0.7 U	2 U	13 U	3 U	2 U
1,1,2-Trichloroethane	5 U	8 U	6 U	6 U	8 U	9 U
Trichloroethene	5 U	8 U	6 U	6 U	8 U	9 U
1,1,2,2-Tetrachloroethane	5 U	8 U	6 U	6 U	8 U	9 U
1,2,3-Trichlorobenzene	5 U	8 U	6 U	6 U	8 U	9 U
Hexachlorobutadiene	5 U	8 U	6 U	6 U	8 U	9 U
Naphthalene	5 U	8 U	6 U	6 U	8 U	9 U
Total Xylenes	5 U	8 U	6 U	6 U	8 U	9 U
2-chlorotoluene	5 U	8 U	6 U	6 U	8 U	9 U
1,2-Dichlorobenzene	5 U	8 U	6 U	6 U	8 U	9 U
1,2,4-Trimethylbenzene	5 U	8 U	6 U	6 U	8 U	9 U
DBCP	5 U	8 U	6 U	6 U	8 U	9 U
1,2,3-Trichloropropane	5 U	8 U	6 U	6 U	8 U	9 U
Tert-Butylbenzene	5 U	8 U	6 U	6 U	8 U	9 U
Isopropylbenzene	5 U	8 U	6 U	6 U	8 U	9 U
p-Isopropyltoluene	5 U	8 U	6 U	6 U	8 U	9 U
Ethylbenzene	5 U	8 U	6 U	6 U	8 U	9 U
Styrene	5 U	8 U	6 U	6 U	8 U	9 U
Propylbenzene	5 U	8 U	6 U	6 U	8 U	9 U
Butylbenzene	5 U	8 U	6 U	6 U	8 U	9 U
4-Chlorotoluene	5 U	8 U	6 U	6 U	8 U	9 U
1,4-Dichlorobenzene	5 U	8 U	6 U	6 U	8 U	9 U
1,2-Dibromoethane	10 U	16 U	12 U	13 U	16 U	17 U
1,2-Dichloroethane	5 U	8 U	6 U	6 U	8 U	9 U
Vinyl Acetate	10 U	16 U	12 U	13 U	16 U	17 U
4-Methyl-2-Pentanone	10 U	16 U	12 U	13 U	16 U	17 U
1,3,5-Trimethylbenzene	5 U	8 U	6 U	6 U	8 U	9 U
Bromobenzene	5 U	8 U	6 U	6 U	8 U	9 U
Toluene	2 BJ	8 U	6 U	0.1 U	0.2 U	0.3 U
Chlorobenzene	5 U	8 U	6 U	6 U	8 U	9 U
1,2,4-Trichlorobenzene	5 U	8 U	6 U	6 U	8 U	9 U
Dibromochloromethane	5 U	8 U	6 U	6 U	8 U	9 U
Tetrachloroethene	5 U	8 U	6 U	6 U	8 U	9 U
Sec-Butylbenzene	5 U	8 U	6 U	6 U	8 U	9 U
1,3-Dichloropropane	5 U	8 U	6 U	6 U	8 U	9 U
Cis-1,2-Dichloroethene	5 U	8 U	6 U	6 U	8 U	9 U
Trans-1,2-Dichloroethene	5 U	8 U	6 U	6 U	8 U	9 U
1,3-Dichlorobenzene	5 U	8 U	6 U	6 U	8 U	9 U
1,1-Dichloropropene	5 U	8 U	6 U	6 U	8 U	9 U
2,2-Dichloropropane	5 U	8 U	6 U	6 U	8 U	9 U
2-Hexanone	0.6 J	16 U	12 U	13 U	16 U	17 U
1,1,1,2-Tetrachloroethane	5 U	8 U	6 U	6 U	8 U	9 U
cis-1,3-Dichloropropene	5 U	8 U	6 U	6 U	8 U	9 U
Trans-1,3-Dichloropropene	5 U	8 U	6 U	6 U	8 U	9 U

Results of BNA priority pollutant scan - WEYCO, 5/88.

		Sediments (ug/kg dry wt.)				
		At Cow Point (Control Station)	At Outfall			
Lab ID:	Effluent (ug/L) #228085	#228132	Dupl. 1 #228133	Dupl. 2 #228135	Repl. #228134	Below Outfall #228136
Benzo(a)Pyrene	1 U	100 U	76 U	77 U	91 U	100 U
2,4-Dinitrophenol	5 U	480 U	370 U	370 U	440 U	480 U
Dibenz(a,h)Anthracene	1 U	100 U	76 U	77 U	91 U	100 U
Benzo(a)Anthracene	4	16 J	19 J	23 J	91 J	23 J
4-Chloro-3-Methylphenol	1 U	100 U	76 U	77 U	91 U	100 U
Benzoic Acid	7	480 U	370 U	370 U	440 U	480 U
Hexachloroethane	1 U	100 U	76 U	77 U	91 U	100 U
Hexachlorocyclopentadiene	2 U	200 U	150 U	150 U	180 U	200 U
Isophorone	1 U	100 U	76 U	77 U	91 U	100 U
Acenaphthene	2	100 U	31 J	30 J	71 J	24 J
Diethylphthalate	0.6 J	100 BU	76 BU	77 U	91 BU	100 BU
Di-n-Butylphthalate	1 U	29 BJ	25 BJ	33 BJ	91 BU	35 BJ
Phenanthrene	11	51 J	110	120	530	120
Butylbenzylphthalate	1 BU	100 BU	76 BU	77 BU	91 BU	100 BU
N-Nitrosodiphenylamine	1 U	100 U	76 U	77 U	91 U	12 J
Fluorene	2	10 J	23 J	22 J	79 J	27 J
Carbazol	1 U	100 U	76 U	77 U	91 U	100 U
Hexachlorobutadiene	1 U	100 U	76 U	77 U	91 U	100 U
Pentachlorophenol	5 U	480 U	370 U	370 U	440 U	480 U
2,4,6-Trichlorophenol	5	100 U	76 U	77 U	91 U	100 U
2-Nitroaniline	5 U	480 U	370 U	370 U	440 U	480 U
2-Nitrophenol	1 U	100 U	76 U	77 U	91 U	100 U
1-Methylnaphthalene	1 U	100 U	20 J	77 U	91 U	100 U
Naphthalene	1 J	34 BJ	45 BJ	44 BJ	110 B	87 BJ
2-Methylnaphthalene	0.7 J	9 J	11 J	11 J	13 J	19 J
2-Chloronaphthalene	1 U	100 U	76 U	77 U	91 U	100 U
3,3'-Dichlorobenzidine	1 U	100 U	76 U	77 U	91 U	100 U
2-Methylphenol	1 U	100 U	76 U	77 U	91 U	100 U
1,2-Dichlorobenzene	1 U	100 U	76 U	77 U	91 U	100 U
o-Chlorophenol	NA	100 U	76 U	77 U	91 U	100 U
2,4,5-Trichlorophenol	5 U	480 U	370 U	370 U	440 U	480 U
Nitrobenzene	1 U	100 U	76 U	77 U	91 U	100 U
3-Nitroaniline	5 U	480 U	370 U	370 U	440 U	480 U
4-Nitroaniline	5 U	480 U	370 U	370 U	440 U	480 U
4-Nitrophenol	NA	480 U	370 U	370 U	440 U	480 U
Benzyl Alcohol	1 U	100 U	76 U	77 U	91 U	100 U
4-Bromophenyl-phenylether	1 U	100 U	76 U	77 U	91 U	100 U
2,4-Dimethylphenol	1 U	100 U	76 U	77 U	91 U	100 U
4-Methylphenol	37	51 J	46 J	77 U	130	520
1,4-Dichlorobenzene	1 U	100 U	76 U	77 U	91 U	100 U
4-Chloroaniline	1 U	100 U	76 U	77 U	91 U	100 U
Phenol	0.8 J	16 BJ	76 U	77 U	91 U	24 BJ
bis(2-Chloroethyl)Ether	1 U	100 U	76 U	77 U	91 U	100 U
bis(2-Chloroethoxy)Methane	1 U	100 U	76 U	77 U	91 U	100 U
bis(2-Ethylhexyl)Phthalate	1 U	97 BJ	320 B	270 B	82 BJ	46 BJ
Di-n-Octylphthalate	1 U	15 BJ	76 BU	46 BJ	91 BU	100 BU
Hexachlorobenzene	1 U	100 U	76 U	77 U	91 U	100 U
Anthracene	1 J	100 U	76 U	77 U	53 J	15 J
1,2,4-Trichlorobenzene	1 U	100 U	76 U	77 U	91 U	100 U
2,4-Dichlorophenol	0.6 J	100 U	76 U	77 U	91 U	100 U
2,4-Dinitrotoluene	1 U	100 U	76 U	77 U	91 U	100 U
Pyrene	11	76 J	110	120	640	120
Dimethylphthalate	1 U	100 U	76 U	77 U	91 U	100 U
Dibenzofuran	1 J	100 U	19 J	19 J	31 J	21 J
Benzo(ghi)Perylene	1 U	100 U	76 U	77 U	91 U	100 U
Indeno(1,2,3-cd)Pyrene	1 U	100 U	76 U	77 U	91 U	100 U
Benzo(b)Fluoranthene	1 U	100 U	76 U	20 J	39 J	100 U
Fluoranthene	16	57 J	95	100	840	88 J
Benzo(k)Fluoranthene	3	100 U	76 U	77 U	91 U	100 U
Acenaphthylene	0.3 J	7 J	4 J	4 J	8 J	13 J
Chrysene	11	100 U	76 U	77 U	57 J	18 U
Retene	1 U	110	76 U	64 J	120	150
4,6-Dinitro-2-Methylphenol	5 U	480 U	370 U	370 U	440 U	480 U
1,3-Dichlorobenzene	1 U	100 U	76 U	77 U	91 U	100 U
2,6-Dinitrotoluene	1 U	100 U	76 U	77 U	91 U	100 U
N-Nitroso-Di-n-Propylamine	1 U	100 U	76 U	77 U	91 U	100 U
4-Chlorophenyl-phenylether	1 U	100 U	76 U	77 U	91 U	100 U
2-Methylphenol	1 U	100 U	76 U	77 U	91 U	100 U
bis(2-chloroisopropyl)ether	1 U	100 U	76 U	77 U	91 U	100 U
2-Chlorophenol	1 U	NA	NA	NA	NA	NA

Results Pesticide/PCB priority pollutant scans - WEYCO, 5/88.

	Effluent (ug/L)	Sediments (ug/kg dry wt.) At Cow Point (Control Station)	At Outfall Dupl. 1	Dupl. 2	Repl.	Below Outfall
Lab ID:	#228085	#228132	#228133	#228135	#228134	#228136
4,4'-DDT	0.017 U	1 U	1 U	1 U	1 U	1 U
Chlordane	0.03 U	1 U	1 U	1 U	1 U	1 U
Gamma-BHC (Lindane)	0.017 U	1 U	1 U	1 U	1 U	1 U
Dieldrin	0.017 U	1 U	1 U	1 U	1 U	1 U
Endrin	0.017 U	1 U	1 U	1 U	1 U	1 U
Methoxychlor	0.03 U	--	--	--	--	--
4,4'-DDD	0.017 U	1 U	1 U	1 U	1 U	1 U
4,4'-DDE	0.017 U	1 U	1 U	1 U	1 U	1 U
Heptachlor	0.017 U	1 U	1 U	1 U	1 U	1 U
Aldrin	0.017 U	1 U	1 U	1 U	1 U	1 U
Alpha-BHC	0.017 U	1 U	1 U	1 U	1 U	1 U
Beta-BHC	0.017 U	1 U	1 U	1 U	1 U	1 U
Delta-BHC	0.017 U	1 U	1 U	1 U	1 U	1 U
Heptachlor Epoxide	0.017 U	1 U	1 U	1 U	1 U	1 U
Endosulfan Sulfate	0.017 U	1 U	1 U	1 U	1 U	1 U
Endrin Aldehyde	0.017 U	1 U	1 U	1 U	1 U	1 U
Toxaphene	0.5 U	30 U	30 U	30 U	30 U	30 U
Aroclor-1260	0.17 U	10 U	10 U	10 U	10 U	10 U
Aroclor-1254	0.17 U	10 U	10 U	10 U	10 U	10 U
Aroclor-1221	0.17 U	10 U	10 U	10 U	10 U	10 U
Aroclor-1232	0.17 U	10 U	10 U	10 U	10 U	10 U
Aroclor-1248	0.17 U	10 U	10 U	10 U	10 U	10 U
Aroclor-1016	0.17 U	10 U	10 U	10 U	10 U	10 U
beta-Endosulfan	0.017 U	1 U	1 U	1 U	1 U	1 U
Aroclor-1242	0.17 U	10 U	10 U	10 U	10 U	10 U
Misc Pesticides/Herbicides						
Aldicarb	1 U	230 U	170 U	180 U	200 U	230 U
Simazine	0.4 U	120 U	86 U	90 U	100 U	120 U
Diuron	0.4 U	120 U	86 U	90 U	100 U	120 U
Atrazine	0.2 U	45 U	34 U	34 U	40 U	46 U
Butylate	0.2 U	45 U	34 U	34 U	40 U	46 U
Metribuzin	0.2 U	45 U	34 U	34 U	40 U	46 U
Fenamiphos	0.1 U	23 U	17 U	17 U	20 U	23 U
Pronamide	0.4 U	120 U	86 U	90 U	100 U	120 U
Hexazinone	0.1 U	23 U	17 U	17 U	20 U	23 U
Organo Phosphorus Pesticides						
Fenthion	0.04 U					
Parathion	0.04 U					
Coumaphos	0.04 U					
Dimethoate	0.04 U					
Dichlorvos (DDVP)	0.04 U					
Dioxathion	0.04 U					
DEF	0.04 U					
Azinphos	0.04 U					
Malathion	0.04 U					
Folex	0.04 U					
Methyl Parathion	0.04 U					
Phorate	0.04 U					
Disulfoton (Di-Syston)	0.04 U					
Ronnel	0.04 U					
Diazinon	0.04 U					
Ethion	0.04 U					
Imidan	0.04 U					
Carbophenothion	0.04 U					
EPN	0.04 U					
Phencapton	0.04 U					
Ethyl Azinphos (Ethyl + Methyl)	0.04 U					
Monocrotophos	0.04 U					
Mevinphos	0.04 U					

APPENDIX II

Laboratory Procedure Review Sheet

Discharger: WEYCO, Cosmopolis

Date: 5/24/88

Discharger representative: Steve Avery

Ecology reviewer: Pat Hallinan, Carlos Ruiz

Instructions

Questionnaire for use reviewing laboratory procedures. Circled numbers indicate work is needed in that area to bring procedures into compliance with approved techniques. References are sited to help give guidance for making improvements. References sited include:

Ecology = Department of Ecology Laboratory User's Manual, December 8, 1986.

SM = APHA-AWWA-WPCF, Standard Methods for the Examination of Water and Wastewater, 16th ed., 1985.

SSM = WPCF, Simplified Laboratory Procedures for Wastewater Examination, 3rd ed., 1985.

Sample Collection Review

1. Are grab, hand composite, or automatic composite samples collected for influent and effluent BOD and TSS analysis?

2. If automatic compositor, what type of compositor is used? Isco
   The compositor should have pre and post purge cycles unless it is a flow through type. Check if you are unfamiliar with the type being used.

3. Are composite samples collected based on time or flow?

4. What is the usual day(s) of sample collection? everyday

5. What time does sample collection usually begin? tide cycle
   6am-6am - cooling water

6. How long does sample collection last?
   2-3 hrs at Pond D / 24 hrs cooling water

7. How often are subsamples that make up the composite collected? —

8. What volume is each subsample? unkn

9. What is the final volume of sample collected? ~3 gallons

10. Is the composite cooled during collection? yes

11. To what temperature? ✓
The sample should be maintained at approximately 4 degrees C (SM p41, #5b: SSM p2).

12. How is the sample cooled?
Mechanical refrigeration or ice are acceptable. Blue ice or similar products are often inadequate.

13. How often is the temperature measured? *unknown*
The temperature should be checked at least monthly to assure adequate cooling.

14. Are the sampling locations representative? ✓

15. Are any return lines located upstream of the influent sampling location?
This should be avoided whenever possible.

16. How is the sample mixed prior to withdrawal of a subsample for analysis? ✓
The sample should be thoroughly mixed.

17. How is the subsample stored prior to analysis? *N/A*
The sample should be refrigerated (4 degrees C) until about 1 hour before analysis, at which time it is allowed to warm to room temperature.

18. What is the cleaning frequency of the collection jugs? *monthly*
The jugs should be thoroughly rinsed after each sample is complete and occasionally be washed with a non-phospate detergent.

19. How often are the sampler lines cleaned? *unknown*
Rinsing lines with a chlorine solution every three months or more often where necessary is suggested.

pH Test Review

1. How is the pH measured? ✓
A meter should be used. Use of paper or a colorimetric test is inadequate and those procedures are not listed in Standard Methods (SM p429).

2. How often is the meter calibrated? ✓
The meter should be calibrated every day it is used.

3. What buffers are used for calibration? *4, 7*
Two buffers bracketing the pH of the sample being tested should be used.

If the meter can only be calibrated with one buffer, the buffer closest in pH to the sample should be used. A second buffer, which brackets the pH of the sample should be used as a check. If the meter cannot accurately determine the pH of the second buffer, the meter should be repaired.

BOD Test Review

1. What reference is used for the BOD test? *15th ed standard Methods + WEYCO guidelines*
Standard Methods or the Ecology handout should be used.

2. How often are BODs run? *Daily*
The minimum frequency is specified in the permit.

3. How long after sample collection is the test begun? *4-6 hrs*
The test should begin within 24 hours of composite sample completion (Ecology Lab Users Manual p42). Starting the test as soon after samples are complete is desirable.

4. Is distilled or deionized water used for preparing dilution water?

5. Is the distilled water made with a copper free still? *yes, glass*
Copper stills can leave a copper residual in the water which can be toxic to the test (SSM p36).

6. Are any nitrification inhibitors used in the test? *No* What?
2-chloro-6(trichloro methyl) pyridine or Hach Nitrification Inhibitor 2533 may be used only if carbonaceous BODs are being determined (SM p 527, #4g: SSM p 37).

7. Are the 4 nutrient buffers of powder pillows used to make dilution water? *No*
If the nutrients are used, how much buffer per liter of dilution water are added? ✓
1 mL per liter should be added (SM p527, #5a: SSM p37).

8. How often is the dilution water prepared? *2-5 days ahead of time*
Dilution water should be made for each set of BODs run.

9. Is the dilution water aged prior to use? *2-5 days*
Dilution water with nitrification inhibitor can be aged for a week before use (SM p528, #5b).
Dilution water without inhibitor should not be aged.

10. Have any of the samples been frozen? *sometimes*
If yes, are they seeded? *yes*
Samples that have been frozen should be seeded (SSM p38).

11. Is the pH of all samples between 6.5 and 7.5? *No*
If no, is the sample pH adjusted? *yes*
The sample pH should be adjusted to between 6.5 and 7.5 with 1N NaOH or 1N H2SO4 if 6.5 > pH >7.5 if caustic alkalinity or acidity is present (SM p529, #5e1: SSM p37).
High pH from lagoons is usually not caustic. Place the sample in the dark to warm up, then check the pH to see if adjustment is necessary.

If the sample pH is adjusted, is the sample seeded? *yes*
The sample should be seeded to assure adequate microbial activity if the pH is adjusted (SM p528, #5d).

12. Have any of the samples been chlorinated or ozonated? No

If chlorinated are they checked for chlorine residual and dechlorinated as necesssary?

How are they dechlorinated?

Samples should be dechlorinated with sodium sulfite (SM p529, #5e2: SSM p38), but dechlorination with sodium thiosulfate is common practice. Sodium thiosufate dechlorination is probably acceptable if the chlorine residual is < 1-2 mg/L.

If chlorinated or ozonated, is the sample seeded?

The sample should be seeded if it was disinfected (SM p528, #5d&5e2: SSM p38).

13. Do any samples have a toxic effect on the BOD test? No

Specific modifications are probably necessary (SM p528, #5d: SSM p37).

14. How are DO concentrations measured? YSI Meter

If with a meter, how is the meter calibrated? Air

Air calibration is adequate. Use of a barometer to determine saturation is desirable, although not manditory. Checks using the Winkler method of samples found to have a low DO are desirable to assure that the meter is accurate over the range of measurements being made.

How frequently is the meter calibrated? Daily

The meter should be calibrated before use.

15. Is a dilution water blank run? yes

A dilution water blank should always be run for quality assurance (SM p527, #5b: SSM p40, #3).

What is the usual initial DO of the blank? 8.9-9.1 mg/L

The DO should be near saturation; 7.8 mg/L @ 4000 ft, 9.0 mg/L @ sea level (SM p528, #5b). The distilled or deionized water used to make the dilution water may be aged in the dark at ~20 degrees C for a week with a cotton plug in the opening prior to use if low DO or excess blank depletion is a problem .

What is the usual 5 day blank depletion? 0.0-0.3

The depletion should be 0.2 mg/L or less. If the depletion is greater, the cause should be found (SM p527-8, #5b: SSM p41, #6).

16. How many dilutions are made for each sample? 2

At least two dilutions are recommended. The dilutions should be far enough apart to provide a good extended range (SM p530, #5f: SSM p41).

17. Are dilutions made by the liter method or in the bottle?

Either method is acceptable (SM p530, #5f).

18. How many bottles are made at each dilution? 2 per sample; 1 for cooling water

How many bottles are incubated at each dilution? same

When determining the DO using a meter only one bottle is necessary. The DO is measured, then the bottle is sealed and incubated (SM p530, #5f2).

When determining the DO using the Winkler method two bottles are necessary. The initial DO is found of one bottle and the other bottle is sealed and incubated (Ibid.).

19. Is the initial DO of each dilution measured? yes
What is the typical initial DO? ~9.0 mg/L
The initial DO of each dilution should be measured. It should approximate saturation (see #14).

20. What is considered the minimum acceptable DO depletion after 5 days? ✓
What is the minimum DO that should be remaining after 5 days? ✓
The depletion should be at least 2.0 mg/L and at least 1.0 mg/L should be left after 5 days (SM p531, #6: SSM p41).

21. Are any samples seeded? All
Which?
What is the seed source? Aberdeen STP effluent
Primary effluent or settled raw wastewater is the preferred seed. Secondary treated sources can be used for inhibited tests (SM p528, #5d: SSM p41).

How much seed is added to each sample? 8cc per liter
Adequate seed should be used to cause a BOD uptake of 0.6 to 1.0 mg/L due to seed in the sample (SM p529, #5d).

How is the BOD of the seed determined? ✓ yes
Dilutions should be set up to allow the BOD of the seed to be determined just as the BOD of a sample is determined. This is called the seed control (SM p529, #5d: SSM p41).

22. What is the incubator temperature? ✓
The incubator should be kept at 20 +/- 1 degree C (SM p531, #5i: SSM p40, #3).

How is incubator temperature monitored? ✓
A thermometer in a water bath should be kept in the incubator on the same shelf as the BODs are incubated.

How frequently is the temperature checked? 2x's a day
The temperature should be checked daily during the test. A temperature log on the incubator door is recommended.

How often must the incubator temperature be adjusted? weekly
Adjustment should be infrequent. If frequent adjustments (every 2 weeks or more often) are required the incubator should be repaired.

Is the incubator dark during the test period? ✓
Assure the switch that turns off the interior light is functioning.

23. Are water seals maintained on the bottles during incubation? ✓
Water seals should be maintained to prevent leakage of air during the incubation period (SM p531, #5i: SSM p40, #4).

24. Is the method of calculation correct?

Check to assure that no correction is made for any DO depletion in the blank and that the seed correction is made using seed control data.

Standard Method calculations are (SM p531, #6):

for unseeded samples;

$$\text{BOD (mg/L)} = \frac{D1 - D2}{P}$$

for seeded samples;

$$\text{BOD (mg/L)} = \frac{(D1 - D2) - (B1 - B2)f}{P}$$

Where:
- D1 = DO of the diluted sample before incubation (mg/L)
- D2 = DO of diluted sample after incubation period (mg/L)
- P = decimal volumetric fraction of sample used
- B1 = DO of seed control before incubation (mg/L)
- B2 = DO of seed control after incubation (mg/L)

$$f = \frac{\text{amount of seed in bottle D1 (mL)}}{\text{amount of seed in bottle B1 (mL)}}$$

Total Suspended Solids Test Review

Preparation

1. What reference is used for the TSS test? None used

2. What type of filter paper is used? ✓
Std. Mthds. approved papers are: Whatman 934AH (Reeve Angel), Gelman A/E, and Millipore AP-40 (SM p95,footnote: SSM p23)

3. What is the drying oven temperature? ✓
The temperature should be 103-105 degrees C (SM p96, #3a: SSM p23).

4. Are any volatile suspended solids tests run? No
If yes--What is the muffle furnance temperature?
The temperature should be 550+/- 50 degrees C (SM p98, #3: SSM p23).

5. What type of filtering apparatus is used?
Gooch crucibles or a membrane filter apparatus should be used (SM p95, #2b: SSM p23).

6. How are the filters pre-washed prior to use? ✓
The filters should be rinsed 3 times with distilled water (SM p23, #2: SSM p23, #2).

Are the rough or smooth sides of the filters up? ✓
The rough side should be up (SM p96, #3a: SSM p23, #1)

How long are the filters dried? ✓
The filters should be dried for at least one hour in the oven. An additional 20 minutes of drying in the furnance is required if volatile solids are to be tested (Ibid).

How are the filters stored prior to use? ✓
The filters should be stored in a dessicator (Ibid).

7. How is the effectiveness of the dessicant checked? ✓
All or a portion of the dessicant should have an indicator to assure effectiveness.

Test Procedure

8. In what is the test volume of sample measured? ✓
The sample should be measured with a wide tipped pipette or a graduated cylinder.

9. Is the filter seated with distilled water? ✓
The filter should be seated with distilled water prior to the test to avoid leakage along the filter sides (SM p97, #3c).

10. Is the entire measured volume always filtered? ✓
The entire volume should always be filtered to allow the measuring vessel to be properly rinsed (SM p97, #3c: SSM p24, #4).

11. What are the average and minimum volumes filtered?

	Volume	
	Minimum	Average
Influent		~100ml pond D ~300 ml cooling water
Effluent		

12. How long does it take to filter the samples?

	Time
Influent	
Effluent	~10 sec

13. How long is filtering attempted before deciding that a filter is clogged? ✓
Prolonged filtering can cause high results due to dissolved solids being caught in the filter (SM p96, #1b). We usually advise a five minute filtering maximum.

14. What do you do when a filter becomes clogged? ✓
The filter should be discarded and a smaller volume of sample should be used with a new filter.

15. How are the filter funnel and measuring device rinsed onto the filter following sample addition? ✓
Rinse 3x's with approximately 10 mLs of distilled water each time (? ?).

16. How long is the sample dried? ✓
The sample should be dried at least one hour for the TSS test and 20 minutes for the volatile test (SM p97, #3c; p98, #3: SSM p24, #4). Excessive drying times (such as overnight) should be avoided.

17. Is the filter thoroughly cooled in a dessicator prior to weighing? ✓
The filter must be cooled to avoid drafts due to thermal differences when weighing (SM p97, #3c: SSM p97 #3c).

18. How frequently is the drying cycle repeated to assure constant filter weight has ben reached (weight loss <0.5 mg or 4%, whichever is less: SM p97, #3c)?
We recommend that this be done at least once every 2 months.

19. Do calculations appear reasonable? ✓
Standard Methods calculation (SM p97, #3c).

$$\text{mg/L TSS} = \frac{(A - B) \times 1000}{\text{sample volume (mL)}}$$

where: A= weight of filter + dried residue (mg)
B= weight of filter (mg)

Fecal Coliform Test Review

1. Is the Membrane Filtration (MF) or Most Probable Number (MPN) technique used?
   This review is for the MF technique.

2. Are sterile techniques used? ✓

3. How is equipment sterilizated? Autoclave
   Items should be either purchased sterilized or be sterilized. Steam sterilization, 121 degrees C for 15 to 30 minutes (15 psi); dry heat, 1-2 hours at 170 degrees C; or ultraviolet light for 2-3 minutes can be used. See Standard Methods for instruclions for specific items (SSM p67-68).

4. How is sterilization preserved prior to item use? ✓
   Wrapping the items in kraft paper or foil before they are sterilized protects them from contamination (Ibid.).

5. How are the following items sterilized?

	Purchased Sterile	Sterilized at Plant
Collection bottles		✓
Phosphate buffer	✓	
Media	✓	
Media pads	✓	
Petri dishes	✓	
Filter apparatus	✓	
Filters	✓	
Pipettes		✓
Measuring cylinder		✓
Used petri dishes		✓

6. How are samples dechlorinated at the time of collection? N/A
   Sodium thiosulfate (1 mL of 1% solution per 120 mLs (4 ounces) of sample to be collected) should be added to the collection bottle prior to sterilization (SM p856, #2: SSM p68, sampling).

7. Is phosphate buffer made specifically for this test? yes
   Use phosphate buffer made specifically for this test. The phosphate buffer for the BOD test should not be used for the coliform test (SM p855, #12: SSM p66).

8. What kind of media is used? ✓
   M-FC media should be used (SM p896, SSM p66).

9. Is the media mixed or purchased in ampoules?
   Ampoules are less expensive and more convient for under 50 tests per day (SSM p65, bottom).

10. How is the media stored? ✓
    The media should be refrigerated (SM p897, #1a: SSM p66, #5).

11. How long is the media stored?
Mixed media ahould be stored no longer than 96 hours (SM p897, #1a: SSM p66, #5). Ampoules will usually keep from 3-6 months -- read ampoule directions for specific instructions.

12. Is the work bench disinfected before and after testing?
This is a necessary sanitazation procedure (SM p831, #1f).

13. Are forceps dipped in alcohol and flamed prior to use?
Dipping in alcohol and flaming are necessary to sterilize the forceps (SM p889, #1: SSM p73, #4).

14. Is sample bottle thoroughly shaken before the test volume is removed? The sample should be mixed thoroughly (SSM p73, #5).

15. Are special procedures followed when less than 20 mLs of sample is to be filtered?
10-30 mLs of sterile phosphate buffer should be put on the filter. The sample should be put into the buffer water and swirled, then the vacuum should be turned on. More even organism distribution is attained using this technique (SM p890, #5a: SSM P73, #5).

16. Are special procedures followed when less than 1 mL of sample is to be filtered?
Sample dilution is necessary prior to filtration when <1 mL is to be tested (SM p864, #2c: SSM p69).

17. Is the filter apparatus rinsed with phosphate buffer after sample filtration?
Three 20-30 mL rinses of the filter apparatus are recommended (SM p891, #5b: SSM p75, #7).

18. How soon after sample filtration is incubation begun?
Incubation should begin within 20-30 minutes (SM p897, #2d: SSM p77, #10 note).

19. What is the incubation temperature?
44.5 +/- 0.2 degrees C (SM p897, #2d: SSM p75, #9).

20. How long are the filters incubated?
24 +/- 2 hours (Ibid.).

21. How soon after incubation is complete are the plate counts made?
The counts should be made within 20 minutes after incubation is complete to avoid colony color fading (SSM p77, FC).

22. What color colonies are counted?
The fecal coliform colonies vary from light to dark blue (SM p897, #2e: SSM p78).

23. What magnification is used for counting?
10-15 power magnification is recommended (SM p898, #2e: SSM p78).

24. How many colonies blue colonies are usually counted on a plate?
    Valid plate counts are between 20 and 60 colonies (SM p897, #2a: SSM p78).

25. How many total colonies are usually on a plate?
    The plate should have <200 total colonies to avoid inhabition due to crowding (SM p893, #6a: SSM p63, top).

26. When calculating results, how are plates with <20 or >60 colonies considered when plates exist with between 20 and 60 colonies?
    In this case the plates with <20 or >60 colonies should not be used for calculations (SM p898, #3: SSM p78, C&R).

27. When calculating results how are results expressed if all plates have < 20 or > 60 colonies?
    Results should be identified as estimated.
    The exception is when water quality is good and <20 colonies grow. In this case the lower limit can be ignored (SM p893, #6a: SSM p78, C&R).

28. How are results calculated?
    Standard Methods procedure is (SM p893, #6a: SSM p79):

$$\text{Fecal coliforms/100 mL} = \frac{\text{\# of fecal coliform colonies counted}}{\text{sample size (mL)}} \times 100$$