

INLAND EMPIRE PAPER COMPANY  
CLASS II INSPECTION  
SEPTEMBER 1990

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by  
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## ABSTRACT

A Class II Inspection was conducted at the Inland Empire Paper Company (IEP) in Spokane, Washington, on September 10-13, 1990. The inspection was conducted in order to evaluate compliance with the NPDES permit limits. The effluent met NPDES permit requirements for BOD<sub>5</sub>, TSS, pH, rainbow trout bioassay, and discharge. Copper was found at 17.4 µg/L, slightly above the chronic water quality criteria. Mercury was found in the effluent at the level of 0.052 µg/L, which is above the chronic water quality criterion. Hexavalent chromium was detected in the effluent at 0.202 mg/L and 0.023 mg/L levels, which are also above acute and chronic water quality criteria. No effluent or receiving water toxicity was indicated by rainbow trout, *Daphnia pulex*, *Ceriodaphnia dubia*, Fathead minnow larvae, or Microtox®. No volatile organics, pesticides/PCBs, or BNAs were detected in sludge samples. However, a number of metals including chromium, copper, nickel, zinc, barium, and aluminum were detected. Resin/fatty acids and phenol were also detected in sludge. IEP and Ecology lab results from split samples for parameter analysis agreed well. Several remedial actions were recommended to address problems noted during the inspection.

## INTRODUCTION

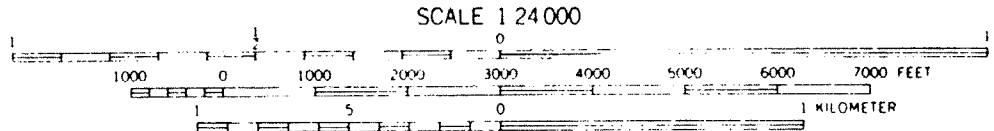
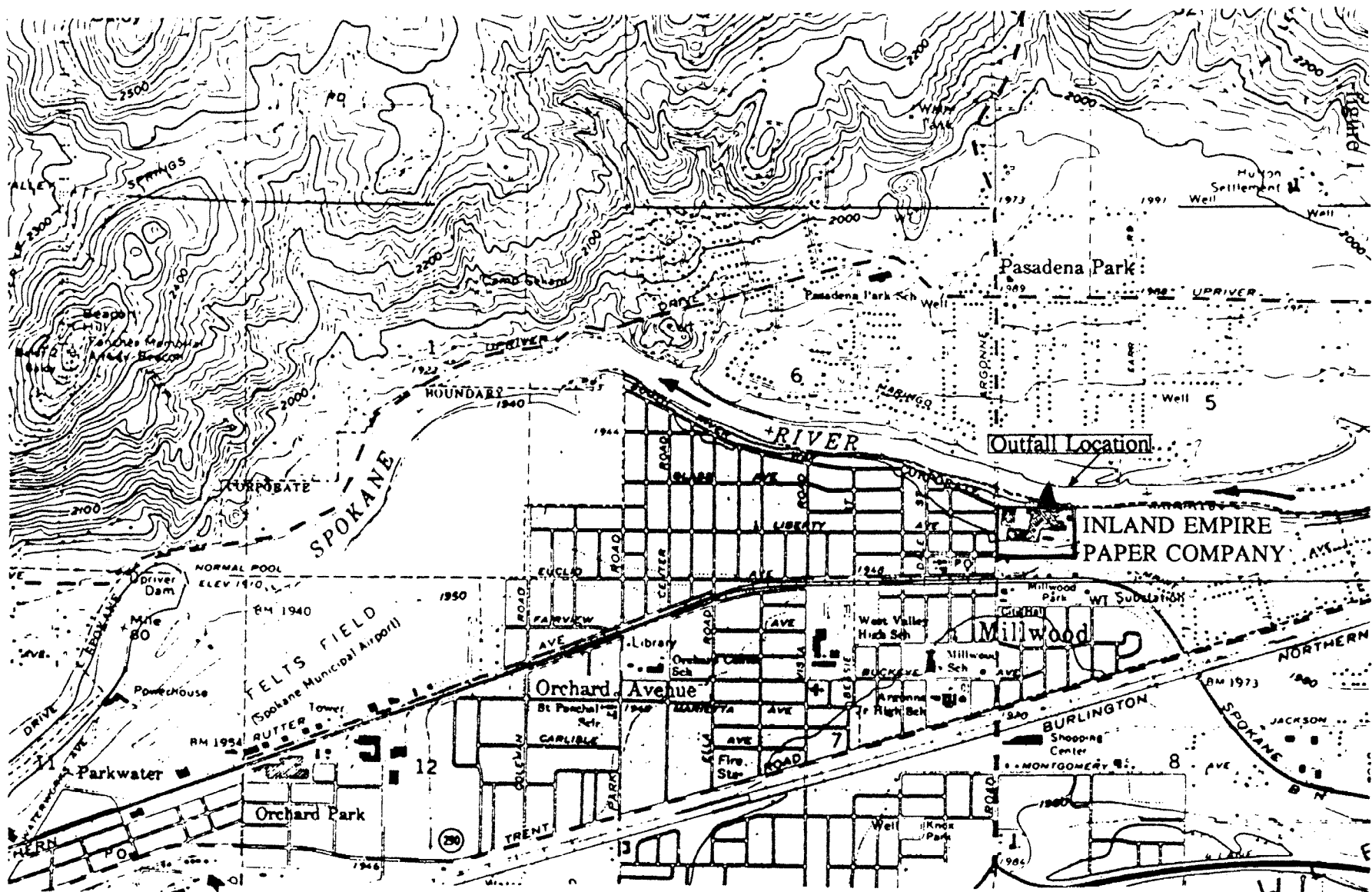
The Washington State Department of Ecology (Ecology) conducted a Class II Inspection at the Inland Empire Paper Company (IEP) near Spokane, Washington, on September 10-13, 1990. Lisa Zinner, Marc Heffner, and Keith Seiders from the Ecology Toxics, Compliance, and Ground Water Investigations Section conducted the inspection. Rick Fink, Inland Empire Paper Technical Supervisor, provided assistance during collection of water samples. The laboratory data were analyzed and the investigative report was written by Tapas Das of the Watershed Assessments Section of Environmental Investigations and Laboratory Services Program (EILS) and Lisa Zinner of the Northwest Regional Office (NWRO). The Enhanced Class II Inspection was performed in support of the reissuance of NPDES permit WA-000082-5, which expired on June 25, 1989.

Inland Empire Paper Company operates a groundwood pulp mill and newsprint paper mill in Millwood, Washington (Figure 1). Pulp is produced by the Groundwood CMN (coarse molded news) process and the Groundwood CMP (chemi-mechanical pulp) process from wood chips. Newsprint is produced from pulp manufactured at the mill. Total paper production is 293 tons per day. IEP will install pulping capacity for old newspapers (ONP) in order to make use of recycled fiber. The ONP Deinking Plant pulp would replace the CMN pulp in the paper making process. No change of paper production capacity would occur.

The wastewater treatment system at the IEP mill was constructed in 1989 (Figure 2). The system consists of the following: a mechanically cleaned coarse screen bar rack, a wastewater pump station, a primary clarifier, an Envirex Orbal aeration basin, and a secondary clarifier. The 2.1 million gallon aeration basin is divided into three stages, in the configuration of three concentric oval oxidation ditches. A Parshall flume measures the effluent from the secondary clarifier prior to discharge to the Spokane River. IEP's automatic composite sampler intake is located at the Parshall flume. Primary and secondary sludge are combined and thickened in a rotary drum thickener, then dewatered in a sludge press. At the time of inspection, dewatered sludge was being disposed of in a landfill on the IEP property.

A survey on the quality of the receiving water of the Spokane River was conducted at the same time; a separate report will be issued (Joy, in progress). The objectives of the inspection were:

1. Assess plant compliance with existing and proposed NPDES permit limits;
2. Analyze priority and non-priority pollutants in the wastewater treatment system influent and effluent;
3. Assess the plant's treatment efficiency;
4. Evaluate effluent toxicity using a suite of acute and chronic bioassays;
5. Analyze dewatered sludge for percent solids and priority pollutant metals;



CONTOUR INTERVAL 20 FEET  
 DOTTED LINES REPRESENT 10-FOOT CONTOURS  
 NATIONAL GEODETIC VERTICAL DATUM OF 1929

▲ Sampling Location



QUADRANGLE LOCATION

Figure 1 - Plant Site and Outfall Location - Inland Empire Paper, 9/90

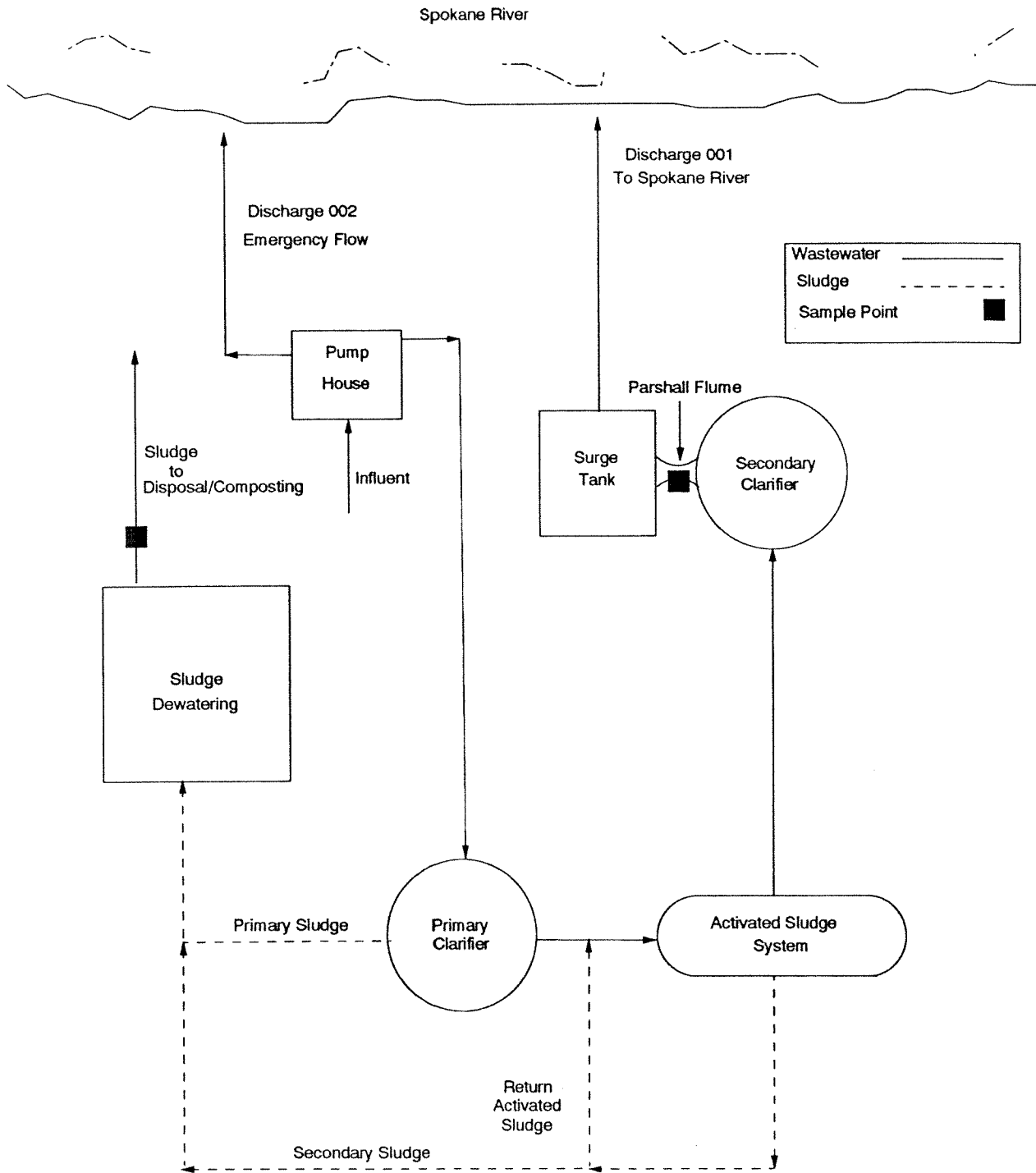


Figure 2 - Wastewater Treatment Plant Schematic and Sample Sites - Inland Empire Paper, 9/90

6. Review lab procedures at the mill to determine adherence to accepted protocols;
7. Split samples with the permittee to determine the comparability of laboratory results; and
8. Advance the state-of-the-art of compliance inspections by contributing to the ongoing developmental efforts with centrifugation.

## PROCEDURES

Ecology collected both 24-hour composite samples and grab samples of effluent wastewater at the entrance to the Parshall flume. ISCO automatic samplers collected an aliquot of wastewater every 30 minutes for 24 hours. The composite sampler was cleaned for priority pollutant sampling prior to the inspection (Table 1). IEP collected their daily effluent composite sample at the same site and time. No influent samples were collected during the inspection.

Hand composites, consisting of three grab samples of effluent, were collected for bioassays. Hand composites of the receiving water were also collected 50 feet upstream of the discharge for bioassays in order to characterize river water toxicity. The receiving water bioassay composite samples consisted of two grabs taken 300 feet downstream of the outfall in equal proportions at depths of 1, 3, and 6 meters.

Field transfer blanks were collected for both grab and composite samples (Table 1).

Hand composites, consisting of three grab samples, were collected of dewatered sludge. The sludge grab samples were taken from a pile underneath the dewatered sludge conveyor.

Effluent particulate matter was collected using two Alfa Laval bowl-type continuous centrifuges (model WSB/MAB 103) following procedures described by Andreasson (1991 in progress). A small peristaltic pump was used to pump effluent to the centrifuges. The centrifuges were cleaned prior to sampling following procedures described by Seiders (1989). The effluent dissolved fraction (centrate) was also sampled using an ISCO composite sampler.

Sampling times, parameters analyzed, and sample splits between Ecology and IEP are included in Table 2. Sampling sites are shown in Figure 2; Ecology and IEP sites were identical. All samples were held on ice until delivery to the Manchester Laboratory. A summary of the analytical methods and the laboratories conducting the analyses is given in Appendix A.

The effluent Parshall flume was checked for appropriate dimensions, installation, and maintenance. Ecology instantaneous flow measurements were made and compared to the IEP flowmeter.

Laboratory quality assurance and quality control (QA/QC) methods, which were followed during the analyses of general chemistry parameters and priority pollutants, are described by Huntameter and Hyre (1991). Recommended holding times were met for all analyses

Table 1. Priority Pollutant Cleaning and Field Transfer Blank Procedure - Inland Empire Paper, 9/90.

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Priority Pollutant Sampling Equipment Cleaning Procedure

1. Wash with laboratory detergent (phosphate free).
2. Rinse several times with tap water.
3. Rinse with 10% nitric acid solution.
4. Rinse three times with distilled/deionized water.
5. Rinse with high purity methylene chloride.
6. Rinse with high purity acetone.
7. Allow to dry and seal with aluminum foil.

Field Transfer Blank Procedure

1. Pour organic free water directly into appropriate bottles for parameters to be analyzed from grab samples (VOCs).
  2. Run approximately 1 liter of organic free water through a compositor and discard.
  3. Run approximately 6 liters of organic free water through the same compositor and pour the water into appropriate bottles for parameters to be analyzed from composite samples (BNAs, Pesticides/PCBs, and priority pollutant metals).
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Table 2. Sampling Times and Parameters Analyzed - Inland Empire Paper, 9/90.

Parameter	Station: Type: Date: Time: Sample ID#:	Effluent grab 9/11 0900 378083	Effluent grab 9/11 1600 378084	Eff-ECO composite 9/11-12 0700-0700 378085	Eff-IEP composite 9/11-12 0700-0700 378086	Blank transfer 9/10 1500 378087	Blank transfer 9/11 1020 378088	Sludge grab/comp 9/11-12 24 hr 378089	River grab/comp 9/11-12 24 hr 378090
<b>GENERAL CHEMISTRY</b>									
Turbidity		E	E	E	E				
Conductivity		E	E	E	E				
Alkalinity		E	E	E	E				
Hardness		E	E	E	E				
Color		E	E	E					
<b>SOLIDS</b>									
TS				E	E				
TNVS				E	E				
TSS		E	E	E/IEP	E/IEP				
TNVSS				E	E				
BOD5				E/IEP	E/IEP				
COD		E	E	E	E				
TOC				E				E	
<b>NUTRIENTS</b>									
NH3-N		E	E	E	E				
NO3+NO2-N		E	E	E	E				
Phosphorus - Total		E	E	E/IEP	E/IEP				
Phosphate - Ortho		E	E	E	E				
Fecal Coliform		E	E						
% Klebsiella		E	E						
E. Coli		E	E						
Enterococci		E	E						
% Solids								E	
% Volatile Solids								E	
<b>ORGANICS AND METALS</b>									
Phenol				E	E	E		E	
EOX		E	E				E	E	
Resin/Fatty Acids				E		E		E	
Chrome (VI)		E	E				E		
Aluminum				E		E		E	
<b>PRIORITY POLLUTANTS</b>									
BNA's				E		E		E	
Pesticides/PCBs				E		E		E	
VOC		E	E				E	E	
Metals				EE**		EE*	EE**	E*	
Cyanide				EE+	EE+	EE+		EE+	
EP Tox Metals								E***	
<b>BIOASSAYS</b>									
Rainbow trout (acute)				E					E
Microtox (acute)				E					E
Daphnia pulex (acute and chronic)				EE					EE
Ceriodaphnia (chronic)				E					E
Fathead minnow (chronic)				E					E
<b>FIELD OBSERVATIONS</b>									
Temp		E	E	E	E				
pH		E	E	E	E				
Conductivity		E	E	E	E				

E - Ecology analysis

EE - Ecology duplicate analyses

IEP - Inland Empire Paper analysis

+ (1) total and (1) weak and dissociable cyanide

\* total recoverable pp metals

\*\* (1) total recoverable and (1) dissolved pp metals

\*\*\* total pp metals



performed. Matrix spike, spike duplicate recoveries, and relative percent difference (RPD), a measure of precision, were acceptable within QC limits for both water and soil. There were no major analytical problems with the analysis of water and soil samples.

## RESULTS AND DISCUSSION

### Flow

Physical measurements taken of the 12" Parshall flume showed the effluent flowmeter was correctly installed and calibrated. Instantaneous flow measurements were recorded several times during the inspection by having one inspector take the depth measurement at the flume while another inspector recorded the plant flowmeter reading. Flow rates for given depths of flow were obtained from a table found in the ISCO Open Channel Flow Measurement Handbook (ISCO, 1985).

Flow measurement data are summarized in Table 3. The IEP effluent totalized flow for a 24-hour time period between 7:00 a.m. (9/10/90) and 7:00 a.m. (9/11/90) was 3.097 MGD; this flow rate was used to calculate the mass loading for permit parameters.

### General Chemistry and NPDES Permit Compliance

Conventional pollutant data collected during the inspection are tabulated in Table 4. The IEP's wastewater treatment plant performed well during the inspection. The conventional parameters of BOD<sub>5</sub> and TSS indicated a well-treated, high quality effluent.

The fecal coliform counts in the effluent streams were 310 #/100mL and 240 #/100mL, with Klebsiella reported to be 81% and 100%, respectively. High fecal coliform counts (due to presence of Klebsiella) are common in pulp mill effluent. These numbers are similar to results from previous Class II inspections at other pulp mills (Andreasson, 1991 and Hallinan and Ruiz, 1990).

Table 3. Instantaneous Flow Measurements at Effluent Flume - Inland Empire Paper, 9/90.

Size of Parshall flume = 12 inches.

Date	Time	Ecology Measurement		IEP Meter Reading (MGD)
		Depth (Ft)	Flow (MGD)	
9/11	11:20	1.25	3.63	3.68
9/11	16:45	1.13	3.11	3.35
9/12	08:46	1.10	2.99	3.02

Table 4. Results of General Chemistry – Inland Empire, 9/90.

Field Station: Date: Type: Time: Lab sample#: 3780-	#1	Blank	#2	Effluent		Eff-ECO	Eff-IEP	Sludge
	9/9		9/10	9/11	9/11	9/11-12	9/11-12	9/11-12
	Transfer		Transfer	grab	grab	comp	comp	grab/comp
	pm		pm	am	pm	24 hr	24 hr	
	87		88	83	84	85	86	89
<b>GENERAL CHEMISTRY</b>								
Turbidity (NTU)				34	34	32.5	29	
Conductivity (µmhos/cm)				510	590(600)*	580	600	
Alkalinity (mg/L CaCO3)				58	62	58	62	
Hardness (mg/L CaCO3)				65	58	100	60	
Color C.U.				250	400	280		
<b>SOLIDS (mg/L)</b>								
TS (mg/L)						530	520	
TNVS %						87	86	
TSS (mg/L)				23	26(24)*	27	24(26)*	
TNVSS %						66.7	57.1	
% Solids								17.5
% Volatile Solids								89.3
BOD5 (mg/L)						23	30	
COD (mg/L)				260	240	250	230	
TOC (water mg/L)						59.5(63.8)*		10.7^^
<b>NUTRIENTS (mg/L)</b>								
NH3-N (mg/L) as N				0.012	<0.005	<0.005	0.018(0.019)*	
NO2+NO3-N (mg/L) as N				0.113	0.108(0.114)*	<0.01	0.092(0.096)*	
Phosphate - Total (mg/L)				0.43(0.42)*	0.42	0.41(0.40)*	0.44	
Phosphate - Ortho+ (mg/L)				0.165	0.175	0.182	0.154	
Phosphate - Ortho++ (mg/L)				0.126	0.143(0.133)*	0.16	0.134(0.136)*	
F-Coliform #/100 mL				310	240			
E. Coli #/100 mL				6	<2.5			
% Klebsiella				81	100			
Enterococci #/100 mL				30	120			
Cyanide total (mg/L)						<0.005	<0.005	
Cyanide (wk & dis mg/L)						<0.005	<0.005	
<b>ORGANICS</b>								
EOX (mg/L)			10U	10U	10U			10U
Phenol (mg/L)	<0.005					0.042	0.047	0.492^
<b>PRIORITY POLLUTANTS</b>								
Hexavalent Chromium (mg/L)			<0.005	0.202	0.023			
Aluminum (total rec µg/L)	10 U					1010		3200^^^
<b>FIELD OBSERVATIONS</b>								
Temp °C				30.7	31.4	4.9+++	7.1+++	
pH S.U.				7.2	7	7.4	7.8	
Conductivity µmho/cm				571	625	563	548	

\* - Duplicate.

^ - µg/kg -dry.

^^ - % dry weight

^^^ - mg/kg - dry.

+ - Manual.

++ - Rapid Flow Analyzer.

+++ - Iced composite sample.

U - Indicates compound was analyzed for but not detected at the given detection limit.

A comparison of effluent parameters to NPDES permit limits is presented in Table 5. The effluent met permit limits for BOD<sub>5</sub>, TSS, and pH. The effluent passed the 96-hour rainbow trout bioassay. At a 65% effluent concentration, 100% survival was observed.

### **Effluent Priority Pollutant Scan**

A complete listing of effluent priority pollutant scan results is included in Appendix B.

No volatile organic compounds (VOCs) or Pesticides/PCBs were detected in the effluent. Among BNA's, a trace amount of benzoic acid was detected at 3.0 µg/L, flagged with a "J" qualifier, indicating an estimated value when result is less than specified detection limit.

A number of resin/fatty acids (RFAs) were detected at low levels in the WTP effluent. The results are given in Table 6. This is common in pulp mill effluent.

A listing of priority pollutant metals detected in blanks and effluent is presented in Table 7. Most metals detected were at a level much less than acute and chronic water quality criteria. Copper was found at the levels of 17.4 µg/L (by dissolved method) and 17 µg/L (by total recoverable method), slightly above acute and chronic water quality criteria (EPA, 1986). However, these results were qualified indicating that the analyte was found in the transfer blank as well as in the sample. Hexavalent chromium was detected in the effluent (Table 4) at 0.202 mg/L and 0.023 mg/L concentrations, which are above acute and chronic water quality criteria.

### **Effluent Bioassays**

Several bioassays were conducted to assess toxicity of effluent. For this inspection, rainbow trout (Ecology, 1981), *Daphnia pulex* (EPA, 1985), *Ceriodaphnia dubia* (EPA, 1989), Fathead minnow (EPA, 1989) and Microtox® (Beckman, 1982) were used as test organisms for both treated effluent and receiving water. The affect that contaminants have on the organisms is determined through observations of death, failure to reproduce, deformity, response, growth, etc., as specified in the referenced method for each test.

In several bioassays, two different laboratories were used to check on variability of results. This was made possible by the availability of EPA grant funds.

Effluent bioassay results are given in Table 8. No effluent toxicity was indicated by rainbow trout. A seven-day survival and reproduction test of *Daphnia pulex* resulted in 90% survival in 100% effluent. No Observed Effects Concentration (NOEC) and Lowest Observed Effects Concentration (LOEC) for the test were both 100%. The average number of young reproduced per female was 29 at 100% effluent, compared to 24 in control water..

Microtox® bioassay indicated low toxicity (reduction in bacterial luminescence).

Table 5. Comparison of Class II Inspection Results to NPDES Permit Limits - Inland Empire Paper, 9/90.

Effluent Parameter	NPDES Permit Daily Limit		Inspection Data	
	Average*	Maximum^	Ecology+	IEP+
BOD5	2374 lbs	4536 lbs	594 lbs	774 lbs
TSS	3854 lbs	7191 lbs	697 lbs	646 lbs
pH	5.0 - 9.0 all times		7.2 - 7.8	-
Rainbow trout	80% survival in 65% effluent for a 96-hr period		100% survival	-

\* - Defined as the average of the measured values obtained over a calendar month.

^ - Maximum value for any one day.

+ - Based on an average effluent discharge rate of 3.097 million gallons per day, 7am(9/11) - 7am(9/12).

Table 6. Results of Effluent and Sediment Organic Analysis - Inland Empire Paper, 9/90.

Field Station: Type: Date: Lab sample#: 3780-	Blank #1 <sup>^</sup> Transfer 9/10 87	Effluent <sup>^</sup> comp 9/11-12 85	Sludge <sup>^^</sup> grab/comp 9/12 89
Resin/Fatty Acids			
Linoleic acid	0.4 J	4	12000
Palmitoleic acid (EE)		5	25000
Decanoic acid, hexa-		15	65000 J
Oleic acid	1 J	7	21000
Pimaric acid		0.7 J	830 J
Sandaracopimaric acid		2 J	3700 J
Isopimaric acid		8 J	11000
Palustric acid		1 J	
Eicosatrienoic acid (EE)			2300 J
Dehydroabietic acid	0.2 J	14	23000
Abietic acid		16	22000
Neoabietic acid		0.3 J	190 J

J - Indicates an estimated value when result is less than specified detection limit.

<sup>^</sup> - Units in  $\mu\text{g/L}$ .

<sup>^^</sup> - Units in  $\text{mg/kg-dry}$ .

Table 7. Results of Effluent and Sludge Metals Analyses – Inland Empire Paper, September, 1990.

Field Blank								Fresh Water Criteria*		
Field Station: Type: Date: Lab Sample: #3780-	#1		#2		Effluent comp 9/11-12 85		Sludge^ grab/comp 9/12 89			
PP Metals (µg/L)	Diss.	Tot. Rec.	Diss.	Tot. Rec.	Diss.	Tot. Rec.	Tot. Rec.	Acute	Chronic	
Aluminum		10 U		1.7 J	442	1010	3200			
Antimony					1.6 J			9000	1600	
Arsenic					3.5 J	3.6 J	1.09	44	40	
Beryllium							0.1 J	130	53	
Cadmium						0.11 J	0.69 J	3.9	1.1	
Chromium							6.7	1700	210	
Copper	3 J				17.4 J	17 B	44.9	18	12	
Lead		11 J			3.8 J	1.8 JB	7.2 J	82	3.2	
Mercury					0.052 J		0.023 J	2.4	0.01	
Nickel							4.6	1800	96	
Zinc	2 J	10 J	4 J		46.2	21.1 B	47.5	120	110	
EP – Tox Metal (µg/L)										
Barium								101.0		

^ – mg/kg-dry.

\* – EPA, 1986. Quality Criteria for Water.

B – Analyte is found in the blank as well as the sample, indicates possible/probable blank contamination.

J – Indicates an estimated value when result is less than the specified limit.

U – Indicates compound was analyzed for but not detected at the given detection limit.

Table 8. Effluent Bioassay Results – Inland Empire Paper, 9/90.

Rainbow trout - 96 hour survival test				
Lab ID# 378085				
Sample (% Vol)	# tested		% survival	
	DOE	ERC	DOE^^	ERC^
Control	30*	20+	100	90
6.25		20		85
12.5		20		95
25		20		100
50		20		100
65	30	20	100	
100		20		100

\* - Three replicates of ten organisms.

+ - Two replicates of ten organisms.

DOE - Department of Ecology, Manchester Lab.

ERC - ERC Environmental and Bioassay Lab.

^ - LC50 for the cadmium chloride reference toxicant was estimated at 2.5 µg/L.

^^ - LC50 for this test was determined to be >100 percent effluent.

**Daphnia pulex - 7-day survival and reproduction test**

*(Daphnia pulex)*

Sample (% Vol)	# tested DOE*	Average # of Live Adults	Average # of Young/Female
Control	10	10	24
6.25	10	10	31
12.5	10	10	30
25	10	10	35
50	10	10	35
100	10	9	29

DOE - Department of Ecology, Manchester Lab., \* - Ten replicates of one organism.

NOEC - 100% Effluent.

LOEC - 100% Effluent.

LC50 (96 hour) - Cadmium chloride reference toxicant was estimated at 10 µg/L.

**Microtox**

Statistical analysis of the data generated by microtox analysis of effluent sample resulted in a large number of negative gammas. This renders the data unsuitable for use in estimating an EC50. Negative gammas are an indication of a lack of negative effect on light output by test organisms. According to Stinson (1990), this is normally interpreted as a lack of toxicity.

Analysis of the deionized water blank also resulted in negative gammas. The estimated EC50 (five minutes) for the reference toxicant was 0.141 mg/L phenol. This value is within the acceptable range normally obtained by Ecology's Manchester Laboratory.

Table 8. Continued – Inland Empire Paper, 9/90.

**Fathead Minnow Larvae – 7 day Survival and Growth Test**

Sample (% Vol)	# Tested		% Survival		Average Weight Per Larvae (mg)	
	NAS*	ERC+	NAS	ERC	NAS	ERC
Control	60	30	55.5	100	0.34	0.82
6.25	60	30	71.7	93.3	0.39	1.03
12.5	60	30	46.7	94.7	0.42	0.84
25	60	30	61.7	100	0.37	0.96
50	60	30	63.3	93.3	0.39	0.94
100	60	30	55	90	0.32	0.88

NAS - Northwestern Aquatic Sciences Lab.

ERC - ERC Environmental and Bioassay Lab.

\* - Four replicates of fifteen organisms.

+ - Three replicates of ten organisms.

NOEC - > 100% effluent (analyzed by ERC Lab).

**Ceriodaphnia dubia – 7 day Survival and Reproduction Test**

*(Ceriodaphnia dubia)*

Sample (% Vol)	# Tested		Average # of Live Adults		Average # of Young/Female	
	DOE*	ERC*	DOE	ERC	DOE	ERC
Control	10	10	9	9	4	23
6.25	10	10	10	9	14	23
12.5	10	10	10	8	31	30
25	10	10	9	8^^	52	29
50	10	10	10	10	45	28
100	10	10	10	9	31	15

\* - Ten replicates of one organism.

^^ - Two animals lost.

DOE - Department of Ecology.

ERC - ERC Environmental and Bioassay Lab.

NOEC - 50% effluent (analyzed by ERC Lab).

LC50 - 2.5 µg/L cadmium chloride reference toxicant (analyzed by Ecology).



A seven-day survival and growth test using fathead minnow larvae resulted in 90% survival in 100% effluent (reported by ERC Lab) and 55% survival in 100% effluent (reported by Northwestern Aquatic Sciences Lab). Northwestern Aquatic Sciences Lab encountered problems with random mortality, even in control samples (Table 8, continued). Survival in the control sample was somewhat lower than usually expected for test validation. However, lack of toxicity evident in the samples and adequacy of the QA/QC data suggest that these results are reliable and should be accepted, according to Caldwell (1990).

A seven-day survival and reproduction test using *Ceriodaphnia dubia* resulted in 90% survival in 100% effluent reported by both Ecology and ERC laboratories. NOEC for the test was 50% (analyzed by ERC Lab). These results indicate that the effluent was not toxic even at 100 percent strength.

### **Receiving Water Bioassays**

Receiving water bioassay results are given in Table 9. No toxicity was indicated by rainbow trout, which experienced 97% survival in 100% receiving water.

A seven-day survival and reproduction test using *Ceriodaphnia dubia* resulted in 100% survival in 100% receiving water.

Microtox® bioassay demonstrated no toxicity (reduction in bacterial luminescence).

A seven-day survival and reproduction test using *Daphnia pulex* resulted in 100% survival in 100% receiving water. The average number of young produced per female was 34 in 100% receiving H<sub>2</sub>O, resulting in a No Observed Effects Concentration (NOEC) of 100% receiving water.

A seven-day survival and reproduction test of Fathead minnow larvae resulted in 89.9% survival in 100% receiving water. No Observed Effects Concentration (NOEC) and Lowest Observed Effects Concentration (LOEC) for the test were 10 µg/L and 33 µg/L, respectively. Lethal Concentration for 50% organisms (LC<sub>50</sub>) for the test was 11.6 µg/L cadmium chloride as a reference toxicant (analyzed by Northwestern Aquatic Sciences). The results indicate that neither of receiving water samples was toxic to aquatic life.

### **Sludge Analyses**

General chemistry data for sludge samples collected during the inspection are listed in Table 4. Percent volatile solids measured was 89.3 while TOC was 10.7%. Phenol was detected in the sludge at 0.49 mg/kg-dry weight.

Resin/fatty acids and metals detected in sludge samples are listed in Tables 6 and 7, respectively. A number of resin/fatty acids (ranging from 190 to 65000 mg/kg-dry) were detected. Among priority pollutant metals, arsenic, chromium, copper, nickel, and zinc were detected. Barium was detected at 101 µg/L level by EP-Tox metal analysis.

Table 9. Receiving Water (Spokane River) Bioassays – Inland Empire Paper, 9/90.

Rainbow trout - 96-hour survival test

Lab ID# 378090

Sample (% Vol)	# Tested (DOE)	Percent Survival
Control^	30*	100
100	30	97

\* - Three replicates of ten organisms.

^ - Manchester City Water (dechlorinated).

Ceriodaphnia dubia - 7-day Survival and Reproduction Test.

(*Ceriodaphnia dubia*)

Sample (% Vol)	# Tested DOE*	Av. no. of Adults	Total no. of Young/Female
Control	10	9	3
6.25	10	10	2
12.5	10	10	6
25	10	10	4
50	10	10	6
100	10	10	14

\* - Ten replicates of one organism, NOEC - 100% Receiving Water.

LC50 - 2.7 µg/L Cadmium chloride reference toxicant (analyzed by Ecology).

Microtox

Statistical analysis of the data generated by Microtox analysis of effluent sample resulted in a large number of negative gammas. This renders the data unsuitable for use in estimating an EC50. Negative gammas are an indication of a lack of negative effect on light output by test organisms. This is normally interpreted as a lack of toxicity (Stinson, 1990).

Table 9. Continued – Inland Empire Paper, 9/90.

*Daphnia pulex* – 7-day Survival and Reproduction.

(*Daphnia pulex*)

Sample (% Vol)	# Tested DOE*	Av. no. of Live Adults	Av. no. of Young/Female
Control	10	10	24
6.25	10	10	25
12.5	10	9	29
25	10	8	34
50	10	9	26
100	10	10	34

DOE - Department of Ecology.

\* - Ten replicates of one organism.

No Observed Effects Concentration (NOEC) - 100% Receiving water.

Fathead minnow larvae – 7-day Survival and Growth Test

(Fathead minnow)

Sample (% Vol)	# Tested NAS*	Percent Survival	Average Weight per Larvae (mg)
Control	60	78.2	0.40
6.25	60	71.7	0.54
12.5	60	70.0	0.56
25	60	80.0	0.48
50	60	88.3	0.48
100	60	89.9	0.48

NAS - Northwestern Aquatic Sciences Lab.

\* - Four replicates of fifteen organisms.

No Observed Effects Concentration (NOEC) - 10  $\mu\text{g/L}$  Cadmium chloride.

Lowest Observed Effects Concentration (LOEC) - 33  $\mu\text{g/L}$  Cadmium chloride.

Lethal Concentration for 50% organisms (LC50) - 11.6  $\mu\text{g/L}$  Cadmium chloride.

No volatile organics, pesticides/PCBs or BNAs were detected in the sludge samples. Complete results are given in Appendix C.

### **Laboratory Review**

Ecology and IEP lab results of split samples for permit parameters compared fairly well, although the IEP lab reported somewhat lower BOD<sub>5</sub> and higher total phosphates (Table 10). An on-site review of IEP's laboratory procedures (conducted by Lisa Zinner) did not indicate any serious procedural problems, including sample collection, BOD<sub>5</sub>, TSS, and pH. The laboratory procedure check sheets are included in Appendix D.

### **Effluent Particulates Characterization by Centrifugation**

The objective of Ecology's ongoing centrifuge study is to separate colloidal and/or settleable particulates from industrial and municipal wastewater effluents and hence to determine the potential sediment contamination level by chemical analysis of recovered particulate matter. By centrifuging thousands of gallons of effluent over a period of several days, enough particulate material is collected for a detailed chemical analysis. Pollutants which would not otherwise be measurable in the effluent, may thus be quantified due to improved detection levels associated with the extremely concentrated particulate materials. A more complete discussion of centrifuge methods and results will be presented in a centrifuge study report (Andreasson, 1991 in progress).

Results including VOCs, BNAs, RFAs and metals obtained from the centrifuge study of IEP's effluent are summarized in Table 11. Whole effluent, centrate, and particulate samples were used in each analysis. Centrifuge particulate analyses for volatile and semi-volatile organics and metals found many of the same compounds found in both plant effluent and sludge.

## **CONCLUSIONS AND RECOMMENDATIONS**

### **Flow**

The IEP's Parshall flume was found to be properly installed and maintained. Comparisons of Ecology's instantaneous flow measurements to WTP effluent flow readings were good. However, a leak was noticed under the Parshall flume where the flume was attached to the concrete clarifier. It is recommended that the leak be fixed immediately.

- Instantaneous flow measurements should be repeated during the next Class II Inspection.

### **General Chemistry and NPDES Permit Compliance**

The IEP's wastewater treatment plant performed well during the inspection. The conventional parameters indicated a well-treated, high quality effluent. The mill was meeting permit limits for BOD<sub>5</sub>, TSS, and pH during the inspection.

Table 10. Comparison of Laboratory Results - Inland Empire Paper, 9/90.

Station	Type	Date	Sampler	Laboratory	BOD5 (mg/L)	TSS (mg/L)	PO4-total (mg/L)	Flow MGD*	pH SU
Effluent	Composite	9/11-12	Ecology	Ecology	23	27	0.41	3.03-	7.2-
"	"	-	Ecology	IEP	17	26	0.53	3.10	7.8
"	"	-	IEP	Ecology	30	25	0.44		
"	"	-	IEP	IEP	19	32	0.73	3.097 (totalizer)	

\* - Million gallons per day.

Table 11. Centrifuge Study Pollutants Detected - Inland Empire Paper, 9/90.

	Effluent Concentrations (grams/1,000,000 gallons)			TOC-based Concentrations (mg/Kg-TOC)	
	Whole	Centrate*	Particulates**	Particulates**	Surrogate+
<u>VOCs</u>					
Acetone	40 U		1.33 DJ	56 DJ	447 J
2-Butanone	40 U		1.12 DJ	47 DJ	76
<u>BNAs</u>					
4-Methylphenol	38 U	38 U	0.1 J	3.2 J	34.4 U
Benzoic Acid	11 J	11 J	0.3 J	13.8 J	6.9 J
Bis(2-Ethylhexyl)Phthalate	38 U	38 U	0.3 J	11.2 J	34.4 U
<u>Metals</u>					
Antimony, Total	NA	5 J	2.0 U	19.11 U	588 U
Antimony, Total recoverable	9 U	9 U	NA	NA	NA
Antimony, Dissolved	6 J	5 J	NA	NA	NA
Arsenic, Total	NA	19 J	0.2 J	1.53 J	29 U
Arsenic, Total recoverable	14 J	15 J	NA	NA	NA
Arsenic, Dissolved	13 J	20	NA	NA	NA
Cadmium, Total	NA	1 J	1.3	12.9	39 U
Cadmium, Total recoverable	0.4 J	1 J	NA	NA	NA
Cadmium, Dissolved	0.4 U	1 J	NA	NA	NA
Chromium, Total	NA	20 U	1.7	17	118 J
Chromium, Total recoverable	20 U	20 U	NA	NA	NA
Chromium, Dissolved	20 U	20 U	NA	NA	NA
Copper, Total	NA	48 B	12.6	124	216
Copper, Total recoverable	64 B	51	NA	NA	NA
Copper, Dissolved	66 B	53 B	NA	NA	NA
Lead, Total	NA	6 J	1 U	13 U	392 U
Lead, Total recoverable	7 JB	31 B	NA	NA	NA
Lead, Dissolved	14 J	8 J	NA	NA	NA
Mercury, Total	NA	0.3 J	0.03 J	0.3 J	5 J
Mercury, Total recoverable	0.2 U	0.0	NA	NA	NA
Mercury, Dissolved	0.2 J	0.1 U	NA	NA	NA
Zinc, Total	NA	51 JB	16.1	157	555
Zinc, Total recoverable	80 B	83 B	NA	NA	NA
Zinc, Dissolved	175	67 JB	NA	NA	NA
Aluminum, Total	NA	2,994	931	9,108	17,647
Aluminum, Total recoverable	3,823	3,081			
Aluminum, Dissolved	1,673	2,808			
<u>Resin/Fatty acids</u>					
Linoleic acid	15	30 J	215 J	9,052 J	44 J
Palmitoleic acid (EE)	19	15 J	143 J	6,034 J	88 J
Decanoic acid, Hexa-	57	68	286 J	12,069 J	579 U
Oleic acid	26	23 U	286 J	12,069 J	44 J
Octadecanoic acid	8 U	11 U	133	5,603	116 U
Pimaric acid	3 J	4 J	14 J	603 J	81 U
Sandaracopimaric acid	8 J	8 J	43	1,810	81 U
Isopimaric acid	30	30 J	133	5,603	11 J
Palustric acid	4 J	4 J	18 U	776 U	81 U
Dehydroabietic acid	53	57	296 J	12,500 J	14 J
Abietic acid	61	23 J	399 J	16,810 J	9 J
Neobietic acid	1 J	23 U	18 U	776 U	81 U

\* Centrate - The portion of the whole effluent that passes through the centrifuge.  
 \*\* Particulates - The portion of the whole effluent retained by the centrifuge.  
 + Surrogate - Return activated sludge  
 U Indicates analyte not detected at quantification limit given.  
 J Estimated amount, concentration is below quantification limit.  
 B Indicates method blank contamination.  
 D Analytical results from diluted sample.

Centrifuge TOC (%) = 23.2  
 Surrogate TOC (%) = 5.70  
 Effluent TSS (mg/l) = 27

A discharge through outfall 002 was discovered on Monday, September 10. This flow appeared to be about 500 gpm of non-process water. It seemed that this problem was corrected during the inspection. However, permit discharge violations such as *this must be reported to Ecology* in accordance with permit general condition G4. During the inspection, it was noted that IEP was adding defoamers downstream of the effluent sampling point. Adding defoamer after final effluent sampling is another permit violation (condition S3.d, representative sample). The effluent appeared to be turbid during the inspection. It should be noted that the treatment system was just restarting following a plant shutdown, but turbidity and color in pulp mill effluent is not uncommon.

Fecal coliform counts in the effluent were 310 #/100 mL and 240 #/100 mL, comprised of 81% and 100% *Klebsiella*, respectively. Enterococci count in the effluent was 120 #/100 mL. The level of microorganisms present in the treated effluent was high, but similar to other pulp mill effluents.

- Effluent should be sanitized prior to discharge in the Spokane River.

### **Treatment Efficiency**

Ecology was not allowed to collect influent wastewater samples during the Class II Inspection. Therefore, the objective of evaluating the plant's treatment efficiency was canceled.

- It would be desirable to have access for collecting influent samples during the next inspection.

### **Effluent Priority Pollutant Scan**

No volatile organic compounds, semi-volatile compounds, BNAs, or pesticides/PCBs were detected in the effluent. However, a number of non-priority pollutant compounds (resin/fatty acids) and priority pollutant metals were detected in WTP effluent. Among those metals detected, copper was found at 17.4  $\mu\text{g/L}$ , slightly above the acute and chronic water quality criteria. Mercury was found in the effluent at a level of 0.052  $\mu\text{g/L}$ , which is above the chronic water quality criterion. Hexavalent chromium was also found at levels above acute and chronic water quality criteria. It is unknown why hexavalent chromium levels detected in effluent were higher than water quality criteria at the time of the inspection.

- It is suggested that the permit manager review Joy's (1991 in progress) dilution ratios in relation to these metals results to determine if water quality based permit limits for metals are recommended.

### **Effluent Bioassays**

No effluent toxicity was indicated by using fathead minnow larvae, *Ceriodaphnia dubia*, and rainbow trout. A seven-day survival and reproduction test of *Daphnia pulex* resulted in 90% survival in 100% effluent. Microtox® indicated low toxicity.

## Receiving Water Bioassays

No toxicity was indicated by rainbow trout. The acute and chronic tests using *Daphnia pulex*, *Ceriodaphnia dubia*, fathead minnow and Microtox® indicated very low receiving water toxicity.

## Sludge Analyses

No pesticides/PCBs was detected in sludge samples. A number of resin/fatty acids (ranging from 190 to 65000 mg/kg-dry) were detected. VOCs and BNAs were detected in the sludge. Among VOCs, acetone, 2-butanone and toluene were detected. BNAs analysis showed that only one compound bis(2-ethyhexyl)phthalate was detected. Priority pollutant metals including aluminum, arsenic, chromium, copper, nickel, zinc, and barium were also detected in the sludge.

A problem in handling sludge was noticed during the inspection. The sludge was piling up in front of the conveyer belt instead of being loaded into a truck and taken to the landfill. There were odor and nuisance fly problems. Since the pile was not covered, the proximity of the pile to the river could cause problems in heavy rain.

- Therefore, it is recommended that runoff controls be implemented for any temporary sludge storage piles.

## Laboratory Review

Both Ecology and Inland Empire Paper laboratory results of split samples for permit parameters agreed reasonably well. An on-site review of IEP's laboratory procedures did not indicate any serious procedural problems in sample collection and analyses (Table 5).

## Centrifuge Study

Many of the compounds and metals found in the centrifuge cake analyses were also detected in sludge samples. Both metals and organics concentrations were higher in the centrifuge cake than in sludge.



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## APPENDICES

Appendix A. Chemical Analytical Methods – Inland Empire Paper, 9/90.

Parameter	Method	Lab Used
<b>GENERAL CHEMISTRY</b>		
Turbidity	EPA, 1979: 180.1	Manchester Lab., WA
Conductivity	EPA, 1979: 120.1	AMTest Inc., WA
Alkalinity	EPA, 1979: 310.1	AMTest Inc., WA
Hardness	EPA, 1979: 130.2	AMTest Inc., WA
Color	EPA, 1979: 110.1	AMTest Inc., WA
<b>SOLIDS</b>		
TS	EPA, 1979: 160.3	AMTest Inc., WA
TNVS	EPA, 1979: 106.4	AMTest Inc., WA
TSS	EPA, 1979: 160.2	AMTest Inc., WA
TNVSS	EPA, 1979: 106.4	AMTest Inc., WA
% Solids	APHA, 17: 2540G	AMTest Inc., WA
% Volatile Solids	EPA, 1979: 160.4	AMTest Inc., WA
BOD5	EPA, 1979: 405.1	AMTest Inc., WA
COD	EPA, 1979: 410.1	AMTest Inc., WA
TOC (water)	EPA, 1979: 415.2	AMTest Inc., WA
TOC (soil)	APHA, 17:5310	AMTest Inc., WA
<b>NUTRIENTS</b>		
NH3-N	EPA, 1979: 350.1	AMTest Inc., WA
NO2+NO3-N	EPA, 1979: 353.2	AMTest Inc., WA
Phosphorous - Total	EPA, 1979: 365.1	AMTest Inc., WA
Phosphate - Ortho	EPA, 1979: 365.3	AMTest Inc., WA
F-Coliform	APHA, 16: 908C	Manchester Lab., WA
% Klebsiella (KES)	APHA, 17: 9222F	Manchester Lab., WA
E. coli	APHA, 1989: 9225C	Manchester Lab., WA
Enterococci	APHA, 17: 9230B	Manchester Lab., WA
Cyanide total	EPA, 1979: 335.2mod	AMTest Inc., WA
Cyanide (wk & dis)	APHA, 17: 4500-CN I.	AMTest Inc., WA
<b>ORGANICS</b>		
EOX	EPA, 1986: 9020	Columbia Analytical Services Inc., WA
VOC (water)	EPA, 1984: 624	Manchester Lab., WA
BNAs (water)	EPA, 1984: 625	Pacific NW Environmental Labs. WA
Pest/PCBs (water)	EPA, 1984: 608	Manchester Lab., WA
Resin/Fatty Acids (water)	NCASI, 1986b: RAFA	Manchester Lab., WA
Phenol (water)	EPA, 1979: 420.2	AMTest Inc., WA
<b>PRIORITY POLLUTANTS</b>		
PP Metals	EPA, 1979: 200	Manchester Lab., WA
Total (soil)	EPA, 1979: 200	Manchester Lab., WA
Total (water)	EPA, 1979: 200	Manchester Lab., WA
Hexavalent Chromium	EPA, 1984: 200	AMTest Inc., WA
EP Tox Metals	EPA, SW-846 #1310	Manchester Lab., WA
<b>BIOASSAYS</b>		
Rainbow trout (acute)	Ecology, 1981	ERCE and Bioassay Lab., CA
Microtox (acute)	Beckman, 1982	Microbics Corporation, CA
Daphnia pulex (acute)	EPA, 1987	Manchester Lab., WA
Daphnia pulex (chronic)	EPA, 1987	Manchester Lab., WA
Ceriodaphnia (chronic)	EPA, 600/4-85/014	ERCE and Bioassay Lab., CA
Fathead minnow (chronic)	EPA, 600/4-89/001	Northwestern Aquatic Sciences, OR

Appendix B. Results of Effluent Pesticides/PCBs and Priority Pollutant Metals Analyses -  
Inland Empire Paper, 9/90.

Parameter ( $\mu\text{g/l}$ )	Field Station:	Effluent	Blank	
	Type:	comp	transfer	
	Date:	9/11-12	9/10	
	Time:	24 hr	pm	
	Lab sample#: 3780 -	85	87	
alpha-BHC		0.05 UJ	0.05 U	
gamma-BHC (Lindane)		0.05 UJ	0.05 U	
beta-BHC		0.05 UJ	0.05 U	
Heptachlor		0.05 UJ	0.05 U	
delta-BHC		0.05 UJ	0.05 U	
Aldrin		0.05 UJ	0.05 U	
Heptachlor Epoxide		0.05 UJ	0.05 U	
Endosulfan I		0.05 UJ	0.05 U	
4,4'-DDE		0.1 UJ	0.1 U	
Dieldrin		0.1 UJ	0.1 U	
Endrin		0.1 UJ	0.1 U	
4,4'-DDD		0.1 UJ	0.1 U	
Endosulfan II		0.1 UJ	0.1 U	
4,4'-DDT		0.1 UJ	0.1 U	
Endrin Ketone		0.1 UJ	0.1 U	
Endosulfan Sulfate		0.1 UJ	0.1 U	
Methoxychlor		0.5 UJ	0.5 U	
Toxaphene		1 UJ	1 U	
alpha-Chlordane		0.5 UJ	0.5 U	
gamma-Chlordane		0.5 UJ	0.5 U	
Aroclor-1016		0.5 UJ	0.5 U	
Aroclor-1221		0.5 UJ	0.5 U	
Aroclor-1232		0.5 UJ	0.5 U	
Aroclor-1242		0.5 UJ	0.5 U	
Aroclor-1248		0.5 UJ	0.5 U	
Aroclor-1254		1 UJ	1 U	
Aroclor-1260		1 UJ	1 U	
Metals ( $\mu\text{g/L}$ )	Field Station:	Effluent		Blank
	Type:	comp	comp	transfer
	Date:	9/11-12	9/11-12	9/10
	Time:	24 hr	24 hr	pm
	Analysis type:	total rec.	diss.	total rec.
	Lab sample#: 3780 -	85	85	87
Antimony		2.5 U	1.5 J	2.5 U
Arsenic		3.6 J	3.5 J	1.5 U
Beryllium		1 U	1 U	1 U
Cadmium		0.11 J	0.1 U	0.1 U
Chromium		5 U	5 U	5 U
Copper		17 B	17.4 B	3 J
Lead		1.8 JB	3.8 J	1 U
Mercury		0.04 U	0.052 J	0.1 J
Nickel		10 U	10 U	10 U
Selenium		2 U	1 U	2 U
Silver		2 U	2 U	2 U
Thallium		2.5 U	2.5 U	2.5 U
Zinc		21.1 B	46.2	4.2 JB
Aluminum		1010	442	

U - Indicates compound was analyzed for but not detected at the given detection limit.

J - Indicates an estimated value when result is less than specified detection limit.

B - Analyte was found in blank as well as a sample, and indicates possible/probable blank contamination.

UJ - Estimated method quantitation limit.

Appendix B – Cont. – Results of Effluent BNA Analyses – Inland Empire Paper, 9/90.

	Field Station:	Effluent	Blank
	Type:	comp	transfer
	Date:	9/11-12	9/10
	Time:	24 hr	pm
Parameter ( $\mu\text{g/L}$ )	Lab sample#:	378085	378087
N-Nitrosodiphenylamine		10 U	10 U
Bis(2-Chloroethyl)Ether		10 U	10 U
1,3-Dichlorobenzene		10 U	10 U
1,4-Dichlorobenzene		10 U	10 U
1,2-Dichlorobenzene		10 U	10 U
Bis(2-chloroisopropyl)ether		10 U	10 U
N-Nitroso-Di-n-Propylamine		10 U	10 U
Hexachloroethane		10 U	10 U
Nitrobenzene		10 U	10 U
Isophorone		10 U	10 U
Bis(2-Chloroethoxy)Methane		10 U	10 U
1,2,4-Trichlorobenzene		10 U	10 U
Naphthalene		10 U	10 U
4-Chloroaniline		10 U	10 U
Hexachlorobutadiene		10 U	10 U
4-Chloro-3-methylphenol		10 U	10 U
2-Methylnaphthalene		10 U	10 U
Hexachlorocyclopentadiene		10 U	10 U
2-Chloronaphthalene		10 U	10 U
2-Nitroaniline		50 U	50 U
Dimethyl Phthalate		10 U	10 U
Acenaphthylene		10 U	10 U
3-Nitroaniline		50 U	50 U
Acenaphthene		10 U	10 U
Dibenzofuran		10 U	10 U
2,4-Dinitrotoluene		10 U	10 U
2,6-Dinitrotoluene		10 U	10 U
Diethyl Phthalate		10 U	10 U
4-Chlorophenyl-Phenylether		10 U	10 U
Fluorene		10 U	10 U
4-Nitroaniline		50 U	50 U
4-Bromophenyl-Phenylether		10 U	10 U
Hexachlorobenzene		10 U	10 U

U - Indicates compound was analyzed for but not detected at the given detection limit.

J - Indicates an estimated value when result is less than specified detection limit.

Appendix B – Cont. – Results of Effluent BNA Analyses – Inland Empire Paper,  
9/90.

Parameter ( $\mu\text{g/L}$ )	Field Station: Type: Date: Time: Lab sample#:	Effluent comp 9/11-12 24 hr 378085	Blank transfer 9/10 pm 378087
Phenanthrene		10 U	10 U
Anthracene		10 U	10 U
Dibutylphthalate		10 U	10 U
Fluoranthene		10 U	10 U
Pyrene		10 U	10 U
Butylbenzylphthalate		10 U	10 U
3,3'-Dichlorobenzidine		20 U	20 U
Benzo(a)Anthracene		10 U	10 U
Bis(2-Ethylhexyl)phthalate		10 U	10 U
Chrysene		10 U	10 U
Di-n-Octyl Phthalate		10 U	10 U
Benzo(b)Fluoranthene		10 U	10 U
Benzo(k)Fluoranthene		10 U	10 U
Benzo(a)Pyrene		10 U	10 U
Indeno(1,2,3-cd)Pyrene		10 U	10 U
Dibenzo(a,h)Anthracene		10 U	10 U
Benzo(g,h,i)Perylene		10 U	10 U
Phenol		10 U	10 U
2-Chlorophenol		10 U	10 U
Benzyl Alcohol		10 U	10 U
2-Methylphenol		10 U	10 U
4-Methylphenol		10 U	10 U
2-Nitrophenol		10 U	10 U
2,4-Dimethylphenol		10 U	10 U
Benzoic Acid		3 J	50 U
2,4-Dichlorophenol		10 U	10 U
2,4,6-Trichlorophenol		50 U	10 U
2,4,5-Trichlorophenol		50 U	50 U
2,4-Dinitrophenol		50 U	50 U
4-Nitrophenol		50 U	50 U
4,6-Dinitro-2-Methylphenol		50 U	50 U
Pentachlorophenol		50 U	50 U

U - Indicates compound was analyzed for but not detected at the given detection limit.

J - Indicates an estimated value when result is less than specified detection limit.

☼ - Compound detected.

Appendix B. Cont. – Results of Effluent Resin/Fatty Acids Analyses – Inland Empire  
Paper, 9/90.

Parameter ( $\mu\text{g/L}$ )	Field Station: Type: Date: Time: Lab sample#:	Effluent comp 9/11-12 24 hr 378085	Blank transfer 9/10 pm 378087
Linoleic acid		4	0.4 J
Palmitoleic acid (EE)		5	2 U
Decanoic acid, hexa-		15	2 U
Oleic acid		7	1 J
Octadecanoic acid		2 U	2 U
Retene		2 U	2 U
Pimaric acid		0.7 J	2 U
Sandaracopimaric acid		2 J	2 U
Isopimaric acid		8	2 U
Palustric acid		1 J	2 U
Eicosatrienoic acid (EE)		2 U	2 U
Dehydroabietic acid		14	0.2 J
Abietic acid		16	2 U
Neoabietic acid		0.3 J	2 U
9,10-Dichlorosteric acid		2 U	2 U
14-Chlorodehydroabietic		2 U	2 U
12-Chlorodehydroabietic		2 U	2 U
Dichlorodehydroabietic acid		2 U	2 U

U – Indicates compound was analyzed for, but not detected at the given limit.

J – Indicates an estimated value when result is less than specified detection limit.

J – Compound detected.



Appendix B. Cont. – Results of Effluent VOC Analysis – Inland Empire Paper,  
9/90.

Parameter (ug/L)	Field Station:	EFFLUENT		BLANK
	Type:	grab	grab	TransBlank
	Date:	9/11	9/11	9/11
	Time:	am	pm	pm
	Lab sample#:	378083	3768084	378088
Chloromethane		10 U	10 U	10 U
Vinyl Chloride		10 U	10 U	10 U
Bromomethane		10 U	10 U	10 U
Chloroethane		10 U	10 U	10 U
1,2-Dichloroethene (total)		5 U	5 U	5 U
1,1-Dichloroethene		5 U	5 U	5 U
Acetone		10 U	10 U	10 U
Carbon Disulfide		5 U	5 U	5 U
Methylene Chloride		5 U	5 U	5 U
2-Butanone		10 U	10 U	10 U
1,1-Dichloroethane		5 U	5 U	5 U
Chloroform		5 U	5 U	5 U
1,1,1-Trichloroethane		5 U	5 U	5 U
Carbon Tetrachloride		5 U	5 U	5 U
Benzene		5 U	5 U	5 U
1,2-Dichloroethane		5 U	5 U	5 U
Vinyl Acetate		10 U	10 U	10 U
Trichloroethene		5 U	5 U	5 U
1,2-Dichloropropane		5 U	5 U	5 U
Bromodichloromethane		5 U	5 U	5 U
Trans-1,3-Dichloropropene		5 U	5 U	5 U
2-Hexanone		10 U	10 U	10 U
4-Methyl-2-Pentanone		10 U	10 U	10 U
Toluene		5 U	5 U	5 U
cis-1,3-Dichloropropene		5 U	5 U	5 U
1,1,2-Trichloroethane		5 U	5 U	5 U
Tetrachloroethene		5 U	5 U	5 U
Dibromochloromethane		5 U	5 U	5 U
Chlorobenzene		5 U	5 U	5 U
Ethylbenzene		5 U	5 U	5 U
Styrene		5 U	5 U	5 U
Total Xylenes		5 U	5 U	5 U
Bromoform		5 U	5 U	5 U
1,1,2,2-Tetrachloroethane		5 U	5 U	5 U

U - Indicates compound was analyzed for but not detected at the given detection limit.

Appendix C. Results of Sludge Priority Pollutant Metals, EP-TOX Metals and Pesticide/PCBs  
Analyses Inland Empire Paper, 9/90.

	Field Station:	Sludge	Sludge
	Type:	grab/comp	grab/comp
	Date:	9/11-12	9/11-12
	Time:	24 hr	24 hr
	Lab sample#:	378089	378089
Parameter		PP Metals (total mg/kg-dry)	EP-TOX Metals (total µg/L)
Antimony		3 U	
Arsenic		1.09	500 U
Barium			101
Beryllium		0.1 J	
Cadmium		0.69 J	50 U
Chromium		6.7	100 U
Copper		44.9	
Lead		7.2 J	500 U
Mercury		0.023 J	x*
Nickel		4.6 J	
Selenium		0.2 U	100 U
Silver		0.2 U	50 U
Thallium		0.25 U	
Zinc		47.5	
Aluminum		3200	
<b>Pest/PCBs (µg/kg-dry)</b>			
alpha-BHC		110 U	
gamma-BHC (Lindane)		110 U	
beta-BHC		110 U	
Heptachlor		110 U	
delta-BHC		110 U	
Aldrin		110 U	
Heptachlor Epoxide		110 U	
Endosulfan I		110 U	
4,4'-DDE		220 U	
Dieldrin		220 U	
Endrin		220 U	
4,4'-DDD		220 U	
Endosulfan II		220 U	
4,4'-DDT		220 U	
Endrin Ketone		220 U	
Endosulfan Sulfate		220 U	
Methoxychlor		1100 U	
Toxaphene		2200 U	
alpha Chlordane		1100 U	
gamma Chlordane		1100 U	
Aroclor-1016		1100 U	
Aroclor-1221		1100 U	
Aroclor-1232		1100 U	
Aroclor-1242		1100 U	
Aroclor-1248		1100 U	
Aroclor-1254		2200 U	
Aroclor-1260		2200 U	

U - Indicates compound was analyzed for but not detected at the given detection limit.

J - Indicates an estimated value when result is less than specified detection limit.

x\* - Barometer was not run.

▨ - Compound detected.

Appendix C. Cont. - Results of Sludge BNA Analyses - Inland Empire Paper, 9/90.

Field Station:	Sludge	Sludge	
Type:	grab/comp	grab/comp	
Date:	9/11-12	9/11-12	
Time:	24 hr	24 hr	
Lab sample#:	378089	378089	
Parameter ( $\mu\text{g}/\text{kg-dry}$ )	Parameter ( $\mu\text{g}/\text{kg-dry}$ )	Parameter ( $\mu\text{g}/\text{kg-dry}$ )	
Phenol	2300 U	3-Nitroaniline	11000 U
Bis(2-Chloroethyl)Ether	2300 U	Acenaphthene	2300 U
2-Chlorophenol	2300 U	2,4-Dinitrophenol	11000 U
1,3-Dichlorobenzene	2300 U	4-Nitrophenol	11000 U
1,4-Dichlorobenzene	2300 U	Dibenzofuran	2300 U
Benzyl Alcohol	2300 U	2,4-Dinitrotoluene	2300 U
1,2-Dichlorobenzene	2300 U	Diethyl Phthalate	2300 U
2-Methylphenol	2300 U	4-Chlorophenyl-Phenylether	2300 U
Bis(2-chloroisopropyl)ether	2300 U	Fluorene	2300 U
4-Methylphenol	2300 U	4-Nitroaniline	11000 U
N-Nitroso-Di-n-Propylamine	2300 U	4,6-Dinitro-2-Methylphenol	11000 U
Hexachloroethane	2300 U	N-Nitrosodimethylamine	2300 U
Nitrobenzene	2300 U	4-Bromophenyl-Phenylether	2300 U
Isophorone	2300 U	Hexachlorobenzene	2300 U
2-Nitrophenol	2300 U	Pentachlorophenol	11000 U
2,4-Dimethylphenol	4600 U	Phenanthrene	2300 U
Benzoic Acid	11000 U	Anthracene	2300 U
Bis(2-Chloroethoxy)Methane	2300 U	Di-n-Butylphthalate	2300 U
2,4-Dichlorophenol	2300 U	Fluoranthene	2300 U
1,2,4-Trichlorobenzene	2300 U	Pyrene	2300 U
Naphthalene	2300 U	Butylbenzylphthalate	2300 U
4-Chloroaniline	11000 U	3,3'-Dichlorobenzidine	4600 U
Hexachlorobutadiene	11000 U	Benzo(a)Anthracene	2300 U
4-Chloro-3-Methylphenol	2300 U	Bis(2-Ethylhexyl)phthalate	1000 J
2-Methylnaphthalene	2300 U	Chrysene	2300 U
Hexachlorocyclopentadiene	2300 U	Di-n-Octyl Phthalate	2300 U
2,4,6-Trichlorophenol	11000 U	Benzo(b)Fluoranthene	2300 U
2,4,5-Trichlorophenol	11000 U	Benzo(k)Fluoranthene	2300 U
2-Chloronaphthalene	2300 U	Benzo(a)Pyrene	2300 U
2-Nitroaniline	11000 U	Indeno(1,2,3-cd)Pyrene	2300 U
Dimethyl Phthalate	2300 U	Dibenzo(a,h)Anthracene	2300 U
Acenaphthylene	2300 U	Benzo(g,h,i)Perylene	2300 U
2,6-Dinitrotoluene	2300 U		

U - Indicates compound was analyzed for but not detected at the given detection limit.

J - Indicates an estimated value when result is less than specified detection limit.

• - Compound detected.

Appendix C. Cont. - Results of Sludge Resin/Fatty Acids - Inland Empire Paper, 9/90.

	Field Station:	Sludge
	Type:	grab/comp
	Date:	9/11-12
	Time:	24 hr
Parameter ( $\mu\text{g}/\text{kg-dry}$ )	Lab sample#:	378089
Linoleic acid		12000
Palmitoleic acid (EE)		25000
Decanoic acid, hexa-		65000 J
Oleic acid		21000
Octadecanoic acid		7700 U
Retene		4500 U
Pimaric acid		830 J
Sandaracopimaric acid		3700 J
Isopimaric acid		11000
Palustric acid		4500 U
Eicosatrienoic acid (EE)		2300 J
Dehydroabietic acid		23000
Abietic acid		22000
Neoabietic acid		190 J
9,10-Dichlorosteric acid		4500 U
14-Chlorodehydroabietic		4500 U
12-Chlorodehydroabietic		4500 U
Dichlorodehydroabietic acid		4500 U

U - Indicates compound was analyzed for, but not detected at the given limit.

J - Indicates an estimated value when result is less than specified detection limit.

☐ - Compound detected.

Appendix C. Cont. – Results of Sludge VOC Analyses – Inland Empire Paper,  
9/90.

Parameter ( $\mu\text{g}/\text{kg-dry}$ )	Field Station: Type: Date: Time: Lab sample#:	Sludge grab/comp 9/11-12 24 hr 378089
Chloromethane		69 U
Bromomethane		69 U
Vinyl Chloride		69 U
Chloroethane		69 U
Methylene Chloride		34 U
Acetone		28000 E
Carbon Disulfide		34 U
1,1-Dichloroethene		34 U
1,1-Dichloroethane		34 U
1,2-Dichloroethene (total)		34 U
Chloroform		34 U
1,2-Dichloroethane		34 U
2-Butanone		4700 E
1,1,1-Trichloroethane		34 U
Carbon Tetrachloride		34 U
Vinyl Acetate		69 U
Bromodichloromethane		34 U
1,2-Dichloropropane		34 U
cis-1,3-Dichloropropene		34 U
Trichloroethene		34 U
Dibromochloromethane		34 U
1,1,2-Trichloroethane		34 U
Benzene		34 U
trans-1,3-Dichloropropene		34 U
Bromoform		34 U
4-Methyl-2-Pentanone		69 U
2-Hexanone		69 U
Tetrachloroethene		34 U
1,1,2,2-Tetrachloroethane		34 U
Toluene		10 J
Chlorobenzene		34 U
Ethylbenzene		34 U
Ethenylbenzene (Styrene)		34 U
Total Xylenes		34 U

U - Indicates compound was analyzed for but not detected at the given detection limit.

E - Estimate, sediment QA/QC was outside acceptable limits.

J - Indicates an estimated value when result is less than specified detection limit.

- Compound detected

APPENDIX D - Laboratory Evaluation

Laboratory Procedure Review Sheet

Discharger: *Inland Empire Paper*

Date: *9-11-90*

Discharger representative: *Nick Cavender*

Ecology reviewer: *Lisa Zinner*

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 cited to help give guidance for making improvements. References cited 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?  
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? *7 d/week*
5. What time does sample collection usually begin? *7 am - 7 am*
6. How long does sample collection last? *24 hrs*
7. How often are subsamples that make up the composite collected? *15 - 30 sec*
8. What volume is each subsample? *50 mls*
9. What is the final volume of sample collected? *3-5 gals*
10. Is the composite cooled during collection? *yes*

11. To what temperature? *38-45 °F*  
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?  
The temperature should be checked at least monthly to assure adequate cooling.
14. Are the sampling locations representative? *yes*
15. Are any return lines located upstream of the influent sampling location? *no* *pressure is returned.*  
This should be avoided whenever possible.
16. How is the sample mixed prior to withdrawal of a subsample for analysis? *stirred with a stick*  
The sample should be thoroughly mixed.
17. How is the subsample stored prior to analysis? *TSS, pH → immediately*  
*BOC @ 4° C*  
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? *every day*  
The jugs should be thoroughly rinsed after each sample is complete and occasionally be washed with a non-phosphate detergent. *effluent washed*
19. How often are the sampler lines cleaned?  
Rinsing lines with a chlorine solution every three months or more often where necessary is suggested. *as needed* *Teflon-coated lines*

## pH Test Review

1. How is the pH measured? *meter / 2-pt. calibration*  
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?  
Standard Methods or the Ecology handout should be used.
2. How often are BODs run? *3/week samples daily*  
The minimum frequency is specified in the permit. *Mon/Wed/Thurs/Fri*
3. How long after sample collection is the test begun?  
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. *personnel problems, because of readings on weekends*
4. Is distilled or deionized water used for preparing dilution water? ✓
5. Is the distilled water made with a copper free still?  
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 <sup>or</sup> ~~of~~ powder pillows used to make dilution water? ✓ *Hach*  
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? *daily as needed*  
Dilution water should be made for each set of BODs run.
9. Is the dilution water aged prior to use? *no*  
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? *no*  
If yes, are they seeded?  
Samples that have been frozen should be seeded (SSM p38).
11. Is the pH of all samples between 6.5 and 7.5? *yes*  
If no, is the sample pH adjusted?  
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?  
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 necessary?

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 thiosulfate 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?

If with a meter, how is the meter calibrated? *yes, meter*

Air calibration is adequate. Use of a barometer to determine saturation is desirable, although not mandatory. 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. *altitude method*

How frequently is the meter calibrated? *✓*

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? *✓*

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? *~0.2*

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? *1*

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*

How many bottles are incubated at each dilution? *2*

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? ✓  
 What is the typical initial DO? 7 - 7.4  
 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? 2 mg/L  
 What is the minimum DO that should be remaining after 5 days? 2 mg/L  
 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? Sometimes on Round Robin sent to NABJ  
 Which? Not IEP effluent  
 What is the seed source? 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?  
 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?  
 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, #51: 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? ✓  
 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? ✓  
 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, #51: 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? *SM*
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 ~~403~~-105 degrees C (SM p96, #3a: SSM p23).
4. Are any volatile suspended solids tests run? *NO*  
If yes--What is the muffle furnace 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 furnace 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?

	Minimum	Average
Influent	50	
Effluent		50

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

	Time
Influent	
Effluent	5 secs

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 sterilized?

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 instructions 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?

Sodium thiosulfate (1mL 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?

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 should 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 inhibition 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):

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