

**Simpson Tacoma Kraft Company
February 1991 Class II Inspection Report**

by
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ABSTRACT

A Class II Inspection was conducted on February 11-13, 1991, at the Simpson Tacoma Kraft Company (Simpson) pulp and paper plant in Tacoma. The plant discharge into Inner Commencement Bay is limited by NPDES permit WA-000085-0. Permit parameters were within daily maximum limits during the inspection. The effluent total suspended solids load approximated the monthly average permit limit while the BOD₅ load was much less than the monthly average limit. The few priority pollutant organics detected in the effluent by the base-neutral acid extractables (BNA) and volatile organic analysis (VOA) scans were at concentrations less than toxicity criteria. Inspection dioxin/furan results require verification. Rainbow trout, *Daphnia pulex*, fathead minnow, and blue mussel bioassays demonstrated no acute toxicity in the Simpson effluent. Fathead minnow and blue mussel demonstrated some chronic toxicity in the effluent.

INTRODUCTION

A Class II Inspection was conducted on February 11-13, 1991 at the Simpson Tacoma Kraft Company (Simpson) pulp and paper plant in Tacoma. The inspection was conducted by Tom Nell and Marc Heffner of the Ecology Compliance Monitoring Section, and Greg Bean of the Ecology Industrial Section. Dick Forsberg represented Simpson and provided assistance.

Simpson operates a bleached and unbleached kraft pulp and paper pulp mill on the Tacoma tide flats. The plant discharge into Inner Commencement Bay is limited by NPDES permit WA-000085-0 (Figure 1). A draft of the updated version of the permit was under review at the time of the inspection and was issued on June 25, 1991. A recently installed new washer line was operating during the inspection.

The inspection was designed to focus on two areas of the mill; the wastewater treatment system and the bleach plant. Specific objectives were:

1. Verify NPDES permit self monitoring.
2. Assess effluent toxicity with bioassays and pollutant scans.
3. Assess secondary wastewater treatment plant efficiency.
4. Assess effluent, bleach plant, and sludge dioxin concentrations.
5. Review permit parameter lab procedures at the mill to determine adherence to accepted protocols. Samples were split with the permittee to determine the comparability of Ecology and permittee laboratory results.

PROCEDURES

Ecology collected composite and grab samples of the secondary influent (Inf), final effluent (Ef), bleach plant alkaline waste stream (Alk), and bleach plant acid waste stream (Acd). Also, a grab composite sample of sludge was collected. Ecology Isco composite samplers were set up to collect equal volumes of sample every 30 minutes for 24 hours. Sampler configurations and locations are summarized in Figure 2.

Simpson also collected effluent composite samples. The Simpson sampler was set to collect equal volumes of sample every 15 minutes for 24 hours. Ecology and Simpson samples were split for analysis by both the Ecology and Simpson labs. Samples collected, sampling times and parameters analyzed are summarized in Appendix A.

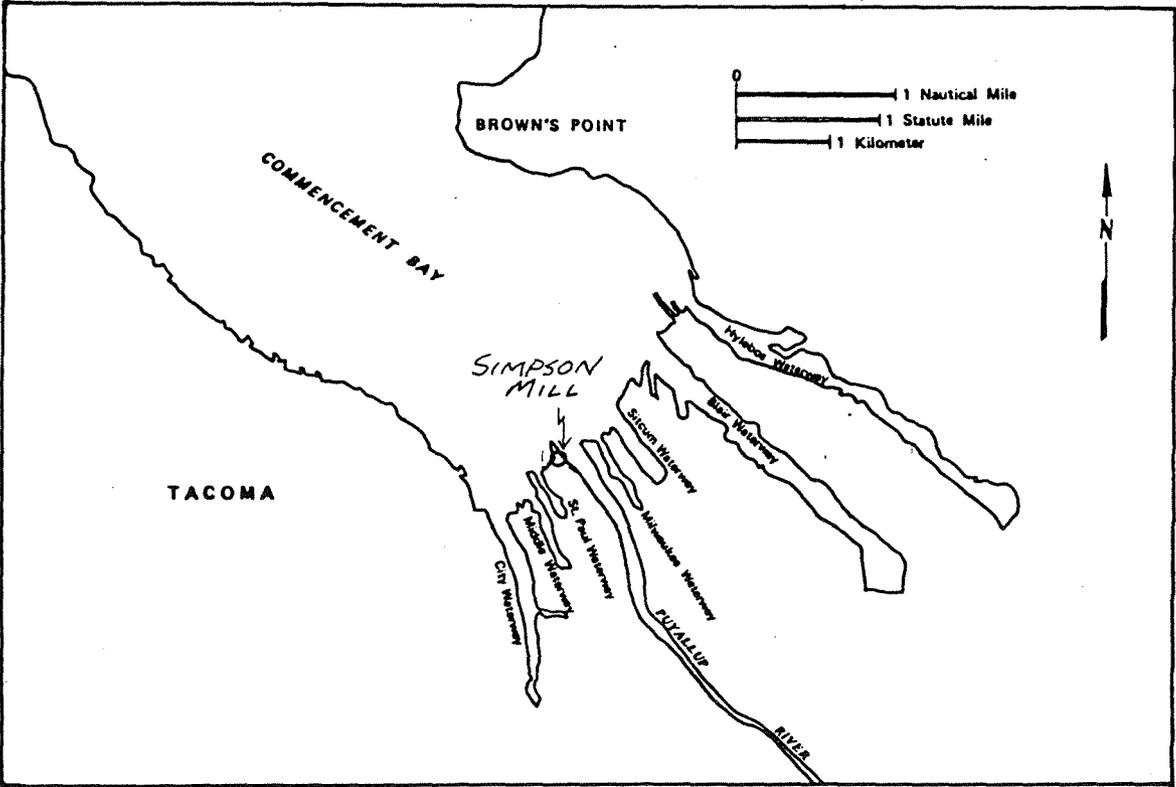
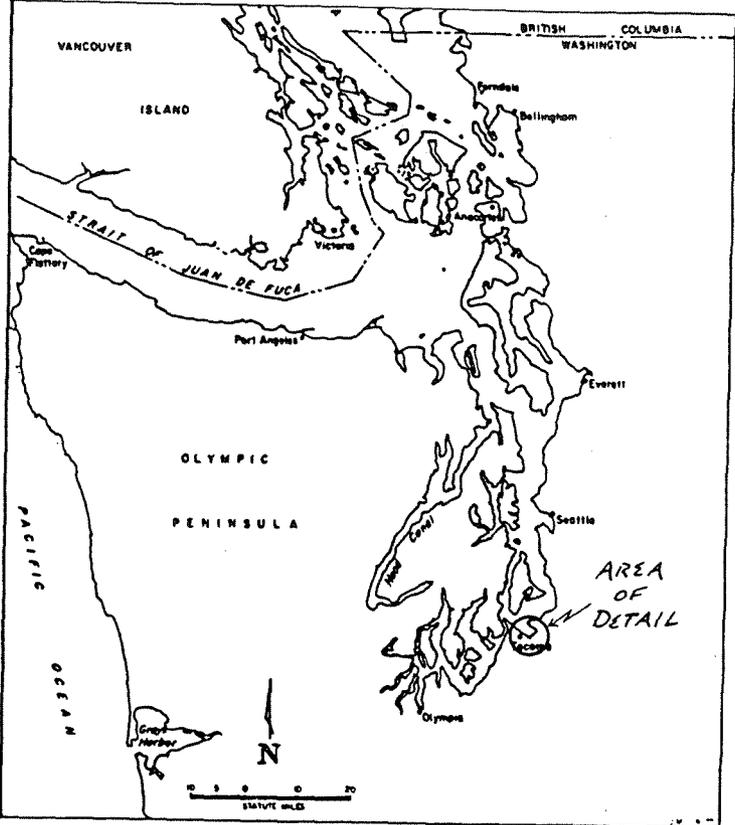
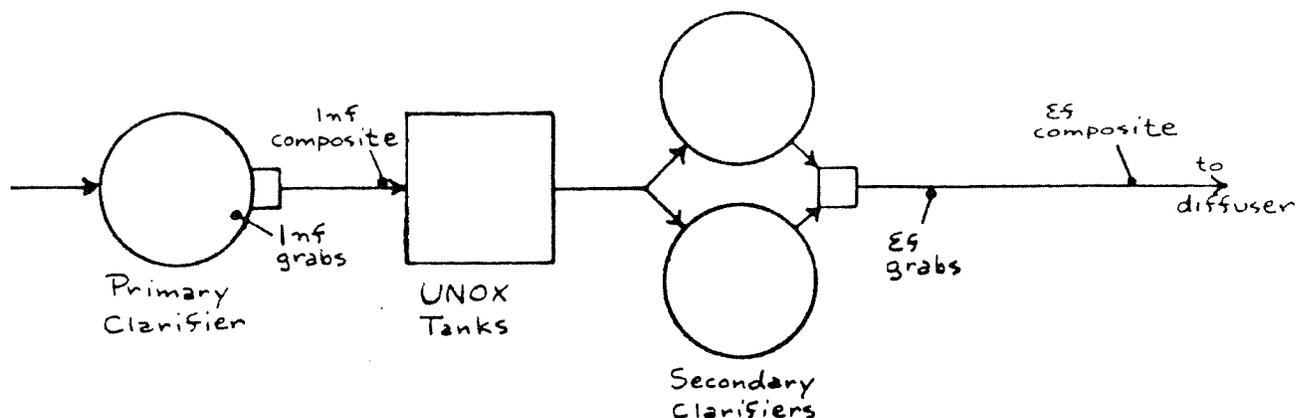


Figure 1. Mill Location - Simpson, February 1991.



Influent (Inf) - influent to the secondary treatment system.

Grab samples - collected at the overflow weir of the primary clarifier.

Composite sample - collected from the inlet box just upstream of the UNOX basins.

Two Ecology samplers were used.

Effluent (Ef) - wastewater treatment plant effluent.

Grab samples and bioassay samples - collected at the Simpson back-up sampler tap into the outfall line. The tap is near the secondary clarifiers.

Composite sample - collected at the Simpson effluent sampler tap into the outfall line.

The tap is located near the chip storage pile. Two Ecology samplers were used.

Bleach Plant Acid Stream (Acd)

Grab samples - collected at the acid stream line tap in the bleach plant.

Composite sample - collected at mid-depth of a priority pollutant cleaned stainless steel bucket filled from the acid stream line tap. The flow rate was set so the bucket was continuously overflowing and mixing was adequate to prevent solids settling. One Ecology sampler was used.

Bleach Plant Alkaline Stream (Alk)

Grab samples - collected at the alkaline stream line tap in the bleach plant.

Composite sample - collected at mid-depth of a priority pollutant cleaned stainless steel bucket filled from the alkaline stream line tap. The flow rate was set so the bucket was continuously overflowing and mixing was adequate to prevent solids settling. One Ecology sampler was used.

Sludge

Samples collected from the conveyer belt below the screw presses. Sample was combined primary and secondary sludge.

Figure 2 - Simplified Treatment System Schematic and Sampling Station Descriptions - Simpson, February 1991.

Samples for Ecology analysis were placed on ice and delivered to the Ecology Manchester Laboratory. Analytical procedures and the laboratories doing the analysis are summarized in Appendix B.

RESULTS AND DISCUSSION

Quality Assurance/Quality Control (QA/QC)

Sampling

Quality assurance/quality control steps included special cleaning of the sampling equipment prior to the inspection to prevent sample contamination by the sampling equipment (Appendix C). Also, a field transfer/equipment blank was collected (Appendix C). The blank was analyzed for parameters noted in Appendix A. Analytes detected in the transfer/equipment blank are noted on the appropriate data tables.

Dissolved Organic Halides (DOX) Analysis

Breakthrough from the first column into the second column ranged from 14-31%; exceeding the criteria of 10%. Only sample 078131 was run in duplicate, whereas the analytical method requires all samples to be run in duplicate. Carbon blanks, daily standards, and results of the duplicate analysis run appeared acceptable. Due to the breakthrough, results may be biased low, and are considered estimates. Results are flagged with a "J."

Priority Pollutant Organics Analysis

Holding times, method blanks, matrix spikes, and surrogate recoveries met Ecology standards for data use without qualification.

Metals Analysis

Holding times, instrument calibration, standard reference material, and ICP serial dilution analysis were acceptable. Copper was found at 2.2 $\mu\text{g}/\text{L}$ in the method blank: all copper results less than 22 $\mu\text{g}/\text{L}$ are flagged with a "B." The "B" flag indicates the accuracy of the result is compromised by the presence of the analyte in the method blank. Spiked sample recoveries were low for arsenic, cadmium, selenium, and mercury, and spike and spike duplicate relative percent differences (RPDs) were high for arsenic and cadmium for sample 078133. As a result samples 078133 and 078136 are flagged as appropriate for possible low recovery and poor precision. Spike recovery data and RPDs for other samples and metals were acceptable.

Guaiacols/Catechols and Resin Acids/Fatty Acids Analysis

Hexadecanoic acid (2 µg/L-estimated) and octadecanoic acid (0.9 µg/L-estimated) were found in the method blank at low concentrations: the two compounds when found in samples at concentrations less than five times the blank concentrations are flagged with a B. Holding times, blank results other than those noted above, surrogate recovery, and matrix spike and spike duplicate results were acceptable.

Dioxins/Furans

Calibration, matrix spike and matrix spike duplicate data met Ecology QA/QC limits. Internal standard recoveries were acceptable for the water samples, but low for several compounds in the sludge sample (compounds with low recoveries are flagged "UJ"). Method blank data were acceptable for all compounds except octachlorodibenzodioxin (OCDD), which was found in the water method blank (26 pg/L) and the solids method blank (7400 pg/Kg). Concentrations within five times the method blank concentration are flagged with a "B."

Bioassays

Control and reference toxicant data were acceptable.

General Chemistry/NPDES Permit Limits

The secondary treatment process removed approximately 95% of the incoming BOD₅ during the inspection (Table 1). The BOD₅ concentration was reduced from 282 mg/L to 15 mg/L. TSS concentrations were only slightly lower in the effluent (65 mg/L) than in the influent (90 mg/L). NH₃-N, NO₂+NO₃-N, and Total-P concentrations in the effluent were low - less than 0.5 mg/L. Fecal coliform concentrations ranged from 240-2200/100 mL, with %Klebsiella (KES) ranging from 23-76 percent. The oil and grease concentration in one of the two effluent grab samples was 41 mg/L; suggesting a need for occasional monitoring to determine typical effluent concentrations. All parameters were within the daily maximum limits established by the NPDES permit issued June 21, 1991 (Table 2). The effluent TSS load was approximately equal to the monthly average permit limit.

Base-Neutral Acid Extractable (BNA), Volatile Organic Analysis (VOA), and Metals Priority Pollutant Scans

Few organic priority pollutants were detected in the effluent with the BNA and VOA scans (Table 3). Chloroform (120-130 µg/L) and acetone (39-42 µg/L) were the organics found at the highest effluent concentrations. All BNA and VOA compounds were well below EPA water quality toxicity criteria concentrations (EPA, 1986). Metals concentrations were low, although the nickel concentration exceeded the marine chronic toxicity concentration, the lead and mercury concentrations exceeded the marine and freshwater chronic toxicity concentrations, and the copper concentration exceed freshwater and marine acute and chronic toxicity criteria. Copper was detected in the method blank (2.2 µg/L) as well as in the effluent sample (16 µg/L).

Table 1 - Ecology General Chemistry Results - Simpson, February 1991.

	Location:	Trms Blk	Inf-1	Inf-2	Inf-C	Ef-1	Ef-2	Ef-C	Ef-GC	Ef-Sim	Sludge
	Type:	grab	grab	grab	comp	grab	grab	comp	gr-comp	S-comp	gr-comp
	Date:	2/11	2/12	2/12	2/12-13	2/12	2/12	2/12-13	2/12	2/12-13	2/12
	Time:	1610	1025	1415	0800-0800	1205	1515	0800-0800	1205&1515	0800-0800	1400&1630
Parameter	Lab Log#:	078130	078131	078132	078133	078134	078135	078136	078137	078138	078139

LABORATORY RESULTS

Conductivity (umhos/cm)					1360			1305	1205	1290	
Alkalinity (mg/L CaCO3)					218			204	176		
Hardness (mg/L CaCO3)					84.0			80.0	70.4	75.5	
Color (C.U.)					1000			1000			
TS (mg/L)					1270			1090			
TNVS (mg/L)					800			710			
TSS (mg/L)					90			65		68	
TNVSS (mg/L)					20			15			
% Solids											31.3
% Volatile Solids											83.2
BOD5 (mg/L)					282			15.0		18.5	
COD (mg/L)					580			450		110	
TOC (mg/L)					227			137		140	
TOC (% dry-wt basis)											43.6
NH3-N (mg/L)								0.27		0.46	
NO2+NO3-N (mg/L)								0.08		0.06	
Total-P (mg/L)								0.39		0.36	
Oil and Grease (mg/L)						41	6.5				
F-Coliform MF (#/100mL)						670	2200				
F-Coliform MPN (#/100mL)						240	1300				
% Klebsiella (KES)						76	23				
DOX (mg/L)			3.8J	4.9J		2.3J	3.2J				
Phenolics - Total (mg/L)		<0.005			2.99			<0.005			

FIELD OBSERVATIONS

Temperature (C)		32.7	31.2			31.2	33.7				
Temp - cooled (C)*					5.8			3.9		6.1	
pH (S.U.)		7.4	9.7	9.3	6.2	6.3	6.9			7.1	
Conductivity (umhos/cm)		1050	1350	1350	1170	1210	1340			1320	
Sulfide (mg/L)					**	**					
Chlorine (mg/L)		**	**		**	**					

* temperature of composite sample at the end of the sampling period

** color in sample prevented analysis due to interference with colorimetric field test.

Clorine residual was not suspected.

J estimated concentration

Acid acid stream from bleach plant

Inf influent to secondary treatment

Ef final effluent

Alk alkaline stream from bleach plant

S-comp Simpson composite sample

Table 1 - (cont'd) - Simpson, February 1991.

Location:	Alk-1	Alk-2	Alk-C	Acid-1	Acid-2	Acid-C	
Type:	grab	grab	comp	grab	grab	comp	
Date:	2/12	2/12	2/12-13	2/12	2/12	2/12-13	
Time:	1105	1455	0800-0800	1055	1450	0800-0800	
Parameter	Lab Log#:	078140	078141	078142	078143	078144	078145

LABORATORY RESULTS

Conductivity (umhos/cm)						
Alkalinity (mg/L CaCO3)						
Hardness (mg/L CaCO3)						
Color (C. U.)						
TS (mg/L)						
TNVS (mg/L)						
TSS (mg/L)			98			497
TNVSS (mg/L)						
% Solids						
% Volatile Solids						
BOD5 (mg/L)						
COD (mg/L)						
TOC (mg/L)			1120			846
TOC (% dry-wt basis)						
NH3-N (mg/L)						
NO2+NO3-N (mg/L)						
Total-P (mg/L)						
Oil and Grease (mg/L)						
F-Coliform MF (#/100mL)						
F-Coliform MPN (#/100mL)						
% Klebsiella (KES)						
DOX (mg/L)	22J	35J		11J	73J	
Phenolics - Total (mg/L)			0.155			0.059

FIELD OBSERVATIONS

Temperature (C)	67.5	69.1		57.8	58.8	
Temp - cooled (C)*			17.0			10.3
pH (S.U.)	10.3	9.8	10.3	2.1	1.8	1.6
Conductivity (umhos/cm)	4990	6130	5740	7820	9570	11330
Sulfide (mg/L)						
Chlorine (mg/L)	**	**		**	**	

Table 2 - NPDES Permit Comparison - Simpson, February 1991.

Parameter	Laboratory	NPDES Permit Limits *		Location:	Ef-1	Ef-2	Ef-C	Ef-Sim	Ef-GC
		Average	Maximum	Type:	grab	grab	E-comp	S-comp	gr-comp
		Monthly	Daily	Date:	2/12	2/12	2/12-13	2/12-13	2/12
				Time:	1205	1515	0800-0800	0800-0800	1205&1515
				Lab Log#:	078134	078135	078136	078138	078137
TSS (mg/L)									
	Ecology						65	68	
	Simpson						40	65.8	
	(lbs/D)	17200	33600						
	Ecology						16480	17240	
	Simpson						10141	16683	
BOD5 (mg/L)									
	Ecology						15.0	18.5	
	Simpson						22.3	21.7	
	(lbs/D)	8850	17200						
	Ecology						3803	4690	
	Simpson						5654	5502	
Soluble Copper (ug/L)									
		58	71						
	Ecology						15B	6.7JB	
pH (S.U.)									
		5.4 to 9.0							
	Ecology				6.2	6.3			
	Simpson				**	**			
Salmonid Bioassay									
		80% survival in 65% effluent							
	(% survival)								
	Ecology								100
Flow (MGD)***									
							30.4	30.4	

E-comp Ecology composite sample

S-comp Simpson composite sample

* limits from permit issued June 25, 1991

** pH on Simpson chart record varied between 6.2 and 6.4

*** flow data provided by Simpson

J estimated concentration

B the analyte was found in the method blank as well as the sample. The sample concentration is less than 10 times the blank concentration.

Ef final effluent

Table 3 - Target Analytes Detected by Priority Pollutant Scans - Simpson, February 1991.

Location:	Trns Blk	Inf-1	Inf-2	Alk-1	Alk-2	Acid-1	Acid-2
Type:	grab	grab	grab	grab	grab	grab	grab
Date:	2/11	2/12	2/12	2/12	2/12	2/12	2/12
Time:	1610	1025	1415	1105	1455	1055	1450
Lab Log#:	078130	078131	078132	078140	078141	078143	078144

VOA Compounds	(ug/L)						
Chloromethane	3.0 U	15 U	15 U	3.4	4.7 J	36	39 M
Methylene Chloride	12	10 U	10 U	5.0 U	4.1	7.5 J	15 U
Acetone	9.0	490	480	160	180	120	110
Carbon Disulfide	1.0 U	10 J	9.5 J	1.0 U	1.0 U	5.8 M	16 J
Chloroform	1.0 U	130	170	240	290	170	180
2-Butanone	5.0 U	300	280	55	51	30 U	33
4-Methyl-2-Pentanone	5.0 U	55	53	5.0 U	5.0 U	25 U	25 U
Toluene	1.0 U	4.6 M	4.1 M	1.0 U	1.0 U	5.0 U	5.0 U
Total Xylenes	2.0 U	10 U	10 U	2.0 U	2.0 U	10 U	10 U

Location:	Trns Blk	Inf-C	Alk-C	Acid-C	Sludge
Type:	grab	comp	comp	comp	gr-comp
Date:	2/11	2/12-13	2/12-13	2/12-13	2/12
Time:	1610	0800-0800	0800-0800	0800-0800	1400&1630
Lab Log#:	078130	078133	078142	078145	078139

BNA Compounds	(ug/L)	(ug/L)	(ug/L)	(ug/L)	(ug/Kg-dry wt)
Phenol	2 U	30		1.5 J	0.8 J 820 J
Benzyl Alcohol	5 U	16		1.4 M	2.4 M 2300 U
2,4-Dimethylphenol	2 U	3.8 M		2 U	2 U 940 U
Benzoic Acid	10 U	10 U		31	27 4700 U
2,4-Dichlorophenol	3 U	0.5 M		4.7 J	4.0 J 1400 U
Naphthalene	1 U	1 U		1 U	1 U 700 J
2-Methylnaphthalene	1 U	1.6 J		1 U	1 U 610 J
2,4,6-Trichlorophenol	5 U	5 U		3.6 J	2.0 J 2300 U
Diethyl Phthalate	1 U	1 U		0.9 J	1 U 470 U
Phenanthrene	1 U	0.9 M		1 U	1 U 420 J
Di-n-Octyl Phthalate	1 U	1 U		1 U	1.3 J 470 U

Metals - total recoverable		
Arsenic	1.5 U	2.1 JR
As (Pent)		
As (Tri)		
Cadmium	0.10 U	0.82 JR
Chromium	5.0 U	10 J
Cr (Hex)		
Cr (Tri)		
Copper	11 B	24.2
Lead	1.0 U	20.6
Mercury (total)	0.04 U	0.04 UR
Nickel	2.0 U	15
Zinc	4.0 U	37 J
Copper (dissolved)	3.5 JB	12 B

U indicates compound was analyzed for but not detected at the given detection limit.
 J indicates an estimated value.
 B This flag is used when the analyte is found in the method blank as well as the sample. Sample concentration is less than 10 times blank concentration.
 M indicates an estimated value of analyte found and confirmed by analyst but with low spectral match parameters.
 UJ indicates compound was analyzed for but not detected at the estimated detection limit.
 R low spike recovery - result may be biased low.
 (a) criteria for Total Halomethanes
 (i) criteria for Total Phthalate Esters
 (n) criteria for Total Polynuclear Aromatic Hydrocarbons
 * insufficient data to develop criteria. Value presented is LOEL - Lowest Observed Effect Level.
 + hardness dependent criteria (70 mg/L used)

Inf influent to secondary treatment
 Alk alkaline stream from bleach plant
 Acid acid stream from bleach plant
 Ef final effluent
 S-comp Simpson composite sample

Table 3 - (cont'd) - Simpson, February 1991.

Location:	Ef-1	Ef-2	EPA Water Quality Criteria Summary (EPA 1986)			
			Acute Fresh	Chronic Fresh	Acute Marine	Chronic Marine
Type:	grab	grab				
Date:	2/12	2/12				
Time:	1205	1515				
Lab Log#:	078134	078135				
VOA Compounds	(ug/L)	(ug/L)	(ug/L)	(ug/L)	(ug/L)	(ug/L)
Chloromethane	3.0 U	3.0 U	11000 *(a)		12000 *(a)	6400 *(a)
Methylene Chloride	5.0 U	4.3	11000 *(a)		12000 *(a)	6400 *(a)
Acetone	42	39				
Carbon Disulfide	1.7 M	1.6 M				
Chloroform	130	120	28900 *	1240 *	12000 *(a)	6400 *(a)
2-Butanone (MEK)	5.0 U	5.0 U				
4-Methyl-2-Pentanone (MIB)	5.0 U	5.0 U				
Toluene	0.6 M	0.9 J	17500 *		6300 *	5000 *
Total Xylenes	1.1 M	1.1 M				
Location:	Ef-C	Ef-Sim				
Type:	comp	S-comp				
Date:	2/12-13	2/12-13				
Time:	0800-0800	0800-0800				
Lab Log#:	078136	078138				
BNA Compounds	(ug/L)	(ug/L)				
Phenol	2 U		10200 *	2560 *	5800 *	
Benzyl Alcohol	5 U					
2,4-Dimethylphenol	0.9 J		2120 *			
Benzoic Acid	10 U					
2,4-Dichlorophenol	3 U		2020 *	365 *		
Naphthalene	1 U		2300 *	620 *	2350 *	
2-Methylnaphthalene	1 U					
2,4,6-Trichlorophenol	5 U			970 *		
Diethyl Phthalate	0.7 J		940 *(i)	3 *(i)	2944 *(i)	3.4 *(i)
Phenanthrene	1 U				300 *(n)	
Di-n-Octyl Phthalate	1 U		940 *(i)	3 *(i)	2944 *(i)	3.4 *(i)
Metals - total recoverable						
Arsenic	1.9 JR					
As (Pent)			850 *	48 *	2319 *	13 *
As (Tri)			360	190	69	36
Cadmium	0.69 JR		2.6 +	0.9 +	43	9.3
Chromium	6.6 J					
Cr (Hex)			16	11	1100	50
Cr (Tri)			1297 +	155 +	10300 *	
Copper	16 B		13 +	8.7 +	2.9	2.9
Lead	14.5		52 +	2.0 +	140	5.6
Mercury (total)	0.07 JR		2.4	0.012	2.1	0.025
Nickel	12		1049 +	117 +	75	8.3
Zinc	11 J		87 +	78 +	95	86
Copper (dissolved)	15 B	6.7 JB				

U indicates compound was analyzed for but not detected at the given detection limit.
 J indicates an estimated value.
 B This flag is used when the analyte is found in the method blank as well as the sample. Sample concentration is less than 10 times blank concentration.
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Inf influent to secondary treatment
 Alk alkaline stream from bleach plant
 Acid acid stream from bleach plant
 Ef final effluent
 S-comp Simpson composite sample

A few more BNA/VOA organics were observed in the influent sample than in the effluent sample. Acetone (480-490 $\mu\text{g/L}$), 2-butanone (280-300 $\mu\text{g/L}$), and chloroform (130-170 $\mu\text{g/L}$) were found at the highest concentrations. Most BNA/VOA organics found in the influent were found in the effluent at lower concentrations or less than detection limits. The exception was chloroform, which had similar influent and effluent concentrations.

Several base-neutral acid extractable (BNA) compounds were found in the sludge sample. Concentrations are reported as estimated values because they were less than the concentration required for accurate quantitation.

Organics detected at the highest concentrations in the bleach plant streams included acetone (110-180 $\mu\text{g/L}$) and chloroform (170-290 $\mu\text{g/L}$). Organic concentrations in the alkaline stream were generally slightly greater than concentrations in the acid stream.

A complete list of analytes and detection limits is included in Appendix D.

Tentatively identified compounds (TICs) were also detected in several of the samples (Appendix E). Estimated influent concentrations of TICs ranged from 28-2800 $\mu\text{g/L}$ for VOAs and 46-480 $\mu\text{g/L}$ for BNAs. Estimated effluent concentrations of TICs ranged from 5-10 $\mu\text{g/L}$ for VOAs and 11-75 $\mu\text{g/L}$ for BNAs. Estimated alkaline bleach plant stream concentrations for VOAs ranged from 6-12 $\mu\text{g/L}$. No TICs were detected in the acid bleach plant stream.

Guaiacols/Catechols and Resin Acids/Fatty Acids

The guaiacol/catechol scan found several compounds present in the influent sample (Table 4). Guaiacol (2-methoxyphenol: 14000 $\mu\text{g/L}$) and α -terpeneol (3300 $\mu\text{g/L}$) were present in the highest concentrations. The secondary treatment process appeared effective in removing compounds present in the influent. 4,5-Dichlorocatechol (10 $\mu\text{g/L}$) was the compound found in the highest concentration in the effluent.

Seven resin acid/fatty acid compounds were found at concentrations ranging from 180-520 $\mu\text{g/L}$ in the influent (Table 4). Secondary treatment apparently reduced concentrations; the highest estimated concentration found in the effluent was 34 $\mu\text{g/L}$. Effluent concentrations for three of the compounds found among those with the highest concentrations were well below available LC_{50} data for salmon (Verschueren, 1983):

Compound	Estimated effluent concentration	LC_{50} (Verschueren, 1983)
Abietic acid	34 $\mu\text{g/L}$	410 $\mu\text{g/L}$
Dehydroabietic acid	25 $\mu\text{g/L}$	500-1760 $\mu\text{g/L}$
Isopimaric acid	29 $\mu\text{g/L}$	220 $\mu\text{g/L}$

Table 4 - Guaiacols/Catechols and Resin Acids/Fatty Acids Results - Simpson, February 1991.

Location:	Trns Blk	Inf-C	Ef-C	Matrix	Matrix
Type:	grab	comp	comp	Spike #1	Spike #2
Date:	2/11	2/12-13	2/12-13		
Time:	1610	0800-0800	0800-0800		
Lab Log#:	078130	078133	078136	078133	078133
GUAIACOLS/CATECH	ug/L	ug/L	ug/L	% recovery	% recovery
4-Chloro-3-Methylpheno	0.5 U	1 U	0.8 U	102 %	95 %
Pentachlorophenol	0.5 U	1 U	0.8 U	96 %	94 %
2,4,6-Trichlorophenol	0.5 U	1 U	0.8 U	104 %	98 %
2-Nitrophenol	0.5 U	1 U	0.8 U	116 %	118 %
Guaiacol (2-methoxyphen	0.5 U	14000	0.6 J		
2-Methylphenol	0.5 U	1 U	0.8 U	128 %	104 %
o-Chlorophenol	0.5 U	1 U	0.8 U	98 %	79 %
2,4,5-Trichlorophenol	0.5 U	1 U	0.8 U	100 %	90 %
4-Allylguaiacol (eugenol)	0.5 UJ	42 J	0.8 UJ	56 %	22 %
4-Propenylguaiacol	0.5 U	3	0.8 U	95 %	80 %
4-Nitrophenol	0.5 U	1 U	0.8 U	452 %	112 %
2,4-Dimethylphenol	0.5 U	1 U	1	119 %	104 %
4-Methylphenol	0.5 U	1 U	0.8 U	108 %	94 %
Phenol	0.9 U	76	1 U		
2,4-Dichlorophenol	0.5 U	1 U	0.8 U	105 %	90 %
2,3,6-Trichlorophenol	0.5 U	1 U	0.8 U	108 %	92 %
Tetrachloroguaiacol	0.5 U	1 U	0.8 U	83 %	90 %
Tri-Cl-tri-MeO-benzene	0.5 U	1 U	0.8 U	97 %	88 %
Tetrachlorocatechol	0.5 U	1 U	0.8 U	50 %	67 %
4-Chlorocatechol	0.5 U	1 U	0.8 U	85 %	82 %
4,5-Dichloroguaiacol	0.5 U	32	2	56 %	46 %
Trichlorosyringol	0.5 U	1 U	0.8 U	92 %	89 %
4,5,6-Trichloroguaiacol	0.5 U	2	1	99 %	96 %
4,5-Dichlorocatechol	0.5 U	2	10	104 %	101 %
a-Terpeneol	0.5 U	3300	0.8 U		
2,3,4-Trichlorophenol	0.5 U	1 U	0.8 U	98 %	95 %
4-Chloroguaiacol	0.5 U	1 U	0.8 U	91 %	86 %
5,6-Dichlorovanillin	0.5 UJ	11 J	1 J	145 %	148 %
6-Chlorovanillin	0.5 U	58	4	64 %	22 %
3,4,5-Trichlorocatechol	0.5 U	1 J	0.4 J		
3,4,5-Trichloroguaiacol	0.5 U	5	3	90 %	81 %
Surrogates (% recovery):					
2-Ethoxyphenol	89 %	110 %	96 %	116 %	100 %
2,4,6-Tribromo+	88 %	99 %	96 %	99 %	88 %
2-Fluorobiphen+	86 %	85 %	77 %	76 %	62 %
2-Fluorophenol	71 %	84 %	51 %	72 %	59 %
2,6-Dibromophe+ *	101 %	46 %	106 %	93 %	98 %
D5-Nitrobenzene	73 %	111 %	56 %	97 %	73 %
D5-Phenol	70 %	90 %	50 %	82 %	72 %
RESIN ACIDS/FATTY ACIDS					
Decanoic Acid, Hexa-	2 U	140	32 U		
Octadecanoic acid	1 U	33 U	13 U		
Linoleic acid	0.5 U	270	9 U		
Oleic acid	0.5 U	300	9 U		
Pimaric acid	0.5 U	110 J	7 J		
Palmitoleic acid	0.5 U	15 U	9 U	132 %	108 %
Sandaracopimaric acid	0.5 U	440	26 J		
Neobietic Acid	0.5 U	130	11 J		
Retene	0.5 U	15 U	9 U	77 %	88 %
Abietic acid	0.5 U	430	34 J		
14-Chlorodehydroabietic	0.5 U	15 U	9 U	122 %	118 %
12-Chlorodehydroabietic	0.5 U	15 U	7 J	134 %	124 %
Dehydroabietic acid	0.5 U	520	25 J		
Palustric acid	0.5 U	180	13 J		
Dichlorostearic Acid	0.5 U	29 J	9 U		
Isopimaric acid	0.5 U	320	29 J		
Dichlorodehydroabietic A	0.5 U	15 U	9 U	92 %	89 %
Surrogates (% recovery):					
Et-o-Methylpod+	10 %	30 %	18 %	94 %	91 %
d31-Hexadecanoic aci	9 %	17 %	16 %	57 %	70 %

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

J Indicates an estimated value.

UJ Indicates compound was analyzed for but not detected at the estimated quantitation limit.

* internal standard

Inf influent to secondary treatment

Ef final effluent

Dioxins/Furans

The Ecology dioxin/furan analytical results were not as expected (Table 5). Concentrations found by Ecology in the bleach plant effluent, the area of dioxin/furan generation, were less than concentrations found in the secondary treatment plant influent and effluent. As was expected, concentrations in the treatment plant effluent were less than in the influent.

The explanation for higher concentrations in the treatment plant than in the bleach plant is unclear. The laboratory double checked and found no apparent problems or confusion with sample handling or labelling. The only unusual operating condition during the inspection was a general plant clean-up in preparation for a Simpson management visit.

Simpson dioxin/furan analysis was limited to analysis of 2,3,7,8-TCDD and 2,3,7,8-TCDF in the Ecology effluent composite sample. The Simpson analysis did not detect 2,3,7,8-TCDD (< 1.9 pg/L) and detected a 2,3,7,8-TCDF concentration of 15 pg/L. The concentrations were less than the Ecology analytical results (2,3,7,8-TCDD - 79 pg/L; 2,3,7,8-TCDF - 71 pg/L). Additional splits for dioxin/furan analysis should be made for Ecology and Simpson analysis.

Several dioxin/furan compounds were also detected in the sludge (Table 5). 2,3,7,8-TCDD was found in the highest concentration - 239000 pg/Kg-dry wt basis (0.239 μ g/Kg-dry wt basis). The sludge serves as a partial source of fuel for the boiler. Analysis of the boiler ash for dioxin/furan compounds may provide useful information.

Bioassays

Rainbow trout (*Oncorhynchus mykiss*) and *Daphnia pulex* survival tests in the Simpson effluent found no acute toxicity (Table 6). Fish behavior during the rainbow trout test was erratic at the beginning of the test, "each fish was spinning on its tail and gulping at the surface" (Noble, 1991). The erratic behavior stopped within 12 hours and test survival was not affected.

Microtox[®] results were also interesting: the 5-minute EC₅₀ (34.6% effluent) was less than the 15-minute EC₅₀ (53.3% effluent). The data suggest a possible fast-acting sub-lethal toxicant to which the organisms were able to recover over time (Stinson, 1991).

Survival of the fathead minnow (*Pimephales promelas*) and blue mussel larvae (*Mytilus edulis*) was not significantly affected by the effluent. Both test organisms showed some sensitivity to the effluent in the chronic portion of the test. The no observed effect concentration (NOEC) in the chronic portion of the fathead minnow test was 50% effluent. The NOEC in the chronic portion of the blue mussel larvae test was 3.125% effluent.

The echinoderm (*Strongylocentrotus purpuratus*) sperm cell test found a NOEC of 12.5% effluent. The significance of the observation is confused by the behavior of the salinity control. The salinity control NOEC was 25% dilution water, but percent fertilized eggs dropped from 80.3% at 12.5% dilution water to 43.7% at 25% dilution water. Although the salinity control

Table 5 - Dioxin/Furan Results - Simpson, February 1991.

Location:	Trns Blk	Inf-C	Ef-C	Alk-C	Acid-C	Duplicate	Sludge	Spike &
Type:	grab	comp	comp	comp	comp	Analysis	gr-comp	Spike
Date:	2/11	2/12-13	2/12-13	2/12-13	2/12-13		2/12	Duplicate
Time:	1610	0800-0800	0800-0800	0800-0800	0800-0800		1400&1630	
Lab Log#:	078130	078133	078136	078142	078145	078145	078139	078133
	(pg/L)	(pg/L)	(pg/L)	(pg/L)	(pg/L)	(pg/L)	(pg/Kg - dry wt)	***
TCDD (total)	1 U	552	459	1 U	1 U	1 U	239000	
2,3,7,8-TCDD	1 U	85	79	1 U	1 U	1 U	239000	113 (103) - 9.2
PCDD (total)	1 U	623	530	45	1 U	1 U	6430 U	
1,2,3,7,8-PCDD	1 U	10	7	5	1 U	1 U	6430 U	103 (110) - 6.6
HxCDD (total)	1 U	931	706	1390	119	117	1675000	
1,2,3,4,7,8-HxCDD	1 U	15	8	61	5	5	5230 UJ	104 (105) - 0.9
1,2,3,7,8,9-HxCDD	1 U	15	8	61	5	5	5230 UJ	104 (105) - 0.9
1,2,3,6,7,8-HxCDD	1 U	32	26	102	5	5	5230 UJ	101 (109) - 7.6
HpCDD (total)	2 U	297	276	185	21	23	260000	
1,2,3,4,6,7,8-HpCDD	2 U	162	142	53	6	7	167000	103 (106) - 2.9
OCDD (total)	38 *	219	190	207	73	78	227000	101 (106) - 4.8
TCDF (total)	1 U	349	283	11	1 U	1 U	88600	
2,3,7,8-TCDF	1 U	81	71	11	1 U	1 U	88600	103 (106) - 2.9
PCDF (total)	1 U	226	158	1 U	1 U	1 U	3790 U	
1,2,3,7,8-PCDF	1 U	7	7	1 U	1 U	1 U	3790 U	105 (111) - 5.6
2,3,4,7,8-PCDF	1 U	23	39	1 U	1 U	1 U	3790 U	99 (105) - 5.9
HxCDF (total)	1 U	83	43	1 U	2	2	1880 U	
1,2,3,4,7,8-HxCDF	1 U	14	12	1 U	1 U	1 U	1880 U	112 (101) - 10
1,2,3,7,8,9-HxCDF	1 U	14	12	1 U	1 U	1 U	1880 U	112 (101) - 10
1,2,3,6,7,8-HxCDF	1 U	16	15	1 U	2	2	1880 UJ	108 (104) - 3.8
2,3,4,6,7,8-HxCDF	1 U	1 U	3 U	1 U	1 U	1 U	1880 UJ	99 (111) - 11.4
HpCDF (total)	1 U	25	22	4 U	1 U	1 U	4050 U	
1,2,3,4,6,7,8-HpCDF	1 U	21	17	4 U	1 U	1 U	4050 U	104 (110) - 5.6
1,2,3,4,7,8,9-HpCDF	1 U	2	1	4 U	1 U	1 U	4050 U	100 (105) - 4.9
OCDF (total)	1 U	7	7	3	1	1	7300 U	94 (107) - 12.9

U indicates analyte was analyzed for but not detected at the reported result.
 UJ indicates analyte was analyzed for but not detected at the estimated result.
 * detected in the water method blank at 26 pg/L and in the sludge method blank at 7400 pg/Kg -dry wt. No other compounds were detected in the method blank.
 *** matrix spike % recovery (matrix spike duplicate % recovery) - relative percent difference

Inf influent to secondary treatment
 Alk alkaline stream from bleach plant
 Acid acid stream from bleach plant
 Ef final effluent

Table 6 - Effluent Bioassay Results - Simpson, February 1991.

NOTE: all tests were run on the final effluent (Ef-GC sample) - lab log # 078137

Daphnia pulex - 48 hour survival test
(*Daphnia pulex*)

Sample	# Tested *	Percent Survival
Control	20	95
100 % Effluent	20	90

Acute
LC50 = >100 % effluent
LOEC = >100 % effluent

* 4 replicates of 5 organisms

Bivalve Larvae - 48 hour survival and development test
blue mussel (*Mytilus edulis*)

Sample +	Average % Survival *	Average % Abnormal **
Control	86.7	16.0
Brine control	83.5	17.7
1.0 % Effluent	100.0	15.7
3.125 % Effluent	100.0	20.3
6.25 % Effluent	93.5	81.7
12.5 % Effluent	91.2	93.3
25 % Effluent	87.6	93.0
50 % Effluent	90.9	98.7

<u>Acute</u>	<u>Chronic</u>
LC50 = >50 % effluent	NOEC = 3.125 % effluent
NOEC = >50 % effluent	LOEC = 6.25 % effluent
LOEC = >50 % effluent	

* average of 3 replicates

** average of 3 replicates - 100 organisms counted per replicate

+ salinity of all tests adjusted to 35 o/oo

Rainbow Trout - 96 hour survival test
(*Oncorhynchus mykiss*)

Sample	# Tested	Percent Survival
Control	30	100
65% Effluent	30	100 *

* upon introduction to the effluent, fish behaved erratically. Normal behavior resumed within 12 hours (Noble, 1991).

Microtox

	EC50 (% effluent)	Ranking *
5 minutes	34.6	moderate
15 minutes	53.3	moderate
15 minutes **	59.9	moderate

* priority ranking for further toxicity evaluation based on the EC50 (EPA, 1980)

** color corrected

Table 6 - (cont'd) - Simpson, February 1991.

Echinoderm Sperm Cell Toxicity Test
(*Strongylocentrotus purpuratus*)

Sample + Concentration	% Fertilized Eggs *	
	Salinity Control **	Effluent **
Seawater control	53.3	
0.8 % Sample	73.0	72.7
1.6 % Sample	76.3	81.0
3.1 % Sample	77.3	80.7
6.3 % Sample	74.0	86.3
12.5 % Sample	80.3	80.7
25 % Sample	43.7	0.0
50 % Sample	1.7	0.0
	EC50 = 22 % effluent	EC50 = 16.5 % effluent
	NOEC = 25 % effluent	NOEC = 12.5 % effluent
	LOEC = 50 % effluent	LOEC = 25 % effluent

* average of 3 replicates

** salinity adjusted with filtered Clam Bay water. Salinity adjustment was first attempted with hypersaline brine, but control fertilization was insufficient for a valid test.

Fathead Minnow - 7 day survival and growth test
(*Pimephales promelas*)

Sample	# Tested *	Percent Survival	Average Growth per Fish (mg)
Control	60	92	0.44
1.56 % Effluent	60	92	0.42
3.12 % Effluent	60	100	0.40
6.25 % Effluent	60	92	0.40
12.5 % Effluent	60	98	0.43
25 % Effluent	60	95	0.40
50 % Effluent	60	95	0.39
100 % Effluent	60	88	0.28
		<u>Acute</u>	<u>Chronic</u>
		LOEC = >100 % effluent	NOEC = 50 % effluent
		LC50 = >100 % effluent	LOEC = 100 % effluent

* four replicates of 15 organisms

NOEC - no observable effects concentration
LOEC - lowest observable effects concentration
LC50 - lethal concentration for 50% of the organisms
EC50 - effect concentration for 50% of the organisms

fertilization was depressed at 25% dilution water, it was not significant compared to the seawater control (Stinson, 1991a). It appears that small differences in salinity could have been important in determining fertilization at the 25% dilutions.

Laboratory Review/Split Samples

The laboratory procedures review found the Simpson BOD₅ and TSS procedures to be acceptable (Appendix F). The Simpson and Ecology effluent composite sample characteristics were very similar for most parameters, indicating both samples were acceptable. Ecology and Simpson analytical results of the split samples compared well for both of the samples analyzed for BOD₅ and one of the two samples analyzed for TSS (Table 7). The Simpson continuous effluent pH monitoring closely approximated Ecology grab sample results.

Simpson continuous effluent temperature monitoring was 2 to 4 degrees higher than the Ecology measurements. Occasional checks of the continuous monitor with a calibrated thermometer are suggested.

Although Ecology and Simpson DOX and chloroform samples were collected at different times during the inspection, results were in the same range.

RECOMMENDATIONS AND CONCLUSIONS

General Chemistry/NPDES Permit Limits

Permit parameters were within daily average limits during the inspection. The effluent total suspended solids load approximated the monthly average permit limit while the BOD₅ load was much less than the monthly average limit. The secondary treatment process provided good BOD₅ removal. One of the two grab samples collected for oil and grease analysis had a higher than expected concentration (41 mg/L).

- Monitoring to establish the typical effluent oil and grease concentration should be considered to determine if permit limits are necessary.

Base-Neutral Acid Extractable (BNA), Volatile Organic Analysis (VOA), and Metals Priority Pollutant Scans

Most organics observed in the influent BNA and VOA scans decreased in concentration through the secondary treatment process. The exception was chloroform which appeared at nearly the same concentrations in the influent and effluent. The few BNA/VOA organics detected in the effluent were at concentrations less than toxicity criteria. Effluent metals concentrations were low, although nickel, lead, mercury, and copper concentrations exceeded one or more of the chronic and/or acute toxicity criteria.

Table 7 - Split Sample Results - Simpson, February 1991.

	Location:	Inf-1	Inf-2	Ef-1	Ef-2	Ef-C	Ef-Sim	Alk-1	Alk-2	Acid-1	Acid-2
Parameter	Type:	grab	grab	grab	grab	comp	S-comp	grab	grab	grab	grab
	Date:	2/12	2/12	2/12	2/12	2/12-13	2/12-13	2/12	2/12	2/12	2/12
Laboratory	Time:	1025	1415	1205	1515	0800-0800	0800-0800	1105	1455	1055	1450
	Lab Log#:	078131	078132	078134	078135	078136	078138	078140	078141	078143	078144
TSS (mg/L)											
Ecology						65	68				
Simpson						40	65.8				
BOD5 (mg/L)											
Ecology						15.0	18.5				
Simpson						22.3	21.7				
DOX (mg/L)											
Ecology		3.8J	4.9J	2.3J	3.2J			22J	35J	11J	73J
Simpson**			5.1+		3.6+				60+		110+
Chloroform (ug/L)											
Ecology		130	170	130	120			240	290	170	180
Simpson**			210**		150**				600**		360**
Temperature (C)											
Ecology				31.2	33.7						
Simpson				*	*						
pH (S.U.)											
Ecology				6.2	6.3						
Simpson				**	**						

- * temperature on Simpson chart record was 96 degrees F (35.6 degrees C)
- ** pH on Simpson chart record varied between 6.2 and 6.4
- *+ Simpson analysis done by a contract laboratory
- + Simpson sample was a grab composite. The first half was collected on 2/12 along with the second Ecology grab sample and the second half was collected on 2/13.
- ** Simpson samples for chloroform analysis collected on 2/13 between 1130 and 1330.
- J estimated concentration
- Inf influent to secondary treatment
- Alk alkaline stream from bleach plant
- Acid acid stream from bleach plant
- Ef final effluent
- S-comp Simpson composite sample

Guaiacols/Catechols and Resin Acids/Fatty Acids

Guaiacol/catechol group members present in the influent were near or below detection limits in the effluent. Resin acid/fatty acid group members present in the influent were also found at substantially lower concentrations in the effluent. Both groups appeared amenable to secondary treatment.

Dioxins/Furans

Dioxin/furan concentrations were higher in the treatment system samples than in the bleach plant samples. The reason is unclear. 2,3,7,8-TCDD was detected by the Ecology analysis of the Ecology effluent sample, but was not detected by the Simpson analysis of the Ecology effluent sample.

- Additional sample splits for Ecology and Simpson analysis are recommended. The Ecology data appear unusual, but should be given more weight if similar observations occur in the future.

Dioxin/furan compounds were also detected in the sludge.

- Because the sludge is burned as part of the boiler fuel supply, analysis of the boiler ash for dioxin/furan compounds may provide useful information.

Bioassays

Rainbow trout, *Daphnia pulex*, fathead minnow, and blue mussel bioassays demonstrated no acute toxicity in the Simpson effluent. Acute toxicity was observed only in the echinoderm sperm cell bioassay, but stress due to salinity may have been a factor. Fathead minnow (NOEC 50% effluent) and blue mussel (NOEC 3.125% effluent) demonstrated some chronic toxicity in the effluent.

Erratic behavior by the rainbow trout at the beginning of the test suggested a possible sub-lethal toxicant in the effluent. Microtox® results also suggested a possible sub-lethal toxicant in the effluent.

- Requesting that observations of fish behavior be submitted along with survival data for the permit required salmonid tests is recommended.

Laboratory Review/Split Samples

Simpson laboratory procedures, sampling procedures, and BOD₅ and TSS split sample analytical results were acceptable.

- Occasional checks of the continuous effluent temperature monitor with a known accurate thermometer are recommended.

REFERENCES

- EPA, 1986. Quality Criteria for Water, EPA 440/5-86-001.
- Noble, Scott, 1991. "Simpson Results of Salmonid Bioassay," Washington State Department of Ecology memo to Marc Heffner dated February 25, 1991.
- Stinson, Margaret, 1991. "Simpson Paper Class II Inspection Results of Microtox® Bioassay," Washington State Department of Ecology memo to Marc Heffner dated March 5, 1991.
- Stinson, Margaret, 1991a. "Simpson Paper Class II Inspection Results of Echinoderm Sperm Cell Toxicity Test," Washington State Department of Ecology memo to Marc Heffner dated March 20, 1991.
- Verschueren, Karel, 1983. Handbook of Environmental Data on Organic Chemicals, second edition. Van Norstrand Reinhold Company, New York. 1,310 pp.

APPENDICES

Appendix A – Sampling Schedule and Parameters Analyzed – Simpson, February 1991.

Parameter	Location:	Trns Blk	Inf-1	Inf-2	Inf-C	Ef-1	Ef-2	Ef-C	Ef-GC	Ef-Sim	Sludge
	Type:	grab	grab	grab	comp	grab	grab	comp	gr-comp	S-comp	gr-comp
	Date:	2/11	2/12	2/12	2/12-13	2/12	2/12	2/12-13	2/12	2/12-13	2/12
	Time:	1610	1025	1415	0800-0800	1205	1515	0800-0800	1205&1515	0800-0800	1400&1630
	Lab Log#:	078130	078131	078132	078133	078134	078135	078136	078137	078138	078139
GENERAL CHEMISTRY											
Conductivity						E			E	E	E
Alkalinity						E			E	E	E
Hardness						E			E	E	E
Color						E			E	E	E
TS						E			E	E	E
TNVS						E			E	E	E
TSS						E			E	E	E
TNVSS						E			E	E	E
% Solids											E
% Volatile Solids											E
BOD5						E			E	E	E
COD						E			E	E	E
TOC (water)						E			E	E	E
TOC (soil)											E
NH3-N									E	E	E
NO2+NO3-N									E	E	E
Total-P									E	E	E
Oil and Grease						E			E	E	E
F-Coliform MF						E			E	E	E
F-Coliform MPN						E			E	E	E
% Klebsiella (KES)						E			E	E	E
ORGANICS											
DOX			E	ES**		E	ES**				
VOA (water)		E	E	ES*		E	ES*				
BNAs (water)		E			E			E			
BNAs (soil)											E
Resin/Fatty Acids (eff)		E			E			E			
Guaiacols (effluent)		E			E			E			
Phenolics Total(water)		E			E			E			
Dioxin/Furans		E			E			E			E
METALS											
PP Metals		E			E			E			E
Soluble Cu		E			E			E			E
BIOASSAYS											
Salmonid (acute)									E		
Microtox (acute)									E		
Daphnia pulex (acute)									E		
Fathead Minnow (chronic)									E		
Bivalve Larvae									E		
Echinoderm sperm cell									E		
FIELD OBSERVATIONS											
Temperature			E	E	E	ES	ES	E		E	
pH			E	E	E	ES	ES	E		E	
Conductivity			E	E	E	ES	ES	E		E	
Sulfide						+	+				
Chlorine			+	+		+	+				

* Simpson VOA sample for chloroform analysis only. Simpson samples were collected on 2/13 between 1130 and 1330.
 ** Simpson sample was a grab composite. The first half was collected on 2/12 along with the second Ecology grab sample and the second half was collected on 2/13.
 *+ color in sample prevented analysis due to interference with colorimetric field test
 E Ecology laboratory analysis
 S Simpson laboratory analysis

Inf influent to secondary treatment
 Alk alkaline stream from bleach plant
 Acid acid stream from bleach plant
 Ef final effluent
 S-comp Simpson composite sample

Appendix A – (cont'd) – Simpson, February 1991.

Parameter	Location:	Alk-1	Alk-2	Alk-C	Acc-1	Acc-2	Acc-C
	Type:	grab	grab	comp	grab	grab	comp
	Date:	2/12	2/12	2/12-13	2/12	2/12	2/12-13
	Time:	1105	1455	0800-0800	1055	1450	0800-0800
	Lab Log#:	078140	078141	078142	078143	078144	078145
GENERAL CHEMISTRY							
Conductivity							
Alkalinity							
Hardness							
Color							
TS							
TNVS							
TSS				E			E
TNVSS							
% Solids							
% Volatile Solids							
BOD5							
COD							
TQC (water)				E			E
TOC (soil)							
NH3-N							
NO2+NO3-N							
Total-P							
Oil and Grease							
F-Coliform MF							
F-Coliform MPN							
% Klebsiella (KES)							
ORGANICS							
DOX		E	ES**		E	ES**	
VOA (water)		E	ES*		E	ES*	
BNAs (water)				E			E
BNAs (soil)							
Resin/Fatty Acids (eff)							
Guaiacols (effluent)							
Phenolics Total(water)				E			E
Dioxin/Furans				E			E
METALS							
PP Metals							
Soluble Cu							
BIOASSAYS							
Salmonid (acute)							
Microtox (acute)							
Daphnia pulex (acute)							
Fathead Minnow (chronic)							
Bivalve Larvae							
Echinoderm sperm cell							
FIELD OBSERVATIONS							
Temperature		E	E	E	E	E	E
pH		E	E	E	E	E	E
Conductivity		E	E	E	E	E	E
Sulfide							
Chlorine		*+	*+		*+	*+	

Appendix B – Ecology Laboratory Methods – Simpson, February 1991.

<u>Parameter</u>	<u>Method</u>	<u>Laboratory</u>
GENERAL CHEMISTRY		
Conductivity	EPA, 1979: 120.1	Ecology
Alkalinity	EPA, 1979: 310.1	Ecology
Hardness	EPA, 1979: 130.2	Ecology
Color	EPA, 1979: 110.1	AMTEST
TS	EPA, 1979: 160.3	Ecology
TNVS	EPA, 1979: 160.3	Ecology
TSS	EPA, 1979: 160.2	Ecology
TNVSS	EPA, 1979: 160.2	Ecology
% Solids	EPA, 1979: 160.3	AMTEST
% Volatile Solids	EPA, 1979: 160.4	AMTEST
BOD ₅	EPA, 1979: 405.1	Ecology
COD	EPA, 1979: 410.1	AMTEST
TOC (water)	EPA, 1979: 415.1	Ecology
TOC (soil)	APHA, 1989: 5310	AMTEST
NH ₃ -N	EPA, 1979: 350.1	Ecology
NO ₂ +NO ₃ -N	EPA, 1979: 353.2	Ecology
Total-P	EPA, 1979: 365.3	Ecology
Oil and Grease	EPA, 1979: 413.1	AMTEST
F-Coliform MF	APHA, 1989: 9222D	Ecology
F-Coliform MPN	APHA, 1989: 9221C	Ecology
% Klebsiella (KES)	Manchester SOP	Ecology
ORGANICS		
DOX	APHA, 1989: 5320	Laucke
VOA (water)	EPA, 1984: 824	ARI
BNAs (water)	EPA, 1984: 825	ARI
BNAs (soil)	EPA, 1986: 8270	ARI
Resin/Fatty Acids (water)	NCASI, 1986a	Ecology
Guaiacols (water)	NCASI, 1986b	Ecology
Phenolics Total(water)	EPA, 1979: 420.2	AMTEST
Dioxin/Furans	NCASI, 1989	Weyerhaeuser
METALS		
PP Metals	EPA, 1979: 200	Ecology
Soluble Cu	EPA, 1979: 4.1.1	Ecology
BIOASSAYS		
Salmonid (acute)	Ecology, 1981	Ecology
Microtox (acute)	Beckman, 1982	Ecology
Daphnia pulex (acute)	EPA, 1985a	Ecology
Fathead Minnow (chronic)	EPA, 1989	Ecology
Bay Mussel	ASTM, 1986	ERC
Echinoderm sperm cell	Dinnel, 1987	Ecology

AMTEST AmTest, Inc.
ARI Analytical Resources, Inc.
Ecology Ecology Manchester Laboratory
ERC ERC Environmental and Energy Services Company, Inc.
Laucke Laucke Testing Laboratories, Inc.
Weyerhaeuser Weyerhaeuser Analytical and Testing Services

- APHA-AWWA-WPCF, 1989. Standard Methods for the Examination of Water and Wastewater, 17th ed.
- ASTM, 1986. Standard Practice for Conducting Static Acute Toxicity Tests with Larvae of Four Species of Bivalve Mollusks. pp. 368-384. In: Annual Book of ASTM Standards, Water and Environmental Technology, Volume 11.04. American Society for Testing and Materials, Philadel. Pa.
- Beckman Instruments, Inc., 1982. Microtox System Operating Manual.
- Dinnel, P.A., et.al, 1987. Improved Methodology for a Sea Urchin Sperm Cell Bioassay for Marine Waters. Arch. Environ. Contam. Toxicol., 16, 23-32.
- Ecology, 1981. Static Acute Fish Toxicity Test, DOE 80-12, revised July 1981.
- EPA, 1979. Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020 (Rev. March, 1983).
- EPA, 1984. 40 CFR Part 136, October 26, 1984.
- EPA, 1985a. Methods for Measuring the Acute Toxicity of Effluents to Freshwater and Marine Organisms. EPA/600/4-85/013.
- EPA, 1986. Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846, 3rd. ed., November, 1986.
- EPA, 1989. Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving waters to Freshwater Organisms. Second edition. EPA/600/4-89/001.
- NCASI, 1986a. Procedures for Analysis of Resin and Fatty Acids in Pulp Mill Effluents. Tech. Bull. no. 501. National Council of Paper Industry for air and Stream Improvement Inc., New York, NY.
- NCASI, 1986b. Methods for the Analysis of Chlorinated Phenolics in Pulp Industry Wastewater, Tech. Bull. no. 498. National Council of Paper Industry for air and Stream Improvement Inc., New York, NY.
- NCASI, 1989. NCASI Procedures for the Preparation and Isomer Specific Analysis of Pulp and Paper Industry Samples for 2,3,7,8-TCDD and 2,3,7,8-TCDF, NCASI Technical Bulletin No. 551.

Appendix C - Priority Pollutant Cleaning and Field Transfer Blank
Procedures - Simpson, February 1991.

PRIORITY POLLUTANT SAMPLING EQUIPMENT CLEANING PROCEDURES

1. Wash with laboratory detergent.
2. Rinse several times with tap water.
3. Rinse with 10% HNO₃ solution.
4. Rinse three (3) 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 (VOA).
2. Run approximately 1L of organic free water through a compositor and discard.
3. Run approximately 6L of organic-free water through the same compositor and put the water into appropriate bottles for parameters to be analyzed from composite samples (BNA, Pesticide/PCB, resin acids, guaiacols, dioxins, phenolics, and metals).

Appendix D – VOA, BNA, and Metals Scan Results – Simpson, February 1991.

Location:	Trns Blk	Inf-1	Inf-2	Ef-1	Ef-2	Alk-1	Alk-2	Acc-1	Acc-2
Type:	grab	grab	grab	grab	grab	grab	grab	grab	grab
Date:	2/11	2/12	2/12	2/12	2/12	2/12	2/12	2/12	2/12
Time:	1610	1025	1415	1205	1515	1105	1455	1055	1450
Lab Log#:	078130	078131	078132	078134	078135	078140	078141	078143	078144
VOA Compounds	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L
Chloromethane	3.0 U	15 U	15 U	3.0 U	3.0 U	3.4	4.7 J	36	39 M
Bromomethane	3.0 U	15 U	15 U	3.0 U	3.0 U	3.0 U	3.0 U	15 U	15 U
Vinyl Chloride	3.0 U	15 U	15 U	3.0 U	3.0 U	3.0 U	3.0 U	15 U	15 U
Chloroethane	3.0 U	15 U	15 U	3.0 U	3.0 U	3.0 U	3.0 U	15 U	15 U
Methylene Chloride	12	10 U	10 U	5.0 U	4.3	5.0 U	4.1	7.5 J	15 U
Acetone	9.0	490	480	42	39	160	180	120	110
Carbon Disulfide	1.0 U	10 J	9.5 J	1.7 M	1.6 M	1.0 U	1.0 U	5.8 M	16 J
1,1-Dichloroethene	1.0 U	5.0 U	5.0 U	1.0 U	1.0 U	1.0 U	1.0 U	5.0 U	5.0 U
1,1-Dichloroethane	1.0 U	5.0 U	5.0 U	1.0 U	1.0 U	1.0 U	1.0 U	5.0 U	5.0 U
trans-1,2-Dichloroethene	1.0 U	5.0 U	5.0 U	1.0 U	1.0 U	1.0 U	1.0 U	5.0 U	5.0 U
cis-1,2-Dichloroethene	1.0 U	5.0 U	5.0 U	1.0 U	1.0 U	1.0 U	1.0 U	5.0 U	5.0 U
Chloroform	1.0 U	130	170	130	120	240	290	170	180
1,2-Dichloroethane	1.0 U	5.0 U	5.0 U	1.0 U	1.0 U	1.0 U	1.0 U	5.0 U	5.0 U
2-Butanone	5.0 U	300	280	5.0 U	5.0 U	55	51	30 U	33
1,1,1-Trichloroethane	1.0 U	5.0 U	5.0 U	1.0 U	1.0 U	1.0 U	1.0 U	5.0 U	5.0 U
Carbon Tetrachloride	1.0 U	5.0 U	5.0 U	1.0 U	1.0 U	1.0 U	1.0 U	5.0 U	5.0 U
Vinyl Acetate	1.0 U	5.0 U	5.0 U	1.0 U	1.0 U	1.0 U	1.0 U	5.0 U	5.0 U
Bromodichloromethane	1.0 U	5.0 U	5.0 U	1.0 U	1.0 U	1.0 U	1.0 U	5.0 U	5.0 U
Trichlorofluoromethane	2.0 U	10 U	10 U	2.0 U	2.0 U	2.0 U	2.0 U	10 U	10 U
1,2-Dichloropropane	1.0 U	5.0 U	5.0 U	1.0 U	1.0 U	1.0 U	1.0 U	5.0 U	5.0 U
trans-1,3-Dichloropropene	1.0 U	5.0 U	5.0 U	1.0 U	1.0 U	1.0 U	1.0 U	5.0 U	5.0 U
Trichloroethene	1.0 U	5.0 U	5.0 U	1.0 U	1.0 U	1.0 U	1.0 U	5.0 U	5.0 U
Dibromochloromethane	1.0 U	5.0 U	5.0 U	1.0 U	1.0 U	1.0 U	1.0 U	5.0 U	5.0 U
1,1,2-Trichloroethane	1.0 U	5.0 U	5.0 U	1.0 U	1.0 U	1.0 U	1.0 U	5.0 U	5.0 U
Benzene	1.0 U	5.0 U	5.0 U	1.0 U	1.0 U	1.0 U	1.0 U	5.0 U	5.0 U
cis-1,3-Dichloropropene	1.0 U	5.0 U	5.0 U	1.0 U	1.0 U	1.0 U	1.0 U	5.0 U	5.0 U
2-Chloroethylvinylether	1.0 U	5.0 U	5.0 U	1.0 U	1.0 U	1.0 U	1.0 U	5.0 U	5.0 U
Bromoform	1.0 U	5.0 U	5.0 U	1.0 U	1.0 U	1.0 U	1.0 U	5.0 U	5.0 U
4-Methyl-2-Pentanone	5.0 U	55	53	5.0 U	5.0 U	5.0 U	5.0 U	25 U	25 U
2-Hexanone	5.0 U	25 U	25 U	5.0 U	5.0 U	5.0 U	5.0 U	25 U	25 U
Tetrachloroethene	1.0 U	5.0 U	5.0 U	1.0 U	1.0 U	1.0 U	1.0 U	5.0 U	5.0 U
1,1,2,2-Tetrachloroethane	1.0 U	5.0 U	5.0 U	1.0 U	1.0 U	1.0 U	1.0 U	5.0 U	5.0 U
Toluene	1.0 U	4.6 M	4.1 M	0.6 M	0.9 J	1.0 U	1.0 U	5.0 U	5.0 U
Chlorobenzene	1.0 U	5.0 U	5.0 U	1.0 U	1.0 U	1.0 U	1.0 U	5.0 U	5.0 U
Ethylbenzene	1.0 U	5.0 U	5.0 U	1.0 U	1.0 U	1.0 U	1.0 U	5.0 U	5.0 U
Styrene	1.0 U	5.0 U	5.0 U	1.0 U	1.0 U	1.0 U	1.0 U	5.0 U	5.0 U
Total Xylenes	2.0 U	10 U	10 U	1.1 M	1.1 M	2.0 U	2.0 U	10 U	10 U
1,1,2-Trichloro-1,2,2-trifluoroethane	1.0 U	5.0 U	5.0 U	1.0 U	1.0 U	1.0 U	1.0 U	5.0 U	5.0 U

Appendix D - (cont'd) - Simpson, February 1991.

	Location: Type: Date: Time: Lab Log#:	Trns Blk grab 2/11 1610 078130	Inf-C comp 2/12-13 0800-0800 078133	Ef-C comp 2/12-13 0800-0800 078136	Alk-C comp 2/12-13 0800-0800 078142	Acid-C comp 2/12-13 0800-0800 078145	Sludge gr-comp 2/12 1400&1630 078139
BNA Compounds	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	(ug/Kg-dry wt)
Phenol	2 U	30	2 U	1.5 J	0.8 J	820 J	
Bis(2-Chloroethyl)Ether	1 U	1 U	1 U	1 U	1 U	470 U	
2-Chlorophenol	1 U	1 U	1 U	1 U	1 U	470 U	
1,3-Dichlorobenzene	1 U	1 U	1 U	1 U	1 U	470 U	
1,4-Dichlorobenzene	1 U	1 U	1 U	1 U	1 U	470 U	
Benzyl Alcohol	5 U	16	5 U	1.4 M	2.4 M	2300 U	
1,2-Dichlorobenzene	1 U	1 U	1 U	1 U	1 U	470 U	
2-Methylphenol	1 U	1 U	1 U	1 U	1 U	470 U	
Bis(2-chloroisopropyl)ether	1 U	1 U	1 U	1 U	1 U	470 U	
4-Methylphenol	1 U	1 U	1 U	1 U	1 U	470 U	
N-Nitroso-Di-n-Propylamine	1 U	1 U	1 U	1 U	1 U	470 U	
Hexachloroethane	2 U	2 U	2 U	2 U	2 U	940 U	
Nitrobenzene	1 U	1 U	1 U	1 U	1 U	470 U	
Isophorone	1 U	1 U	1 U	1 U	1 U	470 U	
2-Nitrophenol	5 U	5 U	5 U	5 U	5 U	470 U	
2,4-Dimethylphenol	2 U	3.8 M	0.9 J	2 U	5 U	2300 U	
Benzoic Acid	10 U	10 U	10 U	31	27	940 U	
Bis(2-Chloroethoxy)Methane	1 U	1 U	1 U	1 U	1 U	470 U	
2,4-Dichlorophenol	3 U	0.5 M	3 U	4.7 J	1 U	500 U	
1,2,4-Trichlorobenzene	1 U	1 U	1 U	1 U	4.0 J	1400 U	
Naphthalene	1 U	1 U	1 U	1 U	1 U	470 U	
4-Chloroaniline	3 U	3 U	3 U	3 U	1 U	700 J	
Hexachlorobutadiene	2 U	2 U	2 U	2 U	3 U	1400 U	
4-Chloro-3-Methylphenol	2 U	2 U	2 U	2 U	2 U	940 U	
2-Methylnaphthalene	1 U	1.6 J	1 U	2 U	2 U	940 U	
Hexachlorocyclopentadiene	5 U	5 U	5 U	5 U	1 U	610 J	
2,4,6-Trichlorophenol	5 U	5 U	5 U	5 U	5 U	2300 U	
2,4,5-Trichlorophenol	5 U	5 U	5 U	3.6 J	2.0 J	2300 U	
2-Chloronaphthalene	1 U	1 U	1 U	5 U	5 U	2300 U	
2-Nitroaniline	5 U	5 U	5 U	1 U	1 U	470 U	
Dimethyl Phthalate	1 U	1 U	1 U	5 U	5 U	2300 U	
Acenaphthylene	1 U	1 U	1 U	1 U	1 U	470 U	
3-Nitroaniline	5 U	5 U	5 U	1 U	1 U	470 U	
Acenaphthene	1 U	1 U	1 U	5 U	5 U	2300 U	
2,4-Dinitrophenol	10 U	10 U	10 U	1 U	1 U	470 U	
4-Nitrophenol	5 U	5 U	5 U	10 U	10 U	4700 U	
Dibenzofuran	1 U	1 U	1 U	5 U	5 U	2300 U	
2,4-Dinitrotoluene	5 U	5 U	5 U	1 U	1 U	470 U	
2,6-Dinitrotoluene	5 U	5 U	5 U	5 U	5 U	2300 U	
Diethyl Phthalate	1 U	1 U	0.7 J	5 U	5 U	2300 U	
4-Chlorophenyl-Phenylether	1 U	1 U	1 U	0.9 J	1 U	470 U	
Fluorene	1 U	1 U	1 U	1 U	1 U	470 U	
4-Nitroaniline	5 U	5 U	5 U	1 U	1 U	470 U	
4,6-Dinitro-2-Methylphenol	10 U	10 U	10 U	5 U	5 U	2300 U	
N-Nitrosodiphenylamine	1 U	1 U	1 U	10 U	10 U	4700 U	
4-Bromophenyl-Phenylether	1 U	1 U	1 U	1 U	1 U	470 U	
				1 U	1 U	470 U	

Appendix D - (cont'd) - Simpson, February 1991.

Location:	Trns Blk	Inf-C	Ef-C	Ef-Sim	Alk-C	Acid-C	Sludge
Type:	grab	comp	comp	S-comp	comp	comp	gr-comp
Date:	2/11	2/12-13	2/12-13	2/12-13	2/12-13	2/12-13	2/12
Time:	1610	0800-0800	0800-0800	0800-0800	0800-0800	0800-0800	1400&1630
Lab Log#:	078130	078133	078136	078138	078142	078145	078139
BNA Compounds	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	(ug/Kg-dry wt)
Hexachlorobenzene	1 U	1 U	1 U	1 U	1 U	1 U	470 U
Pentachlorophenol	5 U	5 U	5 U	5 U	5 U	5 U	2300 U
Phenanthrene	1 U	0.9 M	1 U	1 U	1 U	1 U	420 J
Anthracene	1 U	1 U	1 U	1 U	1 U	1 U	470 U
Di-n-Butyl Phthalate	1 U	1 U	1 U	1 U	1 U	1 U	470 U
Fluoranthene	1 U	1 U	1 U	1 U	1 U	1 U	470 U
Pyrene	1 U	1 U	1 U	1 U	1 U	1 U	470 U
Butylbenzylphthalate	1 U	1 U	1 U	1 U	1 U	1 U	470 U
3,3'-Dichlorobenzidine	5 U	5 U	5 U	5 U	5 U	5 U	2300 U
Benzo(a)Anthracene	1 U	1 U	1 U	1 U	1 U	1 U	470 U
Bis(2-Ethylhexyl)phthalate	1 U	1 U	1 U	1 U	1 U	1 U	470 U
Chrysene	1 U	1 U	1 U	1 U	1 U	1 U	470 U
Di-n-Octyl Phthalate	1 U	1 U	1 U	1 U	1 U	1.3 J	470 U
Benzo(b)Fluoranthene	1 U	1 U	1 U	1 U	1 U	1 U	470 U
Benzo(k)Fluoranthene	1 U	1 U	1 U	1 U	1 U	1 U	470 U
Benzo(a)Pyrene	1 U	1 U	1 U	1 U	1 U	1 U	470 U
Indeno(1,2,3-cd)Pyrene	1 U	1 U	1 U	1 U	1 U	1 U	470 U
Dibenzo(a,h)Anthracene	1 U	1 U	1 U	1 U	1 U	1 U	470 U
Benzo(g,h,i)Perylene	1 U	1 U	1 U	1 U	1 U	1 U	470 U
Metals *	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L
Antimony	30 UJ	30 U					
Arsenic	1.5 U	2.1 JR	1.9 JR				
Beryllium	1.0 U	1.0 U	1.0 U	1.0 U	1.0 U	1.0 U	1.0 U
Cadmium	0.10 U	0.82 JR	0.69 JR				
Chromium	5.0 U	10 J	6.6 J	6.6 J	6.6 J	6.6 J	6.6 J
Copper	11 B	24.2	16 B				
Lead	1.0 U	20.6	14.5	14.5	14.5	14.5	14.5
Mercury (total)	0.04 U	0.04 UR	0.07 JR				
Nickel	2.0 U	15	12	12	12	12	12
Selenium	2.0 U	2 UJR					
Silver	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U
Thallium	2.5 U	2.5 U	2.5 U	2.5 U	2.5 U	2.5 U	2.5 U
Zinc	4.0 U	37 J	11 J				
Copper (dissolved)	3.5 JB	12 B	15 B	15 B	15 B	6.7 JB	6.7 JB

- U indicates compound was analyzed for but not detected at the given detection limit.
- J indicates an estimated value.
- B This flag is used when the analyte is found in the method blank as well as the sample. Sample concentration is less than 10 times blank concentration.
- M indicates an estimated value of analyte found and confirmed by analyst but with low spectral match parameters.
- UJ indicates compound was analyzed for but not detected at the estimated detection limit.
- R low spike recovery - result may be biased low.

- * metals results are total recoverable unless otherwise indicated.
- Inf influent to secondary treatment
- Alk alkaline stream from bleach plant
- Acid acid stream from bleach plant
- Ef final effluent
- S-comp Simpson composite sample

Appendix E – VOA and BNA Scan Tentatively Identified Compounds (TICs) –
Simpson, February 1991.

TIC data are presented on the laboratory report sheets that follow. Locations corresponding to the Lab Log# (called Sample No. on the laboratory report sheet) and data qualifiers are summarized on this page.

Location:	Trns Blk	Inf-1	Inf-2	Ef-1	Ef-2
Type:	grab	grab	grab	grab	grab
Date:	2/11	2/12	2/12	2/12	2/12
Time:	1610	1025	1415	1205	1515
Lab Log#:	078130	078131	078132	078134	078135

Location:	Alk-1	Alk-2	Acid-1	Acid-2
Type:	grab	grab	grab	grab
Date:	2/12	2/12	2/12	2/12
Time:	1105	1455	1055	1450
Lab Log#:	078140	078141	078143	078144

Location:	Trns Blk	Inf-C	Ef-C	Alk-C	Acid-C	Sludge
Type:	grab	comp	comp	comp	comp	gr-comp
Date:	2/11	2/12-13	2/12-13	2/12-13	2/12-13	2/12
Time:	1610	0800-0800	0800-0800	0800-0800	0800-0800	1400&1630
Lab Log#:	078130	078133	078136	078142	078145	078139

- J indicates an estimated value.
- JN there is presumptive evidence that the analyte is present.
The associated numerical value is an estimate.
- Inf influent to secondary treatment
- Alk alkaline stream from bleach plant
- Acid acid stream from bleach plant
- Ef final effluent



02-238
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ORGANIC ANALYSIS DATA SHEET - Tentatively Identified Compounds

Sample No: #078131 re-analysis 2

Lab ID: 7785Bre2
Matrix: Water

QC Report No: 7785-WDOE
Project No: Simpson Kraft
VTSR: 2/19/91

Data Release Authorized: *[Signature]*

	CAS Number	Compound Name	Fraction	Scan Number	Estimated Concentration (µg/L)
1	74-93-1	Methanethiol	VOA	236	36 J
2	75-18-3	Thiobismethane	VOA	346	1300 J
3	624-92-0	Dimethyldisulfide	VOA	879	2800 J
4	-	C10.H16 Isomer	VOA	1449	130 J
5	3658-80-8	Dimethyltrisulfide	VOA	1478	980 J
6	-	Methyl-(methylethyl)-benzene Isomer	VOA	1499	28 J
7	-	C10.H16 Isomer	VOA	1613	58 J
8					
9					
10					
11					
12					
13					
14					
15					
16					
17					
18					
19					
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ORGANIC ANALYSIS DATA SHEET - Tentatively Identified Compounds

Sample No: #078132 re-analysis 2

QC Report No: 7785-WDOE
Project No: Simpson Kraft
VTSR: 2/19/91

Lab ID: 7785Cre2
Matrix: Water

Data Release Authorized: *[Signature]*

	CAS Number	Compound Name	Fraction	Scan Number	Estimated Concentration (µg/L)
1	74-93-1	Methanethiol	VOA	235	140 J
2	75-18-3	Thiobismethane	VOA	346	1300 J
3	624-92-0	Dimethyldisulfide	VOA	879	2800 J
4	-	UNKNOWN (BP M/E 93)	VOA	1267	29 J
5	-	C10.H16 Isomer	VOA	1449	130 J
6	3658-80-8	Dimethyltrisulfide	VOA	1478	190 J
7	-	Methyl-(methylethyl)-benzene Isomer	VOA	1499	37 J
8	-	C10.H16 Isomer	VOA	1613	52 J
9					
10					
11					
12					
13					
14					
15					
16					
17					
18					
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ORGANIC ANALYSIS DATA SHEET - Tentatively Identified Compounds

Sample No: #078134

QC Report No: 7785-WDOE
Project No: Simpson Kraft
VTSR: 2/19/91

Lab ID: 7785D
Matrix: Water

Data Release Authorized: *[Signature]*

CAS Number	Compound Name	Fraction	Scan Number	Estimated Concentration (ug/L)
1	UNKNOWN (BP M/E 105)	VOA	1521	6J
2	UNKNOWN (BP M/E 119)	VOA	1563	5J
3	C10.H16 Isomer	VOA	1603	6J
4	UNKNOWN (BP M/E 119)	VOA	1678	6J
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ORGANIC ANALYSIS DATA SHEET - Tentatively Identified Compounds

Sample No: #078135

QC Report No: 7785-WDOE
Project No: Simpson Kraft
VTSR: 2/19/91

Lab ID: 7785E
Matrix: Water

Data Release Authorized: *Chris N. Baker*

CAS Number	Compound Name	Fraction	Scan Number	Estimated Concentration (µg/L)
1 75-18-3	Thiobismethane	VOA	344	9 J
2 -	UNKNOWN (BP M/E 105)	VOA	1523	5 J
3 -	UNKNOWN (BP M/E 119)	VOA	1565	10 J
4 -	C10.H16 Isomer	VOA	1605	5 J
5 -	UNKNOWN (BP M/E 41)	VOA	1627	6 J
6 -	Methylbenzene Isomer	VOA	1730	6 J
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ORGANIC ANALYSIS DATA SHEET - Tentatively Identified Compounds

Sample No: #078140

QC Report No: 7785-WDOE
Project No: Simpson Kraft
VTSR: 2/19/91

Lab ID: 7785F
Matrix: Water

Data Release Authorized: Don T. Cooper

	CAS Number	Compound Name	Fraction	Scan Number	Estimated Concentration (µg/L)
1	66-25-1	Hexanal	VOA	976	7 J
2	-	C7.H14.O Isomer	VOA	1217	6 J
3	-	UNKNOWN (BP M/E 41)	VOA	1627	6 J
4	-	UNKNOWN (BP M/E 188)	VOA	1686	8 J
5	-	UNKNOWN (BP M/E 43)	VOA	1738	12 J
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ORGANIC ANALYSIS DATA SHEET - Tentatively Identified Compounds

Sample No: #078141

QC Report No: 7785-WDOE
Project No: Simpson Kraft
VTSR: 2/19/91

Lab ID: 7785G
Matrix: Water

Data Release Authorized: *[Signature]*

CAS Number	Compound Name	Fraction	Scan Number	Estimated Concentration (µg/L)
1 66-25-1	Hexanal	VOA	976	9 J
2 -	C7.H14.O Isomer	VOA	1216	8 J
3 -	UNKNOWN (BP M/E 41)	VOA	1626	7 J
4 -	UNKNOWN (BP M/E 188)	VOA	1685	10 J
5 -	UNKNOWN (BP M/E 58)	VOA	1733	6 J
6 -	UNKNOWN (BP M/E 43)	VOA	1737	8 J
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ORGANIC ANALYSIS DATA SHEET - Tentatively Identified Compounds

Sample No: #078133

Lab ID: F47785J2
Matrix: Water

QC Report No: 7785-WDOE
Project No: Simpson Kraft
VTSR: 2/19/91

Data Release Authorized: *Dumb. Patton*

CAS Number	Compound Name	Fraction	Scan Number	Estimated Concentration (µg/L)
1 3658-80-8	Trisulfide, Dimethyl	ABN	378	150 J \checkmark
2 -	Unknown Trimethyl-2-cyclopenten-1-one Isomer (BP M/E 109)	ABN	579	480 J
3 -	Unknown Hydrocarbon (BP M/E 43)	ABN	585	51 J
4 -	Unknown (BP M/E 43)	ABN	621	56 J
5 -	Unknown C10.H18.0 Isomer (BP M/E 95)	ABN	663	54 J
6 -	Unknown C10.H18.0 Isomer (BP M/E 71)	ABN	678	61 J
7 -	Unknown (BP M/E 59)	ABN	694	150 J
8 10482-56-1	3-Cyclohexene-1-methanol, .alpha..alpha. 4-trimethyl-, (S)-	ABN	698	70 J
9 -	Unknown (BP M/E 45)	ABN	718	91 J
10 -	Unknown (BP M/E 137)	ABN	796	72 J
11 -	Unknown (BP M/E 151)	ABN	928	240 J
12 -	Unknown (BP M/E 119)	ABN	1001	59 J
13 -	Unknown (BP M/E 151)	ABN	1013	150 J
14 -	Unknown (BP M/E 257)	ABN	1448	64 J
15 10544-50-0	Sulfur, Mol. (58)	ABN	1455	85 J
16 -	Unknown (BP M/E 41)	ABN	1485	290 J
17 -	Unknown (BP M/E 41)	ABN	1557	46 J
18 -	Unknown (BP M/E 41)	ABN	1595	46 J
19 -	Unknown (BP M/E 285)	ABN	1745	53 J
20 -	Unknown (BP M/E 43)	ABN	1771	64 J \downarrow
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ORGANIC ANALYSIS DATA SHEET - Tentatively Identified Compounds

Sample No: #078136

QC Report No: 7785-WDOE
Project No: Simpson Kraft
VTSR: 2/19/91

Lab ID: F47785K
Matrix: Water

Data Release Authorized:

D. B. Patton

CAS Number	Compound Name	Fraction	Scan Number	Estimated Concentration (ug/L)
1	Unknown (BP M/E 67)	ABN	332	31 J <i>N</i>
2	Unknown (BP M/E 126)	ABN	522	18 J <i>1</i>
3	Unknown (BP M/E 111)	ABN	563	11 J
4	Chloroform Unknown (BP M/E 83) <i>UNK 2</i>	ABN	591	24 J
5	Unknown (BP M/E 94)	ABN	642	21 J
6	Unknown (BP M/E 139)	ABN	781	68 J
7	Unknown C15.H24 Isomer (BP M/E 41)	ABN	922	29 J
8	Unknown (BP M/E 41)	ABN	1385	25 J
9	Unknown (BP M/E 43)	ABN	1410	13 J
10	Unknown (BP M/E 41)	ABN	1430	12 J
11	Unknown (BP M/E 257)	ABN	1447	19 J
12	Unknown (BP M/E 41)	ABN	1478	39 J
13	Unknown (BP M/E 272)	ABN	1496	63 J
14	Unknown (BP M/E 43)	ABN	1554	14 J
15	Unknown (BP M/E 43)	ABN	1638	39 J
16	Unknown C20.H40 Isomer (BP M/E 43)	ABN	1769	50 J
17	Unknown (BP M/E 43)	ABN	1814	18 J
18	Unknown (BP M/E 137)	ABN	1859	35 J
19	Unknown (BP M/E 43)	ABN	1890	25 J
20	Unknown Sterol (BP M/E 43)	ABN	2176	29 J
21	Unknown Sterol (BP M/E 43)	ABN	2225	75 J
22	Unknown (BP M/E 43)	ABN	2230	26 J <i>Y</i>
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ORGANIC ANALYSIS DATA SHEET - Tentatively Identified Compounds

Sample No: #078142

QC Report No: 7785-WDOE
Project No: Simpson Kraft
VTSR: 2/19/91

Lab ID: F47785L
Matrix: Water

Data Release Authorized: *Don B. Patton*

CAS Number	Compound Name	Fraction	Scan Number	Estimated Concentration (µg/L)
1	Unknown (BP M/E 83) <i>Chlorinated</i>	ABN	595	92 J <i>SM</i>
2	Unknown (BP M/E 151)	ABN	924	72 J
3	Dichloromethoxyphenol Isomer (BP M/E 177)	ABN	1004	170 J
4	Unknown (BP M/E 185)	ABN	1077	320 J
5	Unknown (BP M/E 211)	ABN	1093	49 J
6	Unknown (BP M/E 43)	ABN	1429	260 J
7	Unknown (BP M/E 43)	ABN	1480	250 J
8	Unknown (BP M/E 43)	ABN	1628	28 J
9	Unknown (BP M/E 43)	ABN	1642	73 J
10	Unknown (BP M/E 269)	ABN	1648	28 J
11	Unknown (BP M/E 55)	ABN	1682	190 J
12	Unknown (BP M/E 43)	ABN	1699	79 J
13	Unknown (BP M/E 43)	ABN	1775	120 J
14	Unknown (BP M/E 55)	ABN	1813	60 J
15	Unknown (BP M/E 43)	ABN	1827	130 J
16	Unknown (BP M/E 43)	ABN	1895	200 J
17	Unknown (BP M/E 43)	ABN	1940	230 J
18	Unknown (BP M/E 43)	ABN	2096	76 J
19	Unknown (BP M/E 43)	ABN	2183	110 J
20	Unknown (BP M/E 43)	ABN	2234	300 J
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ORGANIC ANALYSIS DATA SHEET - Tentatively Identified Compounds

Sample No: #078145

Lab ID: F47785M

Matrix: Water

QC Report No: 7785-WDOE
Project No: Simpson Kraft
VTSR: 2/19/91Data Release Authorized: *Ram B. Patten*

CAS Number	Compound Name	Fraction	Scan Number	Estimated Concentration (µg/L)
1	<i>for</i> (Chlorinated) Unknown (BP M/E 83)	ABN	600	160 J <i>N</i>
2	Unknown (BP M/E 53)	ABN	661	53 J
3	Unknown (BP M/E 45)	ABN	699	84 J
4	Unknown (BP M/E 43)	ABN	818	18 J
5	124-17-4 Ethanol, 2-(2-butoxyethoxy)-, acetate	ABN	893	71 J
6	121-33-5 Benzaldehyde, 4-hydroxy-3-methoxy-	ABN	920	19 J
7	Dichloromethoxyphenol isomer (BP M/E 177)	ABN	1001	61 J
8	Unknown (BP M/E 185)	ABN	1069	84 J
9	Unknown (BP M/E 176)	ABN	1233	16 J
10	Unknown (BP M/E 57)	ABN	1353	24 J
11	Unknown (BP M/E 43)	ABN	1391	31 J
12	Unknown (BP M/E 43)	ABN	1411	31 J
13	Unknown (BP M/E 43)	ABN	1464	15 J
14	Unknown (BP M/E 43)	ABN	1689	15 J
15	Unknown (BP M/E 43)	ABN	1769	34 J
16	112-85-6 Docosanoic Acid	ABN	1816	41 J
17	Unknown (BP M/E 187)	ABN	1851	39 J
18	Unknown (BP M/E 43)	ABN	1891	23 J
19	Unknown (BP M/E 43)	ABN	1934	28 J
20	Unknown (BP M/E 43)	ABN	2231	40 J <i>v</i>
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ORGANIC ANALYSIS DATA SHEET - Tentatively Identified Compounds

Sample No:

#078139

QC Report No: 7785-WDOE

Project No: Simpson Kraft

VTSR: 2/19/91

Lab ID: F47785N

Matrix: Sludge

Data Release Authorized: *Dann B. Patton*

CAS Number	Compound Name	Fraction	Scan Number	Estimated Concentration (µg/kg)
1	Unknown C ₁₀ H ₁₆ Isomer (BP M/E 93)	ABN	320	15000 J ✓
2	Unknown Decahydro-Naphthalene Isomer (BP M/E 67)	ABN	506	11000 J
3	Unknown Hydrocarbon (BP M/E 57)	ABN	526	15000 J
4	Unknown Hydrocarbon (BP M/E 43)	ABN	536	20000 J
5	Unknown Hydrocarbon (BP M/E 57)	ABN	545	21000 J
6	Unknown (BP M/E 55)	ABN	557	14000 J
7	Unknown Hydrocarbon (BP M/E 69)	ABN	565	24000 J
8	Unknown (BP M/E 43)	ABN	589	67000 J
9	Unknown (BP M/E 43)	ABN	608	23000 J
10	Unknown Hydrocarbon (BP M/E 43)	ABN	620	24000 J
11	Unknown (BP M/E 57)	ABN	654	20000 J
12	Unknown Hydrocarbon (BP M/E 43)	ABN	665	15000 J
13	Unknown Hydrocarbon (BP M/E 43)	ABN	709	30000 J
14	Unknown Hydrocarbon (BP M/E 43)	ABN	724	13000 J
15	Unknown C ₁₃ H ₂₈ Isomer (BP M/E 57)	ABN	724	13000 J
16	Unknown (BP M/E 43)	ABN	1416	30000 J
17	Unknown (BP M/E 43)	ABN	1448	32000 J
18	Unknown (BP M/E 41)	ABN	1480	48000 J
19	Unknown (BP M/E 272)	ABN	1497	25000 J
20	Unknown (BP M/E 43)	ABN	1771	21000 J
21	Unknown Sterol (BP M/E 43)	ABN	2227	29000 J ✓
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Laboratory Procedure Review Sheet

Q = 30.4 mg/d

Discharger: Simpson

Date: 2/13

Discharger representative: Paul Sporenberg / Dick Forsberg

Ecology reviewer: Heffner

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? *140*
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? *cont. hours*
5. What time does sample collection usually begin? *0715-0715 (collected 0800-0800)*
6. How long does sample collection last? *24 hours*
7. How often are subsamples that make up the composite collected? *every 15 minutes*

8. What volume is each subsample?
9. What is the final volume of sample collected? *1.5 gal*
10. Is the composite cooled during collection? *refrigerating*
11. To what temperature? *4°C*
The sample should be maintained at approximately 4 degrees C (SM, p. 41, #5b: SSM, p. 2).
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? *daily*
The temperature should be checked at least monthly to assure adequate cooling.
14. Are the sampling locations representative? *OK (tide)*
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. *stir*
17. How is the subsample stored prior to analysis? —
The sample should be refrigerated (4 degrees C) until about one hour before analysis, at which time it is allowed to warm to room temperature.
18. What is the cleaning frequency of the collection jugs? *daily rinse - scrub w/d.*
The jugs should be thoroughly rinsed after each sample is complete and occasionally be washed with a non-phosphate detergent.
19. How often are the sampler lines cleaned? *clean probes monthly*
Rinsing lines with a chlorine solution every three months or more often where necessary is suggested. *change lines every 2 mths*

pH Test Review

1. How is the pH measured? *Ecology*
A meter should be used. Use of paper or a colorimetric test is inadequate and those procedures are not listed in Standard Methods (SM, p. 429).
2. How often is the meter calibrated? *daily*
The meter should be calibrated every day it is used.
3. What buffers are used for calibration? *4-7-10*
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? *Co. manual from Sed Method 14th*
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? *2-4 hours*
The test should begin within 24 hours of composite sample completion (Ecology Lab Users Manual, p. 42). 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? *glass*
Copper stills can leave a copper residual in the water which can be toxic to the test (SSM, p. 36).
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 four nutrient buffers of powder pillows used to make dilution water? *1/ month*
If the nutrients are used, how much buffer per liter of dilution water are added? *OK*
1 mL per liter should be added (SM, p527, #5a: SSM, p37).
8. How often is the dilution water prepared? *daily*
Dilution water should be made for each set of BODs run.
9. Is the dilution water aged prior to use? *OK*
Dilution water with nitrification inhibitor can be aged for a week before use (SM, p. 528, #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? *6.8-7.2 - primary adjusted*
If no, is the sample pH adjusted? *no of OK*
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, p. 529, #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? *primary seeded 0.9/300 mL*
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, p. 529, #5e2: SSM p. 38), 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, p. 528, #5d&5e2: SSM, p.38).
13. Do any samples have a toxic effect on the BOD test? *no*
Specific modifications are probably necessary (SM, p. 528, #5d: SSM, p. 37).

14. How are D.O. concentrations measured? *yes*
If with a meter, how is the meter calibrated? *winkler daily*
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 D.O. 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? *OK*
A dilution water blank should always be run for quality assurance (SM, p.527, #5b; SSM, p.40, #3).

What is the usual initial D.O. of the blank? *8.3*
The D.O. should be near saturation; 7.8 mg/L @ 4000 ft, 9.0 mg/L @ sea level (SM, p. 528, #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 D.O. or excess blank depletion is a problem.

What is the usual five-day blank depletion? *0.1 or less*
The depletion should be 0.2 mg/L or less. If the depletion is greater, the cause should be found (SM, p.527-8, #5b; SSM, p.41, #6).

16. How many dilutions are made for each sample? *4 on 20 of*
At least two dilutions are recommended. The dilutions should be far enough apart to provide a good extended range (SM, p.530, #5f; SSM, p.41).

17. Are dilutions made by the liter method or in the bottle? *bottle*
Either method is acceptable (SM, p.530, #5f).

18. How many bottles are made at each dilution? *one*
How many bottles are incubated at each dilution? *one*
When determining the D.O. using a meter, only one bottle is necessary. The D.O. is measured, then the bottle is sealed and incubated (SM, p. 530, #5f2).

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

19. Is the initial D.O. of each dilution measured? *yes*
What is the typical initial D.O.?
The initial D.O. of each dilution should be measured. It should approximate saturation (see #14).

20. What is considered the minimum acceptable D.O. depletion after five days? *OK*
What is the minimum D.O. that should be remaining after five days?
The depletion should be at least 2.0 mg/L and at least 1.0 mg/L should be left after five days (SM, p.531, #6: SSM, p.41).

21. Are any samples seeded? *10 - Bleach*
Which?

What is the seed source? *20*

Primary effluent or settled raw wastewater is the preferred seed. Secondary treated sources can be used for inhibited tests (SM, p.528, #5d: SSM, p.41).

How much seed is added to each sample? *0.9 mL / 300 mL*

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, p.529, #5d).

How is the BOD of the seed determined? *non-permit samples - connect w/ seed blank*
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, p.529, #5d: SSM, p.41).

22. What is the incubator temperature? *OK*
The incubator should be kept at 20 +/- 1 degree C (SM, p.531, #5i: SSM, p.40, #3).

How is incubator temperature monitored? *OK*

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? *daily*

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? *OK*

Adjustment should be infrequent. If frequent adjustments (every two weeks or more often) are required the incubator should be repaired.

Is the incubator dark during the test period? *OK*

Assure the switch that turns off the interior light is functioning.

23. Are water seals maintained on the bottles during incubation? *OK*
Water seals should be maintained to prevent leakage of air during the incubation period (SM, p.531, #5i: SSM, p.40, #4).

24. Is the method of calculation correct? *OK*
Check to assure that no correction is made for any D.O. depletion in the blank and that the seed correction is made using seed control data.

Standard Method calculations are (SM, p.531, #6):

for unseeded samples; $BOD (mg/L) = \frac{D1 - D2}{P}$

for seeded samples; $BOD (mg/L) = \frac{(D1 - D2) - (B1 - B2)f}{P}$

*seeded done on
primary only - non-
permit - ~~correct~~
w/ seeded blank*

Where:

D1 = D.O. of the diluted sample before incubation (mg/L)

D2 = D.O. of diluted sample after incubation period (mg/L)

P = Decimal volumetric fraction of sample used

B1 = D.O. of seed control before incubation (mg/L)

B2 = D.O. 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? *CO manual*
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, p.95, footnote: SSM, p.23) *GFC whatman -
OK on QA*
3. What is the drying oven temperature? *102 -*
The temperature should be 103-105 degrees C (SM, p.96, #3a: SSM, p.23).
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, p.98, #3: SSM, p.23).
5. What type of filtering apparatus is used? *buchner funnel*
Gooch crucibles or a membrane filter apparatus should be used
(SM, p.95, #2b: SSM, p.23).

6. How are the filters pre-washed prior to use? *yes*
The filters should be rinsed three times with distilled water (SM, p.23, #2: SSM, p.23, #2).
- Are the rough or smooth sides of the filters up? *Keep sealed*
The rough side should be up (SM, p.96, #3a: SSM, p.23, #1)
- How long are the filters dried? *overnight*
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? *OK*
The filters should be stored in a desiccator (Ibid).
7. How is the effectiveness of the desiccant checked? *OK*
All or a portion of the desiccant should have an indicator to assure effectiveness.

Test Procedure

8. In what is the test volume of sample measured? *200 ml's*
The sample should be measured with a wide tipped pipette or a graduated cylinder.
9. Is the filter seated with distilled water? *OK*
The filter should be seated with distilled water prior to the test to avoid leakage along the filter sides (SM, p.97, #3c).
10. Is the entire measured volume always filtered? *OK*
The entire volume should always be filtered to allow the measuring vessel to be properly rinsed (SM, p.97, #3c: SSM, p.24, #4).

11. What are the average and minimum volumes filtered?

	Volume	
	<u>Minimum</u>	<u>Average</u>
Influent		
Effluent		

12. How long does it take to filter the samples? Time 30.00 sec
- | | <u>Time</u> |
|----------|-------------|
| Influent | |
| Effluent | |

13. How long is filtering attempted before deciding that a filter is clogged? < 5 min
Prolonged filtering can cause high results due to dissolved solids being caught in the filter (SM, p.96, #1b). We usually advise a five minute filtering maximum.

14. What do you do when a filter becomes clogged? OK
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? yes
Rinse three times with approximately 10 mLs of distilled water each time (? ?).

16. How long is the sample dried? 2 hrs
The sample should be dried at least one hour for the TSS test and 20 minutes for the volatile test (SM, p.97, #3c; p.98, #3: SSM, p.24, #4). Excessive drying times (such as overnight) should be avoided.

17. Is the filter thoroughly cooled in a desiccator prior to weighing? yes
The filter must be cooled to avoid drafts due to thermal differences when weighing (SM, p.97, #3c: SSM, p.97 #3c).

18. How frequently is the drying cycle repeated to assure constant filter weight has been reached (weight loss <0.5 mg or 4 percent, whichever is less: SM, p.97, #3c)? no
We recommend that this be done at least once every two months.

19. Do calculations appear reasonable? OK
Standard Methods calculation (SM, p.97, #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)