

MILLER CREEK WTP  
CLASS II INSPECTION  
April 1991

by  
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## ABSTRACT

A Class II Inspection was conducted at the Miller Creek Wastewater Treatment Plant in Seattle, Washington, on April 22-24, 1991. The effluent met NPDES permit requirements. Copper, silver, and mercury were found in 100 percent effluent at concentrations above the acute and chronic marine water quality criteria. No whole effluent toxicity was indicated by rainbow trout or fathead minnow bioassays. Seven volatile organic compounds and twelve priority pollutant metals were detected in plant sludge.

## INTRODUCTION

A Class II Inspection was conducted at the Miller Creek (MC) Wastewater Treatment Plant (WTP) on April 22-24, 1991. Conducting the inspection were Tapas Das, Norm Glenn, and Rebecca Inman from the Washington State Department of Ecology's (Ecology) Environmental Investigations and Laboratory Services Program (EILS). Dale Van Donsel and Perry Brake of EILS's Quality Assurance Section conducted an on-site laboratory inspection on April 23, 1991. The inspection was requested by Laura Fricke of the Ecology Northwest Regional Office (NWRO). Tim Yokers, MC's Operations Supervisor, provided assistance during the inspection.

MC's wastewater treatment plant is located in Normandy Park near the City of Seattle (Figure 1). MC's plant is operated by the Southwest Suburban Sewer District of Seattle. MC's collection system serves approximately 36,000 residential users. No industrial wastes are contributed to the plant. The MC discharge into Puget Sound is regulated by NPDES Permit No. WA-002276-4 which expired on July 25, 1991.

The original treatment plant was built in 1965 to provide primary treatment. The plant was upgraded in 1973-74, and underwent extensive modifications to achieve secondary treatment capability in 1988, including the addition of Rotating Biological Contactors (RBCs). The existing wastewater treatment system consists of a mechanically cleaned bar screen, screening grinder, grit chamber, primary clarifier, RBC units, secondary clarifier, and chlorine contact chamber (Figure 2).

Sludge process units include primary and secondary anaerobic digesters, a filter press, and odor scrubbers. Sludge handling includes an option to compost and market the sludge. Currently, MC is composting about one-fourth of the sludge it produces and hauling the rest to an outside composting site or to a land application site.

The objectives of the inspection were:

- Verify flow meter accuracy;
- Assess MC effluent compliance with NPDES permit limits;
- Chemically characterize WTP effluent;
- Determine effluent toxicity using rainbow trout and fathead minnow larvae; and
- Split samples with the permittee to determine comparability of both sampling methods and laboratory analyses.

## PROCEDURES

Twenty-four hour composite samples and grab samples of wastewater were taken at two locations: (1) influent at a point between the bar screen and comminutor; and (2) chlorinated effluent at the end of the chlorine contact chamber prior to the inlet of the Parshall flume

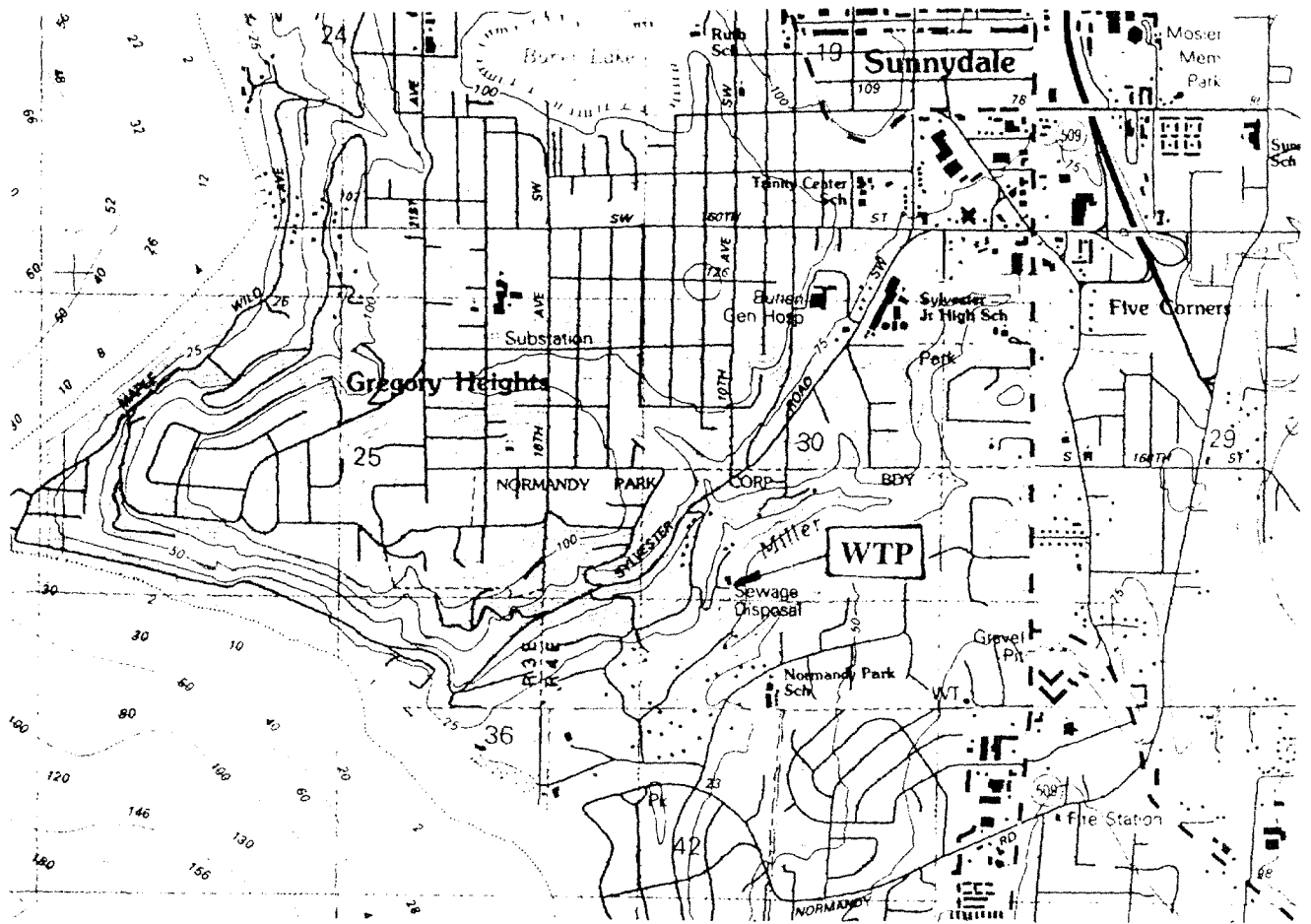
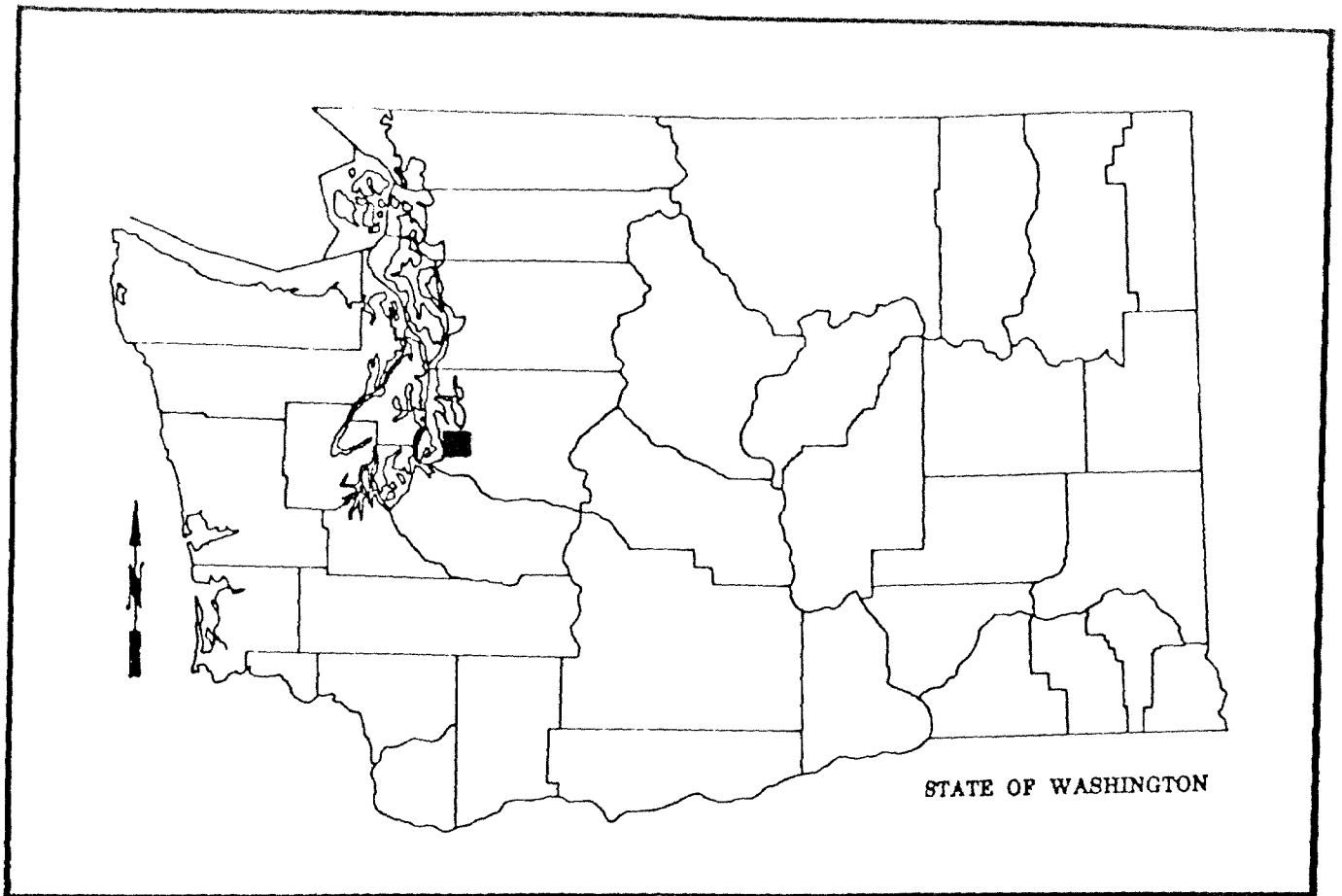


Figure 1 - Location Map - Miller Creek WTP, 4/91

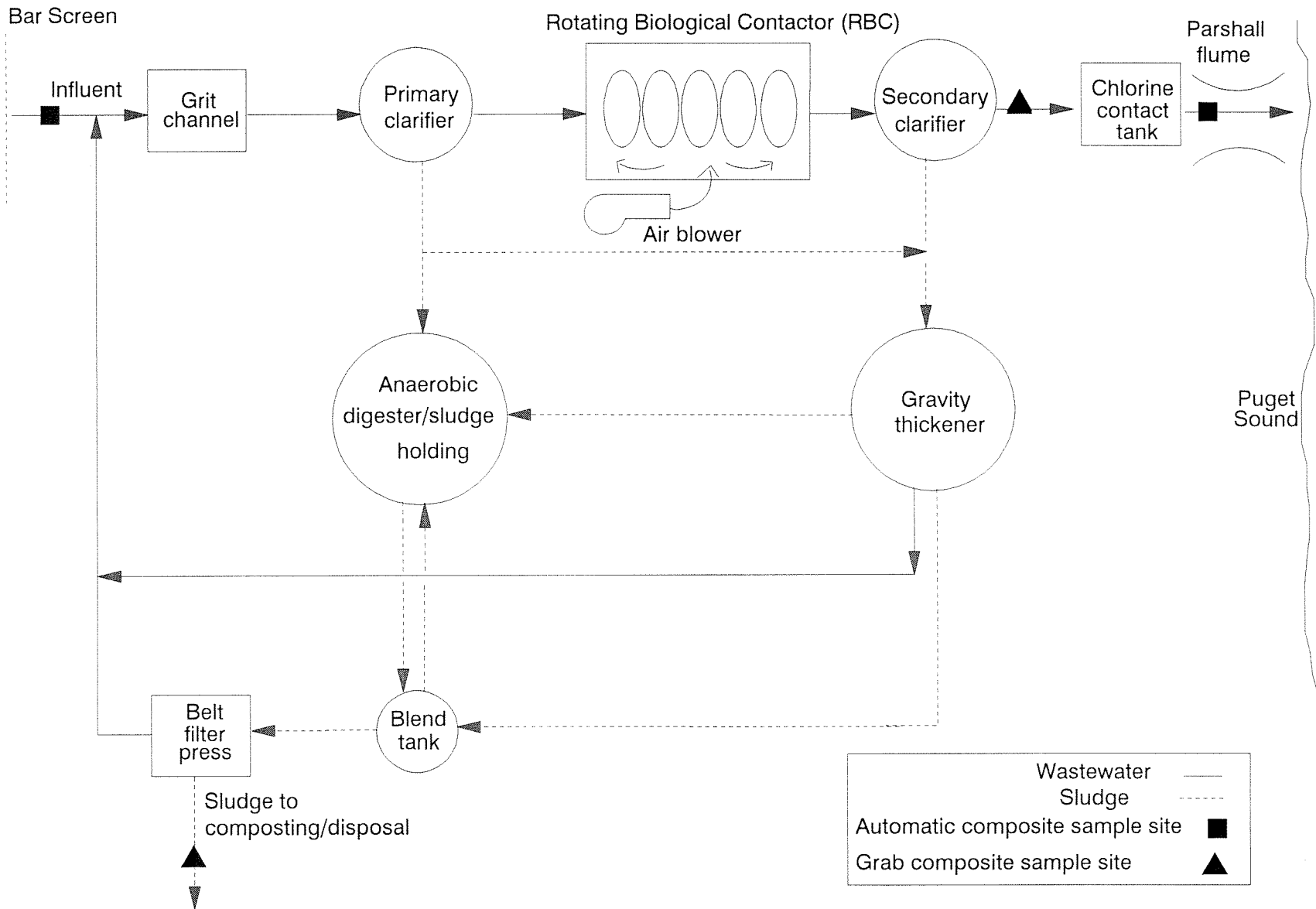


Figure 2 - Schematic and Sample Sites - Miller Creek Wastewater Treatment Plant, 4/91

(Figure 2). An additional composite sampler was set up to collect replicate samples of effluent to assure representativeness of the samples and to observe any variability of lab results. ISCO® compositors were set for time proportional collection of 320 mL of sample every 30 minutes. MC's influent and effluent composite samplers were installed at approximately the same location. They were set for flow proportional collection and took 250 mL of sample every 60,000 gallons.

The composite samplers were cleaned for priority pollutant sampling prior to the inspection (Table 1). Transfer blank samples were taken for total organic carbon (TOC), volatile organic compounds (VOCs), and metals analyses.

Effluent grab samples for fecal coliform, volatile organic compounds, and oil and grease were collected at the end of the chlorine contact chamber. Hand composites, consisting of three consecutive grab samples of unchlorinated effluent, were taken for bioassay tests. They were collected at a wet well between the secondary clarifier and the chlorine contact chamber (Figure 2).

Primary and secondary sludge are combined and thickened, then dewatered in a sludge filter press. Grab composites of dewatered sludge were collected at the end of the belt press as cake dropped into a hauler truck.

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

## QUALITY ASSURANCE/QUALITY CONTROL

Laboratory quality assurance and quality control (QA/QC) methods, which were followed during analyses of general chemistry parameters and priority pollutants, are described by Huntamer and Hyre (1991) and Kirchmer (1988). Recommended holding times were met for all analyses performed except the sludge BNA analysis. The sample was extracted one day beyond the 14 day holding time. However, this delay should not have a measurable effect on the BNA results (Magoon, 1991).

For VOC analyses, the gas chromatograph/mass spectrometer (GC/MS) met contact laboratory protocol (CLP) requirements (EPA, 1990b). All initial and continuing calibration verification standards were within the control limit of  $\pm 10\%$ . All spike recoveries for metals in water and sludge were within the acceptable limits of  $\pm 25\%$ , except for mercury, arsenic, selenium, and thallium, which were low. In sludge metal analyses, results are flagged with a "J" qualifier as estimates due to matrix interferences. For organics analyses, matrix spike/spike duplicate recovery and precision data were reasonable and acceptable, and within QC limits, with two minor exceptions. Spike recoveries for BNAs and VOCs were slightly high; however, no further action or qualifiers were required (Magoon, 1991).

Table 1. Priority Pollutant Cleaning and Field Transfer Blank Procedures - Miller Creek WTP, 4/91

Priority Pollutant Sampling Equipment Cleaning Procedure

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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.
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Field Transfer Blank Procedure

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1. Pour organic free water directly into appropriate bottles for parameters to be analyzed from grab samples, namely 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 put the water into appropriate bottles for parameters to be analyzed from composite samples, namely priority pollutant metals.
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Table 2. Sampling Schedule and Parameters Analyzed – Miller Creek WTP, 4/91

Location:	Inf-E	Inf-MC	Inf-1	Inf-2	Eff-E	Eff-ER	Eff-MC	Eff-1	Eff-2	Effluent	Sludge	Blank	
Type:	comp	comp	grab	grab	comp	comp	comp	grab	grab	grab-comp	grab-comp	trans	
Date:	23-24	23-24	4/23	4/24	23-24	23-24	23-24	4/23	4/24	23-24	4/22	4/23	
Time:	0800-0800	0800-0800	0925	1235	0830-0830	0830-0830	0830-0830	1320	1125	1200-1200	1325	0950	
Parameter	Lab Log #1781:	-05	-06	-07	-08	-09	-10	-11	-12	-13	-14	-15	-16
<b>GENERAL CHEMISTRY</b>													
Conductivity		E	E	E	E	E	E	E	E	E	E		
Alkalinity		E	E			E	E	E			E		
Hardness		E	E			E	E	E			E		
SOLIDS 4		E/MC	E/MC			E/MC	E	E/MC			E		
% Solids												E	
% Volatile Solids												E	
BOD5		E/MC	E/MC			E/MC	E	E/MC					
BOD Soluble		E	E			E	E	E					
TOC (water)		E	E			E	E	E				E	
TOC (sludge)												E	
NH3-N		E	E			E	E	E					
NO2+NO3-N		E	E			E	E	E					
NO2-N		E	E			E	E	E					
NO3-N		E	E			E	E	E					
Phosphorus - Total		E	E			E	E	E					
Oil and Grease				E	E				E	E			
F-Coliform MPN									E	E		E	
T-Coliform MPN (sludge)												E	
<b>ORGANICS and METALS</b>													
VOCs (water)				E					E			E	
VOCs (sludge)												E	
BNAs (water)		E				E							
BNAs (sludge)												E	
Pest/PCBs (water)						E							
PP Metals		E				E		E				E	
<b>BIOASSAYS</b>													
Rainbow Trout (acute)												E	
Fathead Minnow (chronic)												E	
<b>FIELD OBSERVATIONS</b>													
Temp		E	E	E	E	E	E	E	E	E	E	E	
pH		E	E	E	E	E	E	E	E	E	E	E	
Conductivity		E	E	E	E	E	E	E	E	E	E	E	
Chlorine Free									E	E			
Total									E	E			

Inf - Influent, E - Ecology sample, Eff - Effluent, ER - Replicate of E, MC - Miller Creek sample

Ecology collected replicate samples at the effluent station (Eff-ER) to quantify variability of results. A selected number of parameters were analyzed for these samples and the results were in good agreement (Table 3).

## RESULTS and DISCUSSION

### Flow

Physical measurements taken of the 24" Parshall flume showed it was correctly installed and calibrated. Instantaneous flow was recorded during the inspection by having one inspector take the depth measurement at the flume while another inspector recorded the plant flow meter reading. Flow for a given flume depth was obtained from a table found in the ISCO® Open Channel Flow Measurement Handbook (1985). Comparison of Ecology's instantaneous flow measurement to the WTP effluent flow meter reading was reasonably good. MC totalizer readings for a 24-hour time period beginning at 0800 on April 23, 1991, indicated 3.55 MGD; this flow was used to calculate mass loadings for permit parameters.

### General Chemistry and NPDES Permit Compliance

Conventional pollutant data collected during the inspection are tabulated in Table 3. MC's wastewater treatment plant performed well during the inspection. BOD<sub>5</sub> and TSS results indicated a well-treated effluent.

A comparison of effluent parameters to NPDES permit limits is presented in Table 4. The effluent met permit limits for BOD<sub>5</sub>, TSS, fecal coliform, and pH. The permit also specifies that when the actual flow or waste load reaches 85% of design capacity, the permittee shall submit to the department a plan and schedule for continuing to maintain adequate capacity. Table 4 indicates that BOD<sub>5</sub> and flow loadings were within the criteria, however, TSS loading (based on Ecology result of 250 mg/L) exceeded the design criterion.

### 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 stream. Among BNAs, only bis(2-ethylhexyl)phthalate was detected.

A listing of priority pollutant metals detected in transfer blank and effluent samples is presented in Table 5. Most metals detected were at a concentration less than acute and chronic marine water quality criteria. Among these metals, copper was found at 14 µg/L, silver was detected at an estimated value of 3.8 µg/L, and mercury was present in Ecology's composite sample at about 0.07 µg/L. All three metals concentrations were above the acute and chronic marine water quality criteria (EPA, 1986a).

Table 3. Summary of General Chemistry - Miller Creek WTP, 4/91

Parameters	Station:	Inf-E	Inf-MC	Inf-1	Inf-2	Eff-E	Eff-ER	Eff-MC	Eff-1	Eff-2	Effluent	Sludge	Blank
	Type:	comp	comp	grab	grab	comp	comp	comp	grab	grab	grab-comp	grab-comp	trans
	Time:	0800-0800	0800-0800	0925	1235	0830-0830	0830-0830	0830-0830	0830-0830	1125	1200-1200	1325	0950
	Date:	4/23-24	4/23-24	4/23	4/24	4/23-24	4/23-24	4/23-24	4/23	4/24	4/23-24	4/22	4/23
	Lab ID#1781:	-05	-06	-07	-08	-09	-10	-11	-12	-13	-14	-15	-16
<b>GENERAL CHEMISTRY</b>													
Conductivity, umhos/cm		636	639	471	528	650	652	649	628	542	676		
Alkalinity, mg/L CaCO3		137	139			92.6	91.4	89.1			86		
Hardness, mg/L CaCO3		70.4	68.4			76.4	75.9	75.4			80		
TS, mg/L		553	543			423	432	483			444		
TNVSS, mg/L		22	16			1	1	2			3		
TSS, mg/L		250	127			7	9	9			7		
TNVS, mg/L		318	308			324	266	230			311		
% Solids													17.3
% Volatile Solids													70.7
BOD5, mg/L		160	150			13	15	12					
BOD Sol, mg/L		95	100			11	10	10					
TOC (water), mg/L		70	51			24	23	21					1.0 U
TOC (soil)													31.7*
NH3-N, mg/L		19.1	19.5			14.4	11.60	10.85					
NO2+NO3-N, mg/L		0.19	0.02			6.03	5.97	6.07					
NO2-N, mg/L		0.013	0.008			0.88	0.95	0.91					
NO3-N, mg/L		0.18	0.008			5.16	5.02	5.16					
Phosphorus-T, mg/L		5.13	4.84			4.82	4.84	4.78					
Oil & Grease, mg/L				50.4	42.9				3.3	1.8			
F-Coliform, MPN #/100 mL									10 BOF	61			
F-Coliform, MPN #/100 mg													700,000
T-Coliform, MPN #/100 mg													1,400,000
<b>FIELD OBSERVATIONS</b>													
Temp., deg. C		4.1+	16.0+	14.1	15.5	5.3+	2.9+	7.5+	14.3	14.5	8.5		
pH, S.U.		7.61	7.25	7.43	7.82	7.31	7.27	7.36	6.98	7.16	7.26		
Conductivity, umhos/cm		530	430	390	420	580	580	540	520	500	520		
Chlorine Residual, mg/L													
Free									0.2	0.3			
Total									0.2	0.5			

U - The analyte was not detected at the reported limit.

BOF - Bottle overfull; couldn't shake sample.

\* - % dry weight.

+ - Iced composite sample.

Table 4. Comparison of Inspection Results to NPDES Permit Limits - Miller Creek WTP, 4/91

Parameter	NPDES Permit Li Inspection Data			Design Criteria	85 % of DC	Plant Loading	
	Monthly Average	Weekly Average	Ecology Composite			Inspection Results	% of DC
Influent BOD5 (mg/L)			160				
(lbs/day)				6,700	5,695	4,714	70
Effluent BOD5 (mg/L)	30*	45	13				
(lbs/day)	1,005	1,510				384	
(% removal)	85					92	
Influent TSS (mg/L)			250				
(lbs/day)				7,100	6,035	7,366	104
Effluent TSS (mg/L)	30*	45	7				
(lbs/day)	1,065	1,600				206	
(% removal)	85					97	
Fecal Coliform (#/100 mL)	200^	400	10;61				
pH (S.U.)	6.0-9.0 (all times)		6.98-7.16				
Flow (MGD)				4.8+	4.08	3.55	74

\* or 15% of the respective influent concentrations, whichever is more stringent.

^ The average for fecal coliform bacteria is based on the geometric mean of the sample taken.

+ Maximum monthly flow.

DC Design Criteria

Table 5. Results of Effluent and Blank Metals Analyses - Miller Creek WTP, 4/91

Metals (ug/L)	Field Station:	Eff-E	Eff-MC	Marine Water Criteria*	
	Type:	comp	comp	Acute	Chronic
	Date:	23-24	23-24		
	Time:	24 hr	24 hr		
	Lab sample#:	178109	178111		
		Tot. Rec.	Tot. Rec.		
Copper		14	13	2.9	2.9
Mercury		0.07 P	-	2.1	0.025
Silver		3.8 J	3.8 J	2.3	-
Zinc		39	37	95	86

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

J - The analyte was positively identified. The associated numerical result is an estimate.

P - The analyte was detected above the instrument detection limit but below the established minimum quantitation limit.

## Effluent Bioassays

Bioassays determine the relative toxicity of WTP effluent by measuring the response of organisms to solutions containing various percentages of effluent and dilution water. For this inspection, rainbow trout and fathead minnow larvae were used as test organisms. Results are given in Table 6. No effluent toxicity was indicated by rainbow trout. A seven-day survival and growth test using fathead minnow larvae resulted in 67.5% survival in 100% effluent. No Observed Effects Concentration (NOEC) and Lowest Observed Effects Concentration (LOEC) for the test were 50% and 100%, respectively.

## Sludge Analyses

General chemistry data for the sludge sample collected during the inspection are listed in Table 3. Percent volatile solids (VS) reduction was determined through a simple mass balance on VS across the anaerobic digester using the final sludge product per day. The calculation<sup>1</sup> estimated a 80% volatile solids reduction in the digester. According to the proposed EPA regulations, 40 CFR part 503, a sludge is considered to have adequately reduced vector attraction if its volatile solids are reduced by 38% (EPA 1989a).

No BNAs were detected in sludge samples (Appendix C). Metals and volatile organic compounds detected in sludge samples are listed in Table 7. Twelve priority pollutant metals (ranging from 0.24-1,348 mg/kg-dry) were detected. Federal guidelines for land application of sludge were proposed in a draft form in 1990 (EPA, 1990a). Compared to these guidelines, only the cadmium concentration exceeded the National Sewage Sludge Survey geometric mean value (Table 7). Seven volatile organic compounds (ranging from 33-12,000  $\mu\text{g}/\text{kg-dry}$ ) were detected. Priority pollutant analysis of the sludge may be necessary after final regulations are promulgated.

## Laboratory Review

Table 8 shows a comparison of data resulting from the four-way split of composite samples during the inspection. Results from samples collected (e.g., influent) by two different compositors (Ecology and MC) but analyzed at the same lab (e.g., Ecology) address the issue of sample representativeness. For the example presented, BOD<sub>5</sub> data were 160 vs 150 mg/L;

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$$^1\text{Volatile solids reduction} = [(QY)_{\text{raw}} - (QY)_{\text{digested}}] / (QY)_{\text{raw}};$$

where: Q = volumetric sludge flow rate in MGD,

Y = volatile solids concentration, mg/L;

$$\text{Volatile solids reduction} = [(0.011 \times 0.051 \times 0.84) - (0.005 \times 0.028 \times 0.68)] /$$

$$(0.011 \times 0.051 \times 0.84) \approx 80\%;$$

^ Note: data obtained from the MC plant operator (Yokers, 1992).

Table 6. Effluent Bioassay Results - Miller Creek WTP, 4/91

Rainbow trout - 96 hr survival test

Lab ID# 178114

Sample (% vol)	# Tested <sup>^</sup>	Percent Survival
Control	30	100
100	30	100

<sup>^</sup> - Three replicates of ten organisms.

LC50 - Cadmium chloride reference toxicant was estimated at 2.7 µg/L.

Fathead Minnow larvae - 7 day Survival and Growth Test

Sample (% vol)	# Tested*	% Survival	Average Weight per larvae, mg
Control	40	97.5	0.49
1.56	40	95	0.41
3.13	40	95	0.41
6.25	40	100	0.47
12.5	40	75	0.37
25	40	82.5	0.36
50	40	82.5	0.40
100	40	67.5	0.43

\* - Four replicates of ten organisms.

NOEC - 6.25% effluent.

LOEC - 12.5% effluent.

LC50% - Cadmium chloride reference toxicant was estimated at 20 µg/L.

Table 7. Results of Sludge Priority Pollutant Metals and VOC Analyses - Miller Creek WTP, 4/91

**Metals**

Parameters (mg/kg-dry)	Field Station:	Sludge	National Sewage Sludge Survey +		
	Type:	grab-comp	Number of Samples	Percent Detected + +	Geometric Mean
Lab sample#:	Date:	178115			
	Time:	4/22			
		1325			
		178115			
Antimony		4.0 P	--	--	--
Arsenic		4.9 J	70	83	9.72
Beryllium		0.24 P	70	36	0.48
Cadmium		14.6	69	78	9.16
Chromium		56.3	70	99	160.57
Copper		490	70	100	670.68
Lead		112	70	87	156.99
Mercury		1.05 J	70	79	3.96
Nickel		43.1	70	81	48.36
Selenium		4.72 J	70	64	5.59
Silver		84.9	--	--	--
Thallium		0.25 UJ	--	--	--
Zinc		1,348	70	100	1707.99

**Volatile Organic Compounds (VOCs)**

Parameters ( $\mu\text{g}/\text{kg-dry}$ )	Field Station:	Sludge
	Type:	grab-comp
Lab sample#:	Date:	4/22
	Time:	1325
	Lab sample#:	178115
VOCs		
Acetone		12,000
Methylene Chloride		60
2-Butanone (MEK)		410
Toluene		78
Chlorobenzene		33 J
Total Xylenes		250
1,4-Dichlorobenzene		41 J

- 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.
- P - The analyte was detected above the instrument detection limit but below the established minimum quantification limit.
- UJ - The analyte was not detected at or above the reported estimated result.
- + - EPA, 1990. Values presented are for WTPs with flows between 1 and 10 MGD.
- + + - Percent of samples in which the compound was detected above the quantification limit.



Table 8. Comparison of Laboratory Results of Sample Splits – Miller Creek WTP, 4/91

Sample	Sampler	Laboratory	BOD5 (mg/L)	Soluble BOD5 (mg/L)	TSS (mg/L)
Inf-E (178105)	Ecology	Ecology	160	95	250
		Miller Creek	176	86	158
Inf-MC (178106)	Miller Creek	Ecology	150	100	127
		Miller Creek	168	86	163
Eff-E (178109)	Ecology	Ecology	13	11	7
		Miller Creek	12.4	5.8	8.6
Eff-MC (178111)	Miller Creek	Ecology	12	10	9
		Miller Creek	10.8	4.2	7.8

TSS data were 250 vs 127 mg/L. These BOD<sub>5</sub> data show good agreement. On the other hand, TSS data reveal a major disparity, which indicates that one of the samples was not representative of the wastewater. The cause of this disparity is unclear.

Results from samples collected (e.g., influent) by the same compositor but analyzed at two different labs (Ecology and MC) address the issue of lab performance. For the example presented, influent BOD<sub>5</sub> data were 160 vs 176 mg/L; influent TSS data were 250 vs 158 mg/L. No conclusion can be drawn from these limited data on influent TSS. Therefore, in addition to four-way splits, a performance evaluation (PE) sample should be analyzed in the future to help compare lab performance.

Dale Van Donsel and Perry Brake of Ecology's Quality Assurance Section conducted an on-site laboratory evaluation on April 23, 1991. Their report indicates that the MC's laboratory is providing reliable analytical data to the Department. Their complete audit report is included in Appendix D.

## CONCLUSIONS AND RECOMMENDATIONS

### Conclusions

1. MC's Parshall flume was properly installed and calibrated. Comparison of Ecology's instantaneous flow measurement to the WTP effluent flow reading was good.
2. The wastewater treatment plant performed well during the inspection. Conventional parameters indicated a well-treated, high quality effluent. The WTP was meeting permit limits for BOD<sub>5</sub>, TSS, fecal coliform, and pH during the inspection.
3. No volatile organic compound or pesticides/PCBs were detected in WTP effluent. However, a BNA compound, bis(2-ethylhexyl)phthalate, and a number of priority pollutant metals were detected. Among the metals detected, copper, silver, and mercury concentrations in effluent exceeded both acute and chronic marine water quality criteria.
4. No significant effluent toxicity was indicated by using rainbow trout and fathead minnow larvae.
5. No BNAs were detected in the sludge sample. However, several volatile organic compounds (ranging from 33-12,000 µg/kg-dry) were detected. All 13 priority pollutant metals were also detected at concentrations ranging from 0.24-1,348 mg/kg-dry.
6. Both Ecology and MC laboratory results of split samples for permit parameters agreed reasonably well. However, Ecology's influent TSS result (250 mg/L) was much higher than MC's reported result (127 mg/L). An on-site review of MC's laboratory procedures did not indicate any serious procedural problems in sample collection and analyses.

7. Under the proposed EPA 503 regulations, MC's processed sludge did meet the requirement of 38% volatile solids reduction for land application.
8. Table 4 shows that during the inspection, BOD<sub>5</sub> loading (4,714 lbs) and flow (3.55 MGD) to the plant were well within the design criteria. However, TSS loading (7,366 lbs, based on Ecology lab result of 250 mg/L) exceeded the design criterion.

### **Recommendations**

1. Field observation data indicated that MC influent and effluent sample temperatures were much higher than recommended 4°C (Table 3). The likely cause of this was that MC's sample cooler was not adequately functioning. The cooling system should be inspected and repaired as necessary.
2. Priority pollutant metals analyses on the sludge may be necessary after National Sewage Sludge Survey final regulations are promulgated.
3. Exceedance of the design criteria for TSS loading indicates the need to begin planning for an upgrade. However, this recommendation should be considered tentative until the accuracy of TSS test results can be verified.

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## APPENDICES

## Appendix A. Chemical Analytical Methods - Miller Creek WTP, 4/91

Parameters	Method	Lab Used
<b>GENERAL CHEMISTRY</b>		
Conductivity	EPA, 1979: 120.1	Ecology; Manchester, WA
Alkalinity	EPA, 1979: 310.1	Ecology; Manchester, WA
Hardness	EPA, 1979: 130.2	Ecology; Manchester, WA
<b>SOLIDS 4</b>		
TS	EPA, 1979: 160.3	Ecology; Manchester, WA
TNVS	EPA, 1979: 106.4	Ecology; Manchester, WA
TSS	EPA, 1979: 160.2	Ecology; Manchester, WA
TNVSS	EPA, 1979: 106.4	Ecology; Manchester, WA
% Solids	APHA, 1989: 2540G	AMTest Inc.; Redmond, WA
% Volatile Solids	EPA, 1979: 160.4	AMTest Inc.; Redmond, WA
BOD5	EPA, 1979: 405.1	AMTest Inc.; Redmond, WA
BOD5 Soluble	EPA, 1979:405.1	AMTest Inc.; Redmond, WA
TOC (water)	EPA, 1979: 415.2	AMTest Inc.; Redmond, WA
TOC (soil)	APHA, 1989: 5310	AMTest Inc.; Redmond, WA
<b>NUTRIENTS</b>		
NH3-N	EPA, 1979: 350.1	AMTest Inc.; Redmond, WA
NO2 + NO3-N	EPA, 1979: 353.2	AMTest Inc.; Redmond, WA
NO2-N	EPA, 1979: 353.2	AMTest Inc.; Redmond, WA
NO3-N	EPA, 1979: 352.2	AMTest Inc.; Redmond, WA
Phosphorus - Total	EPA, 1979: 365.1	AMTest Inc.; Redmond, WA
Oil and Grease	EPA, 1979: 413.1	Ecology; Manchester, WA
F-Coliform MPN	APHA, 1989:908C	Ecology; Manchester, WA
T-Coliform (sludge)	APHA, 1989:9222B	Ecology; Manchester, WA
<b>ORGANICS</b>		
VOCs (water)	EPA, 1984: 624	Columbia Analytical Services Inc.; Kelso, WA
VOCs (sludge)	EPA, 1986b: 8240	Columbia Analytical Services Inc.; Kelso, WA
BNAs (water)	EPA, 1984: 625	Columbia Analytical Services Inc.; Kelso, WA
BNAs (sludge)	EPA, 1986b: 8270	Columbia Analytical Services Inc.; Kelso, WA
Pest/PCBs (water)	EPA, 1984: 608	Columbia Analytical Services Inc.; Kelso, WA
<b>METALS</b>		
PP Metals		
Total (water)	EPA, 1979: 200	Ecology; Manchester, WA
Total (sludge)	EPA, 1979: 200	Ecology; Manchester, WA
<b>BIOASSAYS</b>		
Rainbow Trout (acute)	Ecology, 1981	Ecology; Manchester, WA
Fathead Minnow (chronic)	EPA, 1989b	Ecology; Manchester, WA

Appendix B. Results of Influent & Effluent Pesticide/PCBs and Priority Pollutant Metals Analyses - Miller Creek WTP, 4/91

Parameter ( $\mu\text{g/L}$ )	Field Station:	Eff-E
	Type:	comp
	Date:	4/23-24
	Time:	0830-0830
	Lab sample#:	178109
alpha-BHC		0.04 U
gamma-BHC (Lindane)		0.04 U
beta-BHC		0.1 U
Heptachlor		0.04 U
delta-BHC		0.04 U
Aldrin		0.04 U
Heptachlor Epoxide		0.04 U
Endosulfan I		0.04 U
4,4'-DDE		0.04 U
Dieldrin		0.04 U
Endrin		0.04 U
4,4'-DDD		0.04 U
Endosulfan II		0.04 U
4,4'-DDT		0.04 U
Endrin Ketone		0.04 U
Endosulfan Sulfate		0.04 U
Methoxychlor		0.1 U
Toxaphene		1.0 U
alpha-Chlordane		0.5 U
gamma-Chlordane		0.2 U
Aroclor-1016		0.2 U
Aroclor-1221		0.2 U
Aroclor-1232		0.2 U
Aroclor-1242		0.2 U
Aroclor-1248		0.2 U
Aroclor-1254		0.2 U
Aroclor-1260		0.2 U

	Field Station:	Inf-E	Eff-E	Eff-MC	Blank
	Type:	comp	comp	comp	trans
	Date:	4/23-24	4/23-24	4/23-24	4/23
	Time:	0800-0800	0830-0830	0830-0830	0950
	Lab sample#1781	-05	-09	-11	-16

Metals ( $\mu\text{g/L}$ )	Inf-E	Eff-E	Eff-MC	Blank
Antimony	30 UJ	30 UJ	30 UJ	30 UJ
Arsenic	1.5 UJ	1.5 UJ	1.5 UJ	1.5 UJ
Beryllium	1.0 U	1.0 U	1.0 U	1.0 U
Cadmium	2.0 U	2.0 U	2.0 U	2.0 U
Chromium	5.0 U	5.0 U	5.0 U	5.0 U
Copper	34.2	14	13	2.0 U
Lead	20 U	20 U	20 U	20 U
Mercury	0.93	0.07 P	0.04 U	0.04 U
Nickel	10 U	10 U	10 U	10 U
Selenium	2.0 U	2 U	2 U	2 U
Silver	6.4 J	3.8 J	3.8 J	2.0 UJ
Thallium	2.5 U	2.5 U	2.5 U	2.5 U
Zinc	81.9	39	37	4.0 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.

UJ - The analyte was not detected at or above the reported estimated result.

P - The analyte was detected above the instrument detection limit but below the established minimum quantitation limit.

Shaded area denotes metals detected.



Appendix B (Cont.) Results of Influent and Effluent BNA Analyses - Miller Creek WTP, 4/91

		Field Station:	Inf-E	Eff-E
		Type:	comp	comp
		Date:	4/23-24	4/23-24
		Time:	0800-0800	0830-0830
Parameters ( $\mu\text{g/L}$ )	Lab sample#1781:		-05	-09
N-Nitrosodiphenylamine			5 U	5 U
Bis(2-Chloroethyl)Ether			5 U	5 U
1,3-Dichlorobenzene			5 U	5 U
1,4-Dichlorobenzene			5 U	5 U
1,2-Dichlorobenzene			5 U	5 U
Bis(2-chloroisopropyl)ether			5 U	5 U
N-Nitroso-Di-n-Propylamine			5 U	5 U
Hexachloroethane			5 U	5 U
Nitrobenzene			5 U	5 U
Isophorone			5 U	5 U
Bis(2-Chloroethoxy)Methane			5 U	5 U
1,2,4-Trichlorobenzene			5 U	5 U
Naphthalene			5 U	5 U
4-Chloroaniline			5 U	5 U
Hexachlorobutadiene			10 U	10 U
4-Chloro-3-methylphenol			5 U	5 U
2-Methylnaphthalene			5 U	5 U
Hexachlorocyclopentadiene			5 U	5 U
2-Chloronaphthalene			5 U	5 U
2-Nitroaniline			20 U	20 U
Dimethyl Phthalate			5 U	5 U
Acenaphthylene			5 U	5 U
3-Nitroaniline			20 U	20 U
Acenaphthene			5 U	5 U
Dibenzofuran			5 U	5 U
2,4-Dinitrotoluene			5 U	5 U
2,6-Dinitrotoluene			5 U	5 U
Diethyl Phthalate			8 J	5 U
4-Chlorophenyl-Phenylether			5 U	5 U
Fluorene			5 U	5 U
4-Nitroaniline			20 U	20 U
4-Bromophenyl-Phenylether			5 U	5 U
Hexachlorobenzene			5 U	5 U
Phenanthrene			5 U	5 U
Anthracene			5 U	5 U
Dibutylphthalate			5 U	5 U
Fluoranthene			5 U	5 U
Pyrene			5 U	5 U
Butylbenzylphthalate			5 U	5 U

U - None detected at or above the method reporting limit.

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

Shaded area denotes compound detected.

Appendix B (Cont.) Results of Influent and Effluent BNA Analyses - Miller Creek WTP, 4/91

Field Station:	Inf-E	Eff-E
Type:	comp	comp
Date:	4/23-24	4/23-24
Time:	0800-0800	0830-0830
Lab sample#1781:	-05	-09
3,3'-Dichlorobenzidine	20 U	20 U
Benzo(a)Anthracene	5 U	5 U
Di-n-Octyl Phthalate	5 U	5 U
Benzo(b)Fluoranthene	5 U	5 U
Benzo(k)Fluoranthene	5 U	5 U
Benzo(a)Pyrene	5 U	5 U
Indeno(1,2,3-cd)Pyrene	5 U	5 U
Dibenzo(a,h)Anthracene	5 U	5 U
Benzo(g,h,i)Perylene	5 U	5 U
Phenol	16	5 U
2-Chlorophenol	5 U	5 U
Benzyl Alcohol	17	5 U
2-Methylphenol	5 U	5 U
4-Methylphenol	27	5 U
2-Nitrophenol	5 U	5 U
2,4-Dimethylphenol	5 U	5 U
Benzoic Acid	50 U	50 U
2,4-Dichlorophenol	5 U	5 U
2,4,6-Trichlorophenol	5 U	5 U
2,4,5-Trichlorophenol	5 U	5 U
2,4-Dinitrophenol	50 U	50 U
4-Nitrophenol	20 U	20 U
4,6-Dinitro-2-Methylphenol	20 U	20 U
Pentachlorophenol	30 U	30 U

U - None detected at or above the method reporting limit.

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

Shaded area denotes compound detected.

Appendix B (Cont.) Results of Influent and Effluent VOC Analysis - Miller Creek WTP, 4/91

	Field Station:	Inf-1	Eff-1	Blank
	Type:	grab	grab	trans
	Date:	4/23-24	4/23-24	4/23
	Time:	0925	1320	0950
Parameters ( $\mu\text{g/L}$ )	Lab sample#1781:	-07	-12	-16
Chloromethane		1 U	1 U	1 U
Vinyl Chloride		1 U	1 U	1 U
Bromomethane		1 U	1 U	1 U
Chloroethane		1 U	1 U	1 U
1,2-Dichloroethene (total)		1 U	1 U	1 U
1,1-Dichloroethene		1 U	1 U	1 U
Acetone		77	20 U	20 U
Carbon Disulfide		1 U	1 U	1 U
Methylene Chloride		10 U	10 U	10 U
2-Butanone		50 U	50 U	50 U
1,1-Dichloroethane		1 U	1 U	1 U
Chloroform		5.0 J	1 U	1 U
1,1,1-Trichloroethane		1 U	1 U	1 U
Carbon Tetrachloride		1 U	1 U	1 U
Benzene		1 U	1 U	1 U
1,2-Dichloroethane		1 U	1 U	1 U
Vinyl Acetate		10 U	10 U	10 U
Trichloroethene		1 U	1 U	1 U
1,2-Dichloropropane		1 U	1 U	1 U
Bromodichloromethane		1 U	1 U	1 U
Trans-1,3-Dichloropropene		1 U	1 U	1 U
2-Hexanone		1 U	1 U	1 U
4-Methyl-2-Pentanone		10 U	10 U	10 U
Toluene		1 U	1 U	1 U
cis-1,3-Dichloropropene		1 U	1 U	1 U
1,1,2-Trichloroethane		1 U	1 U	1 U
Tetrachloroethene (PCE)		1.5 J	1 U	1 U
Dibromochloromethane		1 U	1 U	1 U
Chlorobenzene		1 U	1 U	1 U
Ethylbenzene		1 U	1 U	1 U
Styrene		1 U	1 U	1 U
Total Xylenes		1 U	1 U	1 U
Bromoform		1 U	1 U	1 U
1,1,2,2-Tetrachloroethane		1 U	1 U	1 U
1,3-Dichlorobenzene		1 U	1 U	1 U
1,4-Dichlorobenzene		1.5 J	1 U	1 U
1,2-Dichlorobenzene		1 U	1 U	1 U

U - None detected at or above the method reporting limit.

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

Shaded area denotes compound detected.

Appendix C. Results of Sludge Priority Pollutant Metals Analyses - Miller Creek WTP, 4/9

Field Station:	Sludge
Type:	grab-comp
Date:	4/22
Time:	1325
Lab sample#:	178115
<hr/>	
Metals (mg/kg-dry)	
Antimony	4.0 P
Arsenic	4.9 J
Beryllium	0.24 P
Cadmium	14.6
Chromium	56.3
Copper	490
Lead	112
Mercury	1.05 J
Nickel	43.1
Selenium	4.72 J
Silver	84.9
Thallium	0.25 UJ
Zinc	1348

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

UJ - The analyte was not detected at or above the reported estimated result.

P - The analyte was detected above the instrument detection limit but below the established minimum quantitation limit.

Shaded area denotes metal detected.

Appendix C (Cont.) Results of Sludge BNA Analysis – Miller Creek WTP, 4/91

	Field Station:	Sludge
	Type:	grab-comp
	Date:	4/22
	Time:	1325
Parameter ( $\mu\text{g/L}$ )	Lab sample #:	178115
N-Nitrosodiphenylamine		20 U
Bis(2-Chloroethyl)Ether		20 U
1,3-Dichlorobenzene		20 U
1,4-Dichlorobenzene		20 U
1,2-Dichlorobenzene		20 U
Bis(2-Chloroisopropyl)ether		20 U
N-Nitroso-Di-n-Propylamine		20 U
Hexachloroethane		20 U
Nitrobenzene		20 U
Isophorone		20 U
Bis(2-Chloroethoxy)Methane		20 U
1,2,4-Trichlorobenzene		20 U
Naphthalene		20 U
4-Chloroaniline		20 U
Hexachlorobutadiene		20 U
4-Chloro-3-methylphenol		20 U
2-Methylnaphthalene		20 U
Hexachlorocyclopentadiene		20 U
2-Chloronaphthalene		20 U
2-Nitroaniline		100 U
Dimethyl Phthalate		20 U
Acenaphthylene		20 U
3-Nitroaniline		100 U
Acenaphthene		20 U
Dibenzofuran		20 U
2,4-Dinitrotoluene		20 U
2,6-Dinitrotoluene		20 U
Diethyl Phthalate		20 U
4-Chlorophenyl-Phenylether		20 U
Fluorene		20 U
4-Nitroaniline		100 U
4-Bromophenyl-Phenylether		20 U
Hexachlorobenzene		20 U
Phenanthrene		20 U
Anthracene		20 U
Dibutylphthalate		20 U
Fluoranthene		20 U
Pyrene		20 U
Butylbenzylphthalate		20 U
3,3'-Dichlorobenzidine		20 U
Benzo(a)Anthracene		20 U
Bis(2-Ethylhexyl)phthalate		20 U
Chrysene		20 U

U - None detected at or above the method reporting limit.

Appendix C (Cont.) Results of Sludge BNA Analysis – Miller Creek WTP, 4/91

Parameter ( $\mu\text{g/L}$ )	Field Station: Type: Date: Time: Lab sample #:	Sludge grab-comp 4/22 1325 178115
Di-n-Octyl Phthalate		20 U
Benzo(b)Fluoranthene		20 U
Benzo(k)Fluoranthene		20 U
Benzo(a)Pyrene		20 U
Indeno(1,2,3-cd)Pyrene		20 U
Dibenzo(a,h)Anthracene		20 U
Benzo(g,h,i)Perylene		20 U
Phenol		20 U
2-Chlorophenol		20 U
Benzyl Alcohol		20 U
2-Methylphenol		20 U
4-Methylphenol		20 U
2-Nitrophenol		20 U
2,4-Dimethylphenol		20 U
Benzoic Acid		100 U
2,4-Dichlorophenol		20 U
2,4,6-Trichlorophenol		20 U
2,4,5-Trichlorophenol		20 U
2,4-Dinitrophenol		100 U
4-Nitrophenol		100 U
4,6-Dinitro-2-Methylphenol		100 U
Pentachlorophenol		100 U

U - None detected at or above the method reporting limit.

Appendix C (Cont.) Results of Sludge VOC Analysis – Miller Creek WTP, 4/91

Parameter ( $\mu\text{g}/\text{kg-dry}$ )	Field Station: Type: Date: Time: Lab sample #:	Sludge grab-comp 4/22 1325 178115
Chloromethane		25 U
Vinyl Chloride		25 U
Bromomethane		25 U
Chloroethane		25 U
1,2-Dichloroethene (total)		25 U
1,1-Dichloroethene		25 U
Acetone		12000
Carbon Disulfide		25 U
Methylene Chloride		60
2-Butanone (MEK)		410
1,1-Dichloroethane		25 U
Chloroform		25 U
1,1,1-Trichloroethane		25 U
Carbon Tetrachloride		25 U
Benzene		25 U
1,2-Dichloroethane		25 U
Vinyl Acetate		50 U
Trichloroethene		25 U
1,2-Dichloropropane		25 U
Bromodichloromethane		25 U
Trans-1,3-Dichloropropene		25 U
2-Hexanone		50 U
4-Methyl-2-Pentanone		50 U
Toluene		78
cis-1,3-Dichloropropene		25 U
1,1,2-Trichloroethane		25 U
Tetrachloroethene (PCE)		25 U
Dibromochloromethane		25 U
Chlorobenzene		33 J
Ethylbenzene		25 U
Styrene		25 U
Total Xylenes		250
Bromoform		25 U
1,1,2,2-Tetrachloroethane		25 U
1,3-Dichlorobenzene		25 U
1,4-Dichlorobenzene		41 J
1,2-Dichlorobenzene		25 U

U – None detected at or above the method reporting limit.

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


Shaded area denotes compound detected.



STATE OF WASHINGTON  
DEPARTMENT OF ECOLOGY

Post Office Box 488 • Manchester, Washington 98353-0488 • (206) 895-4649

April 26, 1991

TO: Norm Glenn  
FROM: Cliff Kirchmer   
SUBJECT: Class II Inspection of Miller Creek WWTP Lab

Dale Van Donsel and Perry Brake of this office completed their inspection of the Miller Creek Wastewater Treatment Plant laboratory on April 23, 1991. Their report, which contains no indication that the lab is not providing reliable analytical data to the Department, is attached. A copy of the report has been furnished to the operations supervisor at the plant (Tim Yokers) to assist him in preparing for accreditation of the laboratory.

We have informed Mr. Yokers that if he submits his application for accreditation in a timely manner, we will be able to use the results of this Class II Inspection as the on-site audit for accreditation of the lab, although a follow-up visit will probably be necessary. We intend to follow this same concept for any major NPDES dischargers (e.g., Columbia Aluminum next month) since they must all start using accredited labs by July 1, next year, and are therefore expected to apply for accreditation soon (Columbia has already applied). Feel free to call upon us to assist in the Class II inspections of any majors in the near future. Conversely, after we have accredited a discharger's lab, there would appear to be no need for including the lab in the Class II inspection. Rather, the inspection report could contain a statement recognizing the lab's accreditation.



WASHINGTON STATE DEPARTMENT OF ECOLOGY  
ENVIRONMENTAL INVESTIGATIONS AND LABORATORY SERVICES  
QUALITY ASSURANCE SECTION

SYSTEM AUDIT REPORT

LABORATORY: Miller Creek Wastewater Treatment Plant Laboratory

ADDRESS: 1015 SW 174th  
Seattle, WA 98166


DATE OF AUDIT: April 23, 1991

AUDITORS: Dale Van Donsel                      Microbiology  
Perry Brake                                      General Chemistry

PERSONNEL  
INTERVIEWED: Tim Yokers                      Operations Supervisor  
Terry Hoefel                                      Operator/Lab Analyst

AUTHENTICATION:

  
\_\_\_\_\_  
Dale J. Van Donsel

  
\_\_\_\_\_  
Perry F. Brake

## GENERAL FINDINGS AND RECOMMENDATIONS

### General

1. A system audit was conducted at the Miller Creek Wastewater Treatment Plant laboratory on April 23, 1991, in conjunction with the Class II Inspection of the treatment plant. The purpose of the audit was to verify laboratory capabilities pertaining to analyses required in the treatment plant discharge permit (WA0022764) and to review analytical and quality control data. General audit findings and recommendations are documented below. Significant recommendations for improvement of laboratory operations are highlighted by use of *italics*.

2. A very significant deficiency in the overall lab operation at the Miller Creek plant lab was the lack of a formal (i.e., documented) quality assurance (QA) program designed to assure reliability of analytical data generated in the lab. *A recommendation was made to the operations supervisor that establishment of such a program and publication of a QA manual be made a high priority.* A model QA manual for a wastewater treatment plant lab was given to lab personnel and a commitment made by the visiting team to assist the lab in development of their QA program and manual.

### Personnel

3. Mr. Yokers is responsible for all analytical procedures used in the lab and is the immediate supervisor of laboratory operations. In addition to Mr. Yokers, two other plant personnel rotate between lab and other plant duties on a weekly basis (one week in the lab, two doing plant operations). Mr. Hoefel was assigned to lab duties during the lab visit. Yokers and Hoefel each have several years experience in analytical procedures and are knowledgeable in methods and techniques for which the laboratory is responsible. A third analyst is currently being trained.

### Facility

4. The lab facility consists of one large room which is also used for some administrative functions (i.e., as office space). Other administrative functions are conducted in Mr. Yokers' office which is in close proximity to the lab. Current floor and bench space is adequate to support current lab operations and efficient administrative functions. Significant expansion of lab operations to include any new analytical capability (e.g., atomic absorption, bioassay) would require additional space for efficient operations.

5. The <sup>re-</sup> were no records available to indicate the fume hood used in the lab had ever been check for adequacy of air flow. A check was made by the visiting team and the flow found to be 150 cubic feet per minute which is better than the ASTM-recommended flow of 125 CFM. *A recommendation was made*

*to have the flow checked periodically (e.g., every year) or whenever there is suspicion that flow may have been reduced for some reason. (NOTE: Air velocity measuring devices are available from several suppliers, such as the Velometer Jr., information on which was provided to the lab. The Des Moines Sewer District could purchase a meter for use in all labs in the district or a meter could be borrowed periodically from another lab or perhaps a fire department.)*

#### Equipment and Supplies

6. A log of checks on lab equipment and supplies (e.g., temperature checks on refrigerators and incubators, calibration tests on balances, conductivity tests on distilled water) was not available in the lab. *A recommendation was made that a schedule of such checks be set up and that a log be maintained as a record that the checks were being conducted.*

7. A recommendation was made for the lab to purchase a spill cleanup kit (as a safety matter and not a matter affecting quality of the analytical work done in the lab). Information on "Kolor-safe" liquid neutralizers available from Aldrich was provided to the lab.

8. A nonindicating desiccant was being used in desiccators which provided no means of determining if the dessicant was still effective. *A recommendation was made to replace the desiccant with a product such as Drierite Indicating Desiccant, available from various suppliers.*

#### Sample Management

9. Formal chain-of-custody procedures had not been established (as might be expected, given the absence of a documented QA program in the lab) to assure samples were being properly secured and accounted for from time of receipt in the lab to disposal. *A recommendation was made to establish and implement such procedures without delay to preclude potential problems should future analytical results be involved in litigation.* With minor modifications and proper documentation, sample handling procedures currently used in the lab will suffice for chain-of-custody purposes. A copy of ASTM Standard D 4840-88, "Sampling Chain of Custody Procedures," was provided to Mr. Yokers subsequent to the visit.

#### Data Management

10. Some data (particularly on bench sheets) was being recorded in pencil at the time of the audit. *A recommendation was made to record all data and observations in ink and to correct any errors by crossing out with a single line, entering the correct data, and signing or initialling the change.*

#### PE Samples

11. Performance evaluation (PE) samples for biochemical oxygen demand (BOD<sub>5</sub>), pH, and total suspended solids (TSS) were mailed to the lab prior to

the visit and analytical results were presented to the visiting team during the visit. All three parameters were successfully analyzed at two concentration levels with results as follows:

<u>PARAMETER</u>	<u>REPORTED VALUE</u>	<u>TRUE VALUE</u>
BOD <sub>5</sub>	21.3 mg/L	18.6 mg/L
	45.2 mg/L	59.7 mg/L
pH	5.82	5.80
	7.74	7.80
TSS	27 mg/L	29.7 mg/L
	41.9 mg/L	36 mg/L

#### Quality Assurance/Quality Control

12. The most significant deficiency in the quality assurance area is the lack of a formal QA program, already mentioned in paragraph 2 above. Within the QA program, the most significant deficiency is the lack of any protocol to establish data quality objectives (in terms of bias and precision, or, together, accuracy) and track the lab's capability to meet those objectives. Blind performance evaluation samples are being analyzed annually (with much success) as part of the EPA DMR-QA study program but this alone is not sufficient to determine whether or not the lab is "in control" on a continuing basis. The following recommendations were made to assist the lab in setting up a protocol to establish and track data quality objectives:

a. *The lab should establish a schedule for routinely analyzing quality control (QC) samples along with other analyses.*

(1) *First priority should go to analyzing standard solutions (solutions of known concentration) for those parameters where it is appropriate to do so. The objective in doing this QC test is to control precision as the tests are done repetitively. Over a period of time, the tests can also discover any bias in the test by comparing the observed (mean) value to the known or expected value. For the plant performance parameters reported by the Miller Creek lab, appropriate standard solution tests would be BOD (the glucose-glutamic acid solution) and TSS (using a suspension of a suitable material such as Sigma Cell 20, information on which was provided to the lab by the visiting team).*

(2) *Second priority should go to analyzing duplicate samples, preferably from the effluent stream since duplicates taken elsewhere in the plant are likely to vary widely in concentration. The objective here is to track precision of analysis on real samples (as opposed to the relatively clean standard solutions). For the plant performance parameters reported by the Miller Creek lab, appropriate duplicate tests (on effluent samples) would BOD, TSS, and pH. Duplicate tests can also be done on fecal coliforms if time and manpower resources allow.*

b. *After running sufficient QC tests to provide statistically significant data (ten tests of a given type are enough but 20 are better), control charts should be constructed and used as a means to check precision as a routine procedure.* Information on how to construct and use control charts for both standard solutions and duplicate analyses can be found in the Procedural Manual for the Environmental Laboratory Accreditation Program. Additionally, a LOTUS 1-2-3 program which automates the procedure was demonstrated and a copy given to the operations supervisor during the visit. Consistent use of control charts will provide evidence to interested parties, inside and outside the lab, concerning capability of the lab to accurately analyze environmental samples.

13. Most reagent containers were not annotated to indicate when they were received and opened and when they should be discarded. *A recommendation was made to mark each reagent container with date received, date opened, and (where known) expiration date.*

14. The thermometers being used for both the fecal coliform and BOD incubators were neither NBS (NIST) certified nor traceable to NBS certified thermometers. The BOD thermometer was calibrated against an NBS certified thermometer provided by the audit team and a certificate provided showing traceability to the certified thermometer. The fecal coliforms thermometer is discussed below.

15. Microbiology

a. It is important that the lab establish its own credibility with the fecal coliform test. EPA Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act (corrections to 40 CFR Part 136 dated January 4, 1985) state, "Since the membrane filter technique usually yields low and variable recovery from chlorinated wastewaters, the MPN method will be required to resolve any controversies." There are no equivalents of PE samples or other objective measurements for this parameter. The simplest approach for this lab is to do periodic sample splitting. Comparison of fecal coliform MPN results with this lab's membrane filter results is the verification method of choice. MPN's may be done by a laboratory accredited for this procedure by the Department of Ecology or certified by the Department of Health. The object of these comparisons is not to seek an exact comparison of numbers between the two methods, but to watch for MPN results significantly and consistently higher than the MF which would indicate failure to recover some organisms.

b. There are several steps the lab can take to improve recovery of organisms that are damaged by chlorine or "stressed". A slight modification of the M-FC medium and a change to a specialized type of membrane can help. Several other items that will improve laboratory operation are also noted.

(1) Sample Bottles. Sample bottles do not contain sodium thiosulfate for neutralization of chlorine. After fecal coliform samples

are collected, chlorine level is determined and then neutralized. This is not the recommended method for collection. Sodium thiosulfate should be added before samples are collected, so chlorine is neutralized immediately. One mL of a 1% or 0.1 mL of a 10% solution should be added to bottles before they are sterilized.

(2) Temperature Control. The 44.5°C water bath was operating at the proper temperature when checked with a reference thermometer, but the lab's own thermometers were not suitable for checking this; they were calibrated in 1° intervals. The temperature of the fecal coliform test is one of most critical elements; only a 0.2°C tolerance is allowed. It is recommended that the laboratory acquire several thermometers calibrated in 0.1 or 0.2° increments and that these be checked against a reference thermometer for accuracy.

(3) Membrane Filters. The 0.45μ membranes used for the fecal coliform test are acceptable. However, when new membranes are purchased, it is recommended that the laboratory obtain a type of filter developed for testing chlorinated effluents. The Millipore Corporation type HC filter (or equivalent if available) helps prevent heat damage to chlorine-injured coliforms during the critical first few hours at the very high temperature of the fecal coliform test. Because they have a larger pore size, they are less subject to clogging. Despite quantity discounts, it is good practice to order no more than a year's supply at a time.

(4) M-FC Medium. The lab prepares its own M-FC from dehydrated medium, so it has the option of deleting rosolic acid. This is normally added to suppress "background" organisms that can interfere with the test, but it can also inhibit growth of "stressed" fecal coliforms. It is recommended that the lab do a comparison of the medium with and without rosolic acid with the same samples to see whether it can be eliminated. If there is no overgrowth of nuisance organisms, use of rosolic acid should be discontinued, but it should be kept available in the event background organism numbers increase. The pH of the medium should also be regularly checked, because eliminating rosolic acid also eliminates addition of the 0.2N NaOH.

### Methods

16. Current copies of all methods employed in the lab, including all those for which the lab is requesting accreditation, are present and readily available to analysts at bench level. Procedures actually used by analysts are SOPs which were reviewed during the audit.

17. For the BOD determination, the DO determination obtained with a DO meter was checked during every batch with a Winkler titration. An alternative procedure which would require less effort from lab personnel while still providing an adequate degree of assurance that DO determinations were being accurately performed would be to check the calibration of the DO meter, cross checking with a Winkler titration on a periodic basis (once per month should suffice unless there is an indication of problems with the meter).