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# SPOKANE WASTEWATER TREATMENT PLANT CLASS II INSPECTION

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## ABSTRACT

Ecology conducted a Class II inspection at the Spokane wastewater treatment plant (WTP) on September 20-22, 1988. The 44 MGD design activated sludge facility serves the City of Spokane and portions of the greater Spokane area. This inspection coincided with a receiving water study conducted by the Ecology Surface Water Investigations Section (Carey, report in progress). The WTP was performing well during the inspection, meeting permit limits for BOD, TSS, $NH_3$-N, and total phosphorus. However, fecal coliform bacteria in a treated effluent sample was above the weekly average permit limit. The effluent was not acutely toxic as measured by Rainbow trout, *Daphnia pulex*, and Microtox bioassays.


## INTRODUCTION

Ecology conducted a Class II inspection at the Spokane WTP on September 20-22, 1988. Pat Hallinan and Marc Heffner from the Ecology Compliance Inspection Section conducted the inspection. Otis Hampton, Ecology roving operator, provided assistance during the laboratory review portion of the inspection. Dale Arnold, WTP superintendent, and Tim Pelton, WTP laboratory supervisor, also provided assistance.

The WTP treats mostly residential with some light industrial wastewater. Treated dechlorinated effluent discharges to the Spokane River as limited by NPDES Permit No. WA-002447-3. The permit contains additional limits on effluent ammonia and total phosphorus which are in effect during summer low flow periods. Dewatered digested sludge generated by the WTP is land applied.

Objectives of this inspection included:

- Verify WTP effluent compliance with NPDES permit limits.
- Analyze WTP performance by determining plant loading and efficiency.
- Determine WTP effluent acute toxicity using Trout, Microtox, and *Daphnia pulex* bioassays. These bioassays were also used to test the background toxicity of the receiving water.
- Identify possible chemical pollutants in WTP influent, effluent, and digested sludge samples.
- Determine the leachability of WTP digested sludge, using an Extraction Procedure Toxicity (EP Tox) metal analyses.
- Support the concurrent receiving water study.
- Review lab procedures at the WTP to determine conformance to standard techniques. Samples were split with the permittee to determine the accuracy of laboratory results.

## PROCEDURES

Ecology collected WTP influent and effluent 24-hour iced composite samples. ISCO automatic samplers, fitted with teflon tubing and glass sampling bottles, collected about 360 mLs of sample every 30 minutes for 24 hours. Sampling equipment was acid and solvent washed before use. The Ecology effluent sampler was placed in a channel upstream of the effluent Parshall flume (see Figure 1). The Ecology influent sampler sampled from a wet well below the center channel of three influent Parshall flume channels for about eight hours (2400 to 0830 on September 21). The influent sampler was moved to a diversion box upstream of the channels when it was discovered that flow had been diverted to a side flume channel.

The WTP also collected influent and effluent 24-hour composite samples. The influent and effluent samplers were both flow proportional. The WTP influent sampler sampled in the influent channel to the plant (Figure 1). The WTP effluent sampler was located at the chlorine contact chamber effluent weir. Composite samples were split for permit parameter, COD, and metal analyses by Ecology and WTP labs. Ecology also collected grab samples for field and lab analyses. Sampling times and parameters analyzed are listed in Table 1. In addition, Ecology made an instantaneous flow check of the effluent Parshall flume.

The Ecology Manchester Laboratory analyzed most samples collected during the inspection. Analytical Resources Incorporated (ARI) of Seattle performed volatile organic analyses while Aquatic Research of Seattle conducted nutrient ($NH_3$-N, $NO_3+NO_2$-N, and Total P) analyses. The Ecology Manchester Laboratory also conducted all bioassays. Appendix 1 lists the chemical and bioassay test methods.

## RESULTS AND DISCUSSION

### Wastewater Treatment Plant Description

Wastewater is carried to the WTP by combined (sanitary and stormwater) sewers. Due to heavy rains the day before the inspection, (0.77 inches on September 19, 1988--U.S. Weather Service), the WTP was experiencing a combined sewer overflow on the first day of the inspection. Overflow bypassing the treatment system was diverted to two stormwater clarifiers, chlorinated, held in two chlorine contact chambers, and discharged. Stormwater overflow subsided on the afternoon of September 20. Stormwater that remained in the clarifiers was pumped back to the secondary treatment works for treatment.

The WTP headworks consist of three channels (Figure 1); in each, a bar screen precedes a six foot Parshall flume. Flow from the three channels is combined in a wet well then fed to two aerated grit chambers. Any overflow is routed from the grit chambers to the two stormwater clarifiers. Flow rates to the WTP secondary treatment system are measured by a Venturi meter located after the grit chambers. The wastewater secondary treatment system includes two pre-aeration basins, four primary clarifiers, four aeration basins, and four secondary clarifiers. Effluent from the secondary clarifiers is chlorinated and sent to two chlorine contact chambers.

Table 1. Sampling times and parameters analyzed - Spokane, 9/88.

| Parameters | Station: | Influent | | | | | Effluent | | | | Stormwater | Digested Sludge |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Type: | Grabs | | | | Composite | Grabs | | | Composite | Grab | Grab |
| | Date: | 9/20 | 9/20 | 9/21 | 9/21 | 9/20-21 | 9/20 | 9/21 | 9/21 | 9/20-21 | 9/20 | 9/21 |
| | Time: | 1005 | 1649 | 1116 | 1330 | 2400-2400 | 1520 | 0923 | 1350 | 2400-2400 | 1543 | 1050 |
| GENERAL CHEMISTRY | | | | | | | | | | | | |
| Turbidity (NTU) | | | | | | X | | | | X | | |
| pH (S.U.) | | | | | | X | | | | X | | |
| Conductivity (umhos/cm) | | | | | | X | | | | X | | |
| Alkalinity (mg/L as $CaCO_3$) | | | | | | X | | | | X | | |
| Hardness (mg/L) | | | | | | | | | | X | | |
| $BOD_5$ (mg/L) | | | | | | X | | | | X | | |
| COD (mg/L) | | | | | | X | X | X | X | X | X | |
| Fecal Coliform (#/100 mL) | | | | | | | X | X | X | | X | |
| Chloride (mg/L as Cl-) | | | | | | X | X | X | X | X | X | |
| TOC (mg/L) | | | | | | | | | | | | X |
| % Solids | | | | | | | | | | | | X |
| EP Tox Metals | | | | | | | | | | | | X |
| Solids (mg/L) | | | | | | | | | | | | |
| TS | | | | | | X | | | | X | | |
| TNVS | | | | | | X | | | | X | | |
| TSS | | | | | | X | X | X | X | X | X | |
| TNVSS | | | | | | X | | | | X | | |
| Nutrients (mg/L) | | | | | | | | | | | | |
| $NH_3$-N | | | | | | X | X | X | X | X | X | |
| $NO_3$+$NO_2$-N | | | | | | X | X | X | X | X | X | |
| T-Phosphate | | | | | | X | X | X | X | X | X | |
| PRIORITY POLLUTANTS | | | | | | | | | | | | |
| Semi-volatiles | | | | | | X | | | | X | | X |
| Volatiles | | | | X | | | | X | | | | X |
| Pesticides/PCBs | | | | | | X | | | | X | | X |
| Metals | | | | | | X | | | | X | | X |
| Cyanide (mg/L) | | | | | | X | | | | X | | |
| BIOASSAYS | | | | | | | | | | | | |
| Trout | | | | | | | | | | X* | | |
| Microtox | | | | | | | | | | X* | | |
| Daphnia pulex | | | | | | | | | | X* | | |
| FIELD ANALYSES | | | | | | | | | | | | |
| Temp. (C) | | X | X | X | X | | X | X | X | | X | |
| pH (S.U.) | | X | X | X | X | | X | X | X | | X | |
| Conductivity (umhos/cm) | | X | X | X | X | | X | X | X | | X | |
| Chlorine Residual (mg/L) | | | | | | | | | | | | |
| Free | | | | | | | | | | | X | |
| Total | | | | | | | X | X | X | | X | |

* - 1/3 of samples collected 9/20 at 1530; 1/3 collected 9/21 at 0923; 1/3 collected 9/21 at 1320.

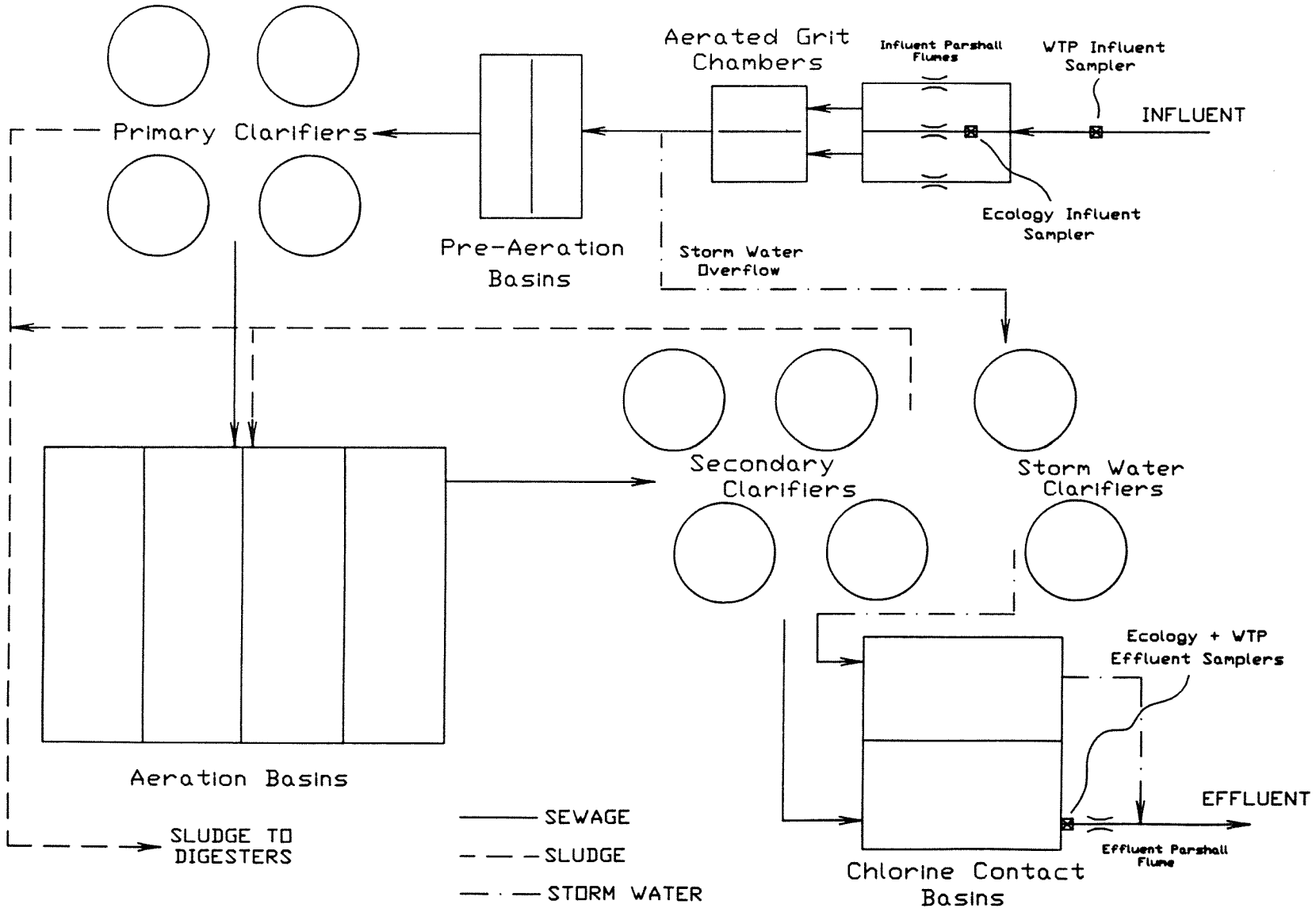


Figure 1. Plant schematic - Spokane, 9/88.

Treated wastewater is dechlorinated by sulfur dioxide and discharged down a steep embankment to the Spokane River.

Primary sludge is thickened by gravity, while secondary sludge is thickened by flotation. Both primary and secondary sludge are anaerobically digested. Digested sludge is dewatered by filter press and applied on land.

During the inspection the WTP was not utilizing the full capacity of its secondary treatment system. One of two pre-aeration basins, three of four primary clarifiers, three of four aeration basins, and two of four secondary clarifiers were in operation. When stormwater runoff was at its peak, three influent Parshall flume channels and two aerated grit chambers were in use. When flow subsided, two influent channels and one grit chamber were shut down.

**Flow Measurements**

During the inspection flow was near the design capacity of the facility (Table 2). About seven million gallons of stormwater overflow discharged on September 19 and about five million gallons on September 20 (Arnold, personal communication). Ecology made an instantaneous check of the effluent Parshall flume. The meter appeared to be accurately calibrated.

**Comparison of Effluent Parameters to NPDES Permit Limits**

Ecology analytical results for general chemistry data collected during the inspection are summarized in Table 3.

The WTP was meeting permit limits for BOD, TSS, and $NH_3$-N (Table 4). Ecology results for the Ecology and Spokane samples indicate that the WTP was below the monthly average 85 percent removal requirement for total phosphorus. However, the Ecology total phosphorus results are suspect. The duplicate phosphorus analyses gave an 11.85 percent variation which is above the acceptable limits of five percent. Using WTP results for the Ecology and Spokane samples, the 85 percent phosphorus removal was met.

Fecal coliform counts (2,400 per 100 mL) in an effluent grab sample taken on the afternoon of September 20 were above the weekly average permit limit of 400 per 100 mL. The WTP control strategy is to maintain a set residual at the end of the chlorine contact chamber. When residual levels fall below the set point, chlorine doses are manually increased at 30 minute intervals. Because of the higher than normal flows on September 20, chlorine residuals were low, indicating the manual control had not provided sufficient chlorine for adequate disinfection. The WTP should consider an automatic chlorination control system to help avoid circumstances like these in the future.

Total residual chlorine levels in the dechlorinated effluent could not be checked by Ecology (using a Chemets Ultra Low Range DPD colorimetric test kit) due to possible interferences from nitrite or chloroamines. WTP discharge monitoring reports (DMRs) indicate total residual chlorine levels in the effluent were below the 0.1 mg/L permit limit during the inspection. Total residual chlorine levels in the stormwater overflow were high (2.5 mg/L). A

Table 2. Flow measurements - Spokane, 9/88.

| Date | Time | Instantaneous Flow (MGD) Influent | Storm* | Total | Totalizer Reading | Flow for Time Increment (MGD) |
|---|---|---|---|---|---|---|
| 9/20 | 921 | 46 | 58 | 104 | 479771 | |
| | | | | | | 43.6 |
| 9/21 | 845 | 39 | 0 | 39 | 484021 | |
| | | | | | | 41.2 |
| 9/21 | 1100 | 40 | 0 | 40 | 484407 | |
| | | | | | | 45.5 |
| 9/21 | 1407 | 38 | 0 | 38 | 484998 | |
| | | | | | | 37.1 |
| 9/22 | 839 | 40 | 0 | 40 | 487864 | |
| | | Average flow during the inspection = | | | | 41.1 |

*Stormwater overflow was 7.1 MGD on 9/19 and 5.0 MGD on 9/21 (from plant records).

Table 3. Ecology results for general chemistry parameters - Spokane, 9/88.

| Parameters | Station: | Influent | | | | | Effluent | | | | Stormwater | Digested Sludge |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Type: | Grabs | | | | Composite | Grabs | | | Composite | Grab | Grab |
| | Date: | 9/20 | 9/20 | 9/21 | 9/21 | 9/20-21 | 9/20 | 9/21 | 9/21 | 9/20-21 | 9/20 | 9/21 |
| | Time: | 1005 | 1649 | 1116 | 1330 | 2400-2400 | 1520 | 0923 | 1350 | 2400-2400 | 1543 | 1050 |
| | Sample ID #: | N/A | N/A | N/A | N/A | 398160 | 398155 | 398157 | 398158 | 398159 | 398156 | 398164 |
| GENERAL CHEMISTRY | | | | | | | | | | | | |
| Turbidity (NTU) | | | | | | 41 | | | | 3 | | |
| pH (S.U.) | | | | | | 7.0 | | | | 7.3 | | |
| Conductivity (umhos/cm) | | | | | | 660 | | | | 600 | | |
| Alkalinity (mg/L as $CaCO_3$) | | | | | | 210 | | | | 130 | | |
| Hardness (mg/L) | | | | | | | | | | 156 | | |
| $BOD_5$ (mg/L) | | | | | | 124 | | | | 9 | | |
| COD (mg/L) | | | | | | 270 | 20 | 32 | 22 | 29 | 91 | |
| Fecal Coliform (#/100 mL) | | | | | | | 2400 | 13 | 57 | | | |
| Chloride (mg/L as Cl-) | | | | | | 65 | 54 | 52 | 67 | 63 | 31 | |
| Cyanide (mg/L) | | | | | | 0.01 | | | | 0.008 | | |
| TOC (mg/L) | | | | | | | | | | 12 | | 21 |
| % Solids | | | | | | | | | | | | 3.8 |
| Solids (mg/L) | | | | | | | | | | | | |
| TS | | | | | | 520 | | | | 390 | | |
| TNVS | | | | | | 370 | | | | 320 | | |
| TSS | | | | | | 110 | 4 | 6 | 8 | 8 | 25 | |
| TNVSS | | | | | | 22 | | | | 2 | | |
| Nutrients (mg/L) | | | | | | | | | | | | |
| $NH_3$-N | | | | | | 9.15 | 0.20 | 1.63 | 1.84 | 2.78 | 3.96 | |
| $NO_3+NO_2$-N | | | | | | 0.64 | 4.62 | 1.73 | 3.80 | 2.29 | 0.51 | |
| T-Phosphate | | | | | | 3.69 | 0.22 | 0.49 | 0.45 | 0.66 | 2.19 | |
| FIELD ANALYSES | | | | | | | | | | | | |
| Temp. (C) | | 13.9 | 18.0 | 18.6 | 18.8 | | 16.9 | 16.7 | 18.1 | | 15.3 | |
| pH (S.U.) | | 8.1 | 7.8 | 7.7 | 7.3 | | 7.1 | 7.4 | 7.2 | | 7.0 | |
| Conductivity (umhos/cm) | | 250 | 760 | 720 | 970 | | 440 | 490 | 660 | | 330 | |
| Chlorine Residual (mg/L) | | | | | | | | | | | | |
| Free | | | | | | | | | | | <0.1 | |
| Total | | | | | | | 0.065* | 0.085* | 0.075* | | 2.5 | |

* - Readings from plant meter.

Table 4. Comparison of NPDES permit limits to analytical results from Ecology's laboratory - Spokane, 9/88.

| Parameter | NPDES Permit Limits | | | Analytical Results | | |
|---|---|---|---|---|---|---|
| | Monthly Average | Weekly Average | Daily Maximum | Ecology Composite Samples | Spokane Composite Samples | Ecology Grab Samples |
| Influent $BOD_5$ (mg/L) | | | | 124 | 108 | |
| $BOD_5$ | | | | | | |
| (mg/L) | 30 | 45 | | 9 | 12 | |
| (lbs/D) | 11,009 | 16,513 | | 3,019 | 4,025 | |
| (% removal) | 85 | 85 | | 93 | 89 | |
| Influent TSS (mg/L) | | | | 110 | 130 | |
| TSS | | | | | | |
| (mg/L) | 30 | 45 | | 8 | 6 | |
| (lbs/D) | 11,009 | 16,513 | | 2,684 | 2,013 | |
| (% removal) | 85 | 85 | | 93 | 95 | |
| Influent $NH_3$-N (mg/L) | | | | 9.2 | 9.5 | |
| $NH_3$-N | | | | | | |
| (mg/L) | | | | 2.8 | 2.9 | |
| (lbs/D) | | 2,700 | 5,000 | 939 | 973 | |
| (% removal) | | | | 70 | 69 | |
| Influent Total P* (mg/L) | | | | 3.70 | 4.60* | |
| Total P | | | | | | |
| (mg/L) | | | | 0.66 | 0.51* | |
| (lbs/D) | 205 | | 275 | 221 | 171 | |
| (% removal) | 85 | | | 82 | 89 | |
| Fecal coliform (#/100 mL) | 200 | 400 | | | | 2,400; 57; 13 |
| pH (S.U.) | 6.0 - 9.0 | | | | | 7.1, 7.4, 7.2 |
| Flow (MGD) | | | | 40.22** | 40.22** | |

*Total P are results obtained by the WTP laboratory; all other results from the Ecology lab.
**Flow from plant records.

total residual chlorine level of 0.5 mg/L usually provides good disinfection. Reducing chlorine levels in stormwater over flow, while maintaining low fecal counts, is recommended.

**Effluent and Influent Priority Pollutant Analyses**

Complete effluent and influent priority pollutant results (volatiles, semivolatiles, pesticides/polychlorinated biphenyls [PCBs], and metals) are given in Appendix 1. The WTP influent sample contained low levels of several volatile organics, including acetone at 130 parts per billion (ppb or ug/L), chloroform at 2.3 ppb, and toluene at 2.4 ppb. Several semivolatiles (phenols and phthalates) were also detected in the low ppb range (Table 5).

A PCB (Aroclor-1260) was detected at 37 ppb in the influent. The influent sample extract was reanalyzed and the presence of the PCB was confirmed (re-analysis result of 18 ppb). No PCBs were detected in the effluent or digested sludge samples suggesting that the influent concentration was atypical. No PCBs were detected in influent or effluent samples collected during an Ecology sampling at the WTP in December 1981 (Bernhardt, 1985). Most recent priority pollutant scans of the sludge by the WTP have only detected trace amounts of PCBs (Arnold, personal communication). The source of the PCB was most likely from surface runoff from a contaminated site.

No volatile or semivolatile organics were detected in the effluent sample. In the pesticide analysis, DDT was found at eight parts per trillion (ppt). Though banned from use in the United States in 1974, DDT is still widely used in other countries (e.g., India, Mexico). Trace quantities (parts per trillion concentrations) of this pesticide can be transported through the atmosphere and deposited by rainwater. Past use may also be the source.

Table 6 lists metals detected in the influent and effluent samples. These values are also compared to Washington State water quality criteria for protection of aquatic life (EPA, 1986). Cadmium in the effluent exceeded freshwater chronic criteria and approached freshwater acute criteria. Effluent silver also neared the freshwater acute level. All other effluent metals were below acute and chronic limits.

**Digested Sludge Priority Pollutant Analyses**

The WTP digested sludge sample contained low levels of organic chemical contamination (Table 5). Appendix 1 lists the complete results of digested sludge priority pollutant analyses. Volatile organics found included carbon disulfide at 6.6 parts per million (ppm or mg/kg wet weight), toluene at 34 ppm, ethylbenzene at 8 ppm, and total xylenes at 40 ppm. Toluene, ethylbenzene, and xylenes can be used as industrial solvents and are also constituents of gasoline. A phthalate (bis-2-ethylhexyl) was found at 200 ppm. None of these compounds are listed in EPA's draft sewage sludge regulations (EPA, 1988).

Metals detected in the sludge fell within ranges found at other activated sludge plants during previous Class II inspections in Washington State (Table 7). Extraction Procedure Toxicity (EP Tox) metal analysis found concentrations well below dangerous waste designations (Ecology, 1988).

Table 5. Organics detected in influent, effluent and sludge samples - Spokane, 9/88.

| | Influent (ug/L) | | Effluent (ug/L) | | Sludge (mg/kg wet) | |
|---|---|---|---|---|---|---|
| **Volatile Organics:** | | | | | | |
| Methylene Chloride | **3.5** | **B** | **3.2** | **B** | **12** | **B** |
| Acetone | **130** | | 0.6 | U | 17 | U |
| Carbon Disulfide | 2.0 | U | 2.0 | U | **6.6** | |
| Chloroform | **2.3** | | 0.9 | U | 2.7 | U |
| Trichloroethene | 0.8 | U | 0.8 | U | **1.4** | **J** |
| Benzene | 0.4 | U | 0.4 | U | **1.8** | **J** |
| Tetrachloroethene | **0.7** | **M** | 0.6 | U | 1.2 | U |
| Toluene | **2.4** | | 0.6 | U | **34** | |
| Ethylbenzene | 1.0 | U | 1.0 | U | **7.9** | |
| Total Xylenes | 1.5 | U | 1.5 | U | **40** | |
| **Phenols:** | | | | | | |
| 2-Methylphenol | **2** | **J** | 2 | U | 180 | U |
| 4-Methylphenol | **2** | **J** | 2 | U | 180 | U |
| **Phthalates:** | | | | | | |
| Diethylphthalate | **3** | **J** | 2 | U | 180 | U |
| Di-n-Butylphthalate | **6** | **J** | 2 | U | 180 | U |
| bis(2-Ethylhexyl)Phthalate | **34** | | **2** | **J** | **200** | |
| **Pesticides/PCBs:** | | | | | | |
| 4,4'-DDT | 0.14 | U | **0.008** | | 130 | U |
| Aroclor-1260 | **37** | | 0.020 | U | 660 | U |

Qualifiers:

U - Not detected at detection limit shown.
J - Estimated amount, concentration is below detection limit.
B - Also detected in method blank.
M - Estimated value, compound found and confirmed but with low spectral match parameters.

Table 6. Metals detected in influent and effluent - Spokane, 9/88.

| | | | Washington State Water Quality Criteria | |
|---|---|---|---|---|
| | Influent (ug/L) | Effluent* (ug/L) | Acute (ug/L) | Chronic (ug/L) |
| Arsenic | 1.7 | 0.6 | 360 | 190 |
| Lead | 93.5 | 3.5 | 144 | 5.6 |
| Silver | 10.2 | 0.6 | 0.77 | -- |
| Mercury | 0.22 | 0.08 U | 2.4 | 0.012 |
| Cadmium | 17 | 5 | 6.5 | 1.6 |
| Copper | 82 | 13 | 27.0 | 17.3 |
| Zinc | 168 | 42 | 171 | 154 |

* - Effluent hardness = 156 mg/L as $CaCO_3$

Qualifier:

U - Not detected at detection limit shown

Table 7. Sludge metals data - Spokane, 9/88.

| | | Data from Previous Inspections* | | | EP Tox Metals | |
|---|---|---|---|---|---|---|
| Metal | WTP** Sample (mg/Kg dry wt) | Range (mg/Kg dry wt) | Geometric Mean (mg/Kg dry wt) | Number of Samples | WTP Sample (ug/L) | Dangerous Waste Minimum Concentration (ug/L) |
| Cadmium | 18.2 | <0.1-25 | 7.6 | 34 | 5 U | 1,000 |
| Chromium | 93.7 | 15-300 | 61.8 | 34 | 10 U | 5,000 |
| Copper | 424 | 75-1700 | 398 | 34 | -- | -- |
| Lead | 225 | 34-600 | 207 | 34 | 50 U | 5,000 |
| Nickel | 18.2 | <0.1-62 | 25.5 | 29 | -- | -- |
| Zinc | 1011 | 165-3370 | 1200 | 33 | -- | -- |
| Barium | -- | -- | -- | -- | 215 | 100,000 |
| Mercury | 3.9 | -- | -- | -- | 0.13 | 200 |
| Arsenic | 13.2 | -- | -- | -- | 50 U | 5,000 |
| Thallium | 0.05 | -- | -- | -- | -- | -- |
| Selenium | 0.3 U | -- | -- | -- | 127 | 1,000 |
| Silver | 0.82 | -- | -- | -- | 5 | 5,000 |
| Antimony | 1.7 | -- | -- | -- | -- | -- |
| Beryllium | 0.26 | -- | -- | -- | -- | -- |

*Data collected during previous Class II inspections at activated sludge plants throughout Washington (Hallinan, 1988).

**Percent solids = 3.8

Qualifier:

U - Not detected at detection limit shown.

### Effluent and Receiving Water Bioassays

Three bioassay tests were performed (Table 8): Rainbow trout (*Oncorhynchus mykiss,* formerly *Salmo gairdneri*), *Daphnia pulex* (a water flea), and Microtox (a luminescent bacteria). The WTP effluent caused no mortalities in either the trout or *Daphnia* bioassays. In the Microtox test, the effluent resulted in no reduction in bacterial luminescence (a measure of sample toxicity).

No mortalities or reduction in bacterial luminescence were observed for the Spokane River sample collected above the WTP discharge. The river historically has exhibited high zinc concentrations due to past mining activities in Northern Idaho. Zinc concentrations vary and often exceed acute water quality criteria. Highest zinc levels are usually observed at higher river flows (Funk, *et al.*, 1983). Zinc concentrations ranged from 24 to 27 ug/L in three river samples collected at Riverside State Park (about 4.5 miles downstream of the WTP discharge) during the concurrent receiving water study (Chern, 1989). This is well below the acute (one hour average) criteria of 100 ug/L (EPA, 1986). Hardness of the river bioassay sample was 85 mg/L as $CaCO_3$.

### Comparison of Laboratory Results

Lab results between the WTP and Ecology laboratories compared favorably (Table 9). However, the fecal coliform results for the effluent grab sample taken on September 20 were significantly different. The Ecology laboratory obtained 2400 colonies per 100 mL while the permittee's result was 640. In addition, influent ammonia measured by the permittee was about 15 percent higher than results obtained by Ecology. Effluent ammonia concentrations agreed closely. A review of both ammonia and fecal coliform procedures and the use of ammonia check standards for influent and effluent samples by the WTP is suggested.

Spokane WTP lab results for TSS, COD, and total volatile suspended solids (TVSS) for the WTP influent composite sample were high compared with other influent sample results from Ecology's lab. Influent total phosphorus samples analyzed by the Ecology lab were low compared with influent samples analyzed by the Spokane lab.

Results of the Ecology influent and effluent composite samples for the two labs compared very well (Table 10). However, the influent sample collected and analyzed by the permittee showed consistently lower concentrations for copper, lead, and zinc than the influent sample collected by Ecology. Ecology did not analyze the Spokane WTP composite for metals.

## LABORATORY REVIEW

A complete laboratory review report is included in Appendix 2 of this report. Circled items indicate where work is needed. Most laboratory procedures were in conformance with standard techniques. An exception was in the fecal coliform procedure. Dilution and rinse water for the test should be made with distilled water buffered with phosphate (APHA, 1985,

Table 8. Effluent and receiving water bioassay results - Spokane, 9/88.

96 hour Rainbow Trout (**Oncorhynchus mykiss**, formerly **Salmo gairdneri**):

| Sample | # of Live Test Organisms Initial | Final | % Mortality |
|---|---|---|---|
| WTP Effluent | 30 | 30 | 0 |
| Spokane River | 30 | 30 | 0 |
| Lab Control | 30 | 30 | 0 |

48-hour **Daphnia pulex**:

| Sample | # of Live Test Organisms Initial | Final | % mortality |
|---|---|---|---|
| WTP Effluent | 25 | 25 | 0 |
| Spokane River | 25 | 25 | 0 |
| Lab Control | 25 | 25 | 0 |

**Microtox**:

No measurable toxic response (reduction in bacterial luminescence) was detected with either the WTP effluent or Spokane River samples.

Table 9. Comparison of laboratory results for general chemistry parameters - Spokane, 9/88.

| Station: | Influent | | | | Effluent | | | | | | | | Storm Water | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Type: | Composite | | | | Composite | | | | Grab | | Grab | | Grab | |
| Date: | 9/20-21 | | | | 9/20-21 | | | | 9/20 | | 9/21 | | 9/20 | |
| Time: | 2400-2400 | | | | 2400-2400 | | | | 1520 | | 1350 | | 1543 | |
| Sampler: | Ecology | | Spokane | | Ecology | | Spokane | | Ecology | Spokane | Ecology | Spokane | Ecology | Spokane |
| Laboratory: | Ecology | Spokane | Ecology | Spokane | Ecology | Spokane | Ecology | Spokane | Ecology | Spokane | Ecology | Spokane | Ecology | Spokane |
| GENERAL CHEMISTRY | | | | | | | | | | | | | | |
| $BOD_5$ (mg/L) | 124 | 111 | 108 | 127 | 9 | 9 | 12 | 10 | | | | | | |
| $COD^5$ (mg/L) | 270 | 279 | 250 | 447 | 29 | 25 | 34 | 17 | | | | | | |
| TSS (mg/L) | 110 | 131 | 130 | 171 | 8 | 7 | 6 | 8 | | | | | | |
| TVSS (mg/L) | 88 | 101 | 116 | 153 | 6 | 7 | 5 | 6 | | | | | | |
| $NH_3$-N (mg/L) | 9.2 | 10.7 | 9.5 | 10.8 | 2.8 | 3.1 | 2.8 | 2.9 | | | | | | |
| $NO_3$+$NO_2$-N (mg/L) | 0.64 | | 0.56 | | 2.3 | | 2.3 | | | | | | | |
| $NO_3$-N (mg/L) | | 0.52 | | 0.45 | | 2.4 | | 2.6 | | | | | | |
| T-Phosphate (mg/L) | 3.7 | 4.3 | 1.9 | 4.6 | 0.66 | 0.51 | 0.83 | 0.51 | | | | | | |
| O-Phosphate (mg/L) | | 2.9 | | 2.8 | | 0.44 | | 0.39 | | | | | | |
| Fecal Coliform (#/100 mL) | | | | | | | | | 2400 | 620 | 57 | 34 | 1 | 0 |

Table 10. Comparison of laboratory results for metal parameters - Spokane, 9/88.

| Station: | Influent | | | Effluent | | |
|---|---|---|---|---|---|---|
| Type: | Composite | | | Composite | | |
| Date: | 9/20-21 | | | 9/20-21 | | |
| Time: | 2400-2400 | | | 2400-2400 | | |
| Sampler: | Ecology | | Spokane | Ecology | | Spokane |
| Metal (ug/L) Laboratory: | Ecology | Spokane | Spokane | Ecology | Spokane | Spokane |
| Cadmium | 17.0 | 14.2 | 10.3 | 5.0 | 1.0 | 1.0 |
| Chromium | <10.0 | 13.0 | 7.0 | <10.0 | 1.0 | 1.0 |
| Copper | 82 | 60 | 47 | 13 | 4 | 6 |
| Lead | 93.5 | 104 | 19 | 3.5 | 2 | 4 |
| Nickel | <20.0 | 14 | 6 | <20.0 | 2 | 3 |
| Zinc | 168 | 178 | 123 | 42 | 39 | 46 |

p885 #1a). This may have been the cause of the poor fecal coliform agreement between the labs.

## CONCLUSIONS AND RECOMMENDATIONS

- The WTP was performing well during the inspection meeting permit limits for BOD, TSS, $NH_3$-N, and total phosphorus. However, fecal coliform in one effluent sample was above the weekly average permit limit. Stormwater overflow was chlorinated at excessive levels. Reducing stormwater chlorine residuals while maintaining low fecal counts is recommended. In addition, the WTP should consider an automatic clorination system to help provide better disinfection of treated effluent.

- Treated effluent did not exhibit acute toxicity as measured by Rainbow trout, *Daphnia pulex,* and Microtox bioassays. No toxicity was exhibited by a Spokane River sample collected above the WTP discharge.

- Treated effluent and digested sludge samples analyzed for priority pollutants indicated low levels of contamination. A PCB (Aroclor-1260) was detected in the influent sample at a relatively high level. No PCBs were detected in the digested sludge sample suggesting that the influent concentration was atypical. Effluent metals, expect cadmium, were within Washington State water quality criteria.

- Lab procedures were good. Minor recommendations are made in the Laboratory Review section of this report.

# REFERENCES

# REFERENCES

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

Arnold, D. Spokane Wastewater Treatment Plant. Personal Communication.

Bernhardt, 1985. Impacts of the Spokane Wastewater Treatment Plant on the Spokane River Including Recommended NPDES Limits for Chlorine and Ammonia. Memo to Roger Ray, August 5, 1985. Department of Ecology, EILS.

Chern, L., 1989. Reconnaissance Survey of the Impacts of Northside Landfill Leachate on Ground/Surface Water Quality, Spokane, Washington. Department of Ecology, EILS, May 1989.

Ecology, 1988. Dangerous Waste Regulations, Chapter 173-303 WAC, Amended September 1988.

EPA, 1988. Guidance for Writing Case-by-Case Permit Requirements for Municipal Sewage Sludge. Permits Division, Draft, September 1988.

EPA, 1986. Quality Criteria for Water, EPA 440/5-86-001, 1986.

Funk, *et al.*, 1983. Funk, W.F., Gibbons, H.L., Duffner, R.M., Notestine, T., and Nielsen, T. Water Quality of the Upper Spokane River and Evaluation of Methods for Measurement of the Effect of Effluent upon Primary and Secondary Producers. State of Washington Water Research Center, Pullman, Washington, January, 1983.

Hallinan, P., 1988. Metals Concentrations Found During Ecology Inspections of Municipal WTPs. Memo to J. Bernhardt, April 11, 1988. Department of Ecology, EILS.

U.S. Weather Service, 1988. Climatologic Data for Washington, September 1988.

# APPENDIX 1

Chemical Analytical Methods - Spokane, 9/88.

| Analyses | Method Used | Laboratory |
|---|---|---|
| TOC (sludge) | APHA, 1985: #505 | Laucks Testing Labs; Seattle, WA |
| % Solids | APHA, 1985: #209F | Laucks Testing Labs; Seattle, WA |
| Cyanide (water) | EPA, 1983: #335.2-1 | Ecology; Manchester, WA |
| Volatiles (water) | EPA, 1984: #624 | Analytical Resources Inc., Seattle, WA |
| Volatiles (sludge) | EPA, 1986: #8240 | Analytical Resources Inc., Seattle, WA |
| Semivolatiles (water) | EPA, 1984: #625 | Ecology; Manchester, WA |
| Semivolatiles (sludge) | EPA, 1986: #8270 | Ecology; Manchester, WA |
| Pest/PCB (water) | EPA, 1984: #608 | Ecology; Manchester, WA |
| Pest/PCB (sludge) | EPA, 1986: #8080 | Ecology; Manchester, WA |
| Metals (water) | EPA, 1983: #200 series | Ecology; Manchester, WA |
| Metals (sludge) | EPA, 1983: #200 series | Ecology; Manchester, WA |
| Ammonia | EPA, 1983: #350.1 | Aquatic Research, Seattle, WA |
| Total Phosphorus | EPA, 1983: #353.2 | Aquatic Research, Seattle, WA |
| Nitrate/Nitrite | EPA, 1983: #365.1 | Aquatic Research, Seattle, WA |

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

EPA, 1983. Methods for Chemical Analysis of Water and Wastes, 600/4/79-020, revised March 1983.

EPA, 1984. 40 CFR Part 136, October 26, 1984.

EPA, 1986. Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846, 3rd ed., November 1986.

Bioassay Methods - Spokane, 9/88.

| Test Organism | Reference Method | Test Laboratory | Test Duration | Test Concentration | Type of Test | Endpoint Measured |
|---|---|---|---|---|---|---|
| Daphnia pulex | 1 | Ecology | 48 hrs | 100% | Acute | Survival |
| Rainbow Trout (Oncorhynchus mykiss) | 2 | Ecology | 96 hrs | 100% | Acute | Survival |
| Microtox | 3 | Ecology | 15 min | 90.9, 45.5, 22.7 11.4% | Acute/ Chronic | Reduction in bacterial luminescence |

1 - EPA/600/4-85/013, "Methods for Measuring the Acute Toxicity of Effluent Water and Marine Organisms."
2 - Department of Ecology procedure "Static Acute Fish Toxicity Test."
3 - Beckman Microtox System Operating Manual.

Results of BNA Priority Pollutant Scan - Spokane, 9/88.

| Compound | Transfer Blank (ug/L) | Influent (ug/L) | Effluent (ug/L) | Sludge (mg/kg wet) |
|---|---|---|---|---|
| Phenol | **2 BU** | **9 BU** | 2 U | 180 U |
| bis(2-Chloroethyl)Ether | 2 U | 9 U | 2 U | 180 U |
| 2-Chlorophenol | 2 U | 9 U | 2 U | 180 U |
| 1,3-Dichlorobenzene | 2 U | 9 U | 2 U | 180 U |
| 1,4-Dichlorobenzene | 2 U | 9 U | 2 U | 180 U |
| Benzyl Alcohol | 2 U | 9 U | 2 U | 180 U |
| 1,2-Dichlorobenzene | 2 U | 9 U | 2 U | 180 U |
| 2-Methylphenol | 2 U | **2 J** | 2 U | 180 U |
| bis(2-chloroisopropyl)ether | 2 U | 9 U | 2 U | 180 U |
| 4-Methylphenol | 2 U | **2 J** | 2 U | 180 U |
| N-Nitroso-Di-n-Propylamine | 2 U | 9 U | 2 U | 180 U |
| Hexachloroethane | 2 U | 9 U | 2 U | 180 U |
| Nitrobenzene | 2 U | 9 U | 2 U | 180 U |
| Isophorone | 2 U | 9 U | 2 U | 180 U |
| 2-Nitrophenol | 2 U | 9 U | 2 U | 180 U |
| 2,4-Dimethylphenol | 2 U | 9 U | 2 U | 180 U |
| Benzoic Acid | 9 U | 45 U | 12 U | 880 U |
| bis(2-Chloroethoxy)Methane | 2 U | 9 U | 2 U | 180 U |
| 2,4-Dichlorophenol | 2 U | 9 U | 2 U | 180 U |
| 1,2,4-Trichlorobenzene | 2 U | 9 U | 2 U | 180 U |
| Naphthalene | 2 U | 9 U | 2 U | 180 U |
| 4-Chloroaniline | 2 U | 9 U | 2 U | 180 U |
| Hexachlorobutadiene | 2 U | 9 U | 2 U | 180 U |
| 4-Chloro-3-Methylphenol | 2 U | 9 U | 2 U | 180 U |
| 2-Methylnaphthalene | 2 U | 9 U | 2 U | 180 U |
| Naphthalene, 1-methyl- | 2 U | 9 U | 2 U | 180 U |
| Hexachlorocyclopentadiene | 2 U | 9 U | 2 U | 180 U |
| 2,4,6-Trichlorophenol | 2 U | 9 U | 2 U | 180 U |
| 2,4,5-Trichlorophenol | 9 U | 45 U | 12 U | 880 U |
| 2-Chloronaphthalene | 2 U | 9 U | 2 U | 180 U |
| 2-Nitroaniline | 9 U | 45 U | 12 U | 880 U |
| Dimethylphthalate | 2 U | 9 U | 2 U | 180 U |
| Acenaphthylene | 2 U | 9 U | 2 U | 180 U |
| 3-Nitroaniline | 9 U | 45 U | 12 U | 880 U |
| Acenaphthene | 2 U | 9 U | 2 U | 180 U |
| 2,4-Dinitrophenol | 9 U | 45 U | 12 U | 880 U |
| 4-Nitrophenol | 9 U | 45 U | 12 U | 880 U |
| Dibenzofuran | 2 U | 9 U | 2 U | 180 U |
| 2,4-Dinitrotoluene | 2 U | 9 U | 2 U | 180 U |
| 2,6-Dinitrotoluene | 2 U | 9 U | 2 U | 180 U |
| Diethylphthalate | 2 U | **3 J** | 2 U | 180 U |
| 4-Chlorophenyl-phenylether | 2 U | 9 U | 2 U | 180 U |
| Fluorene | 2 U | 9 U | 2 U | 180 U |
| 4-Nitroaniline | 9 U | 45 U | 12 U | 880 U |
| 4,6-Dinitro-2-Methylphenol | 9 U | 45 U | 12 U | 880 U |
| N-Nitrosodiphenylamine | **2 BU** | 9 U | **2 BU** | 180 U |
| 4-Bromophenyl-phenylether | 2 U | 9 U | 2 U | 180 U |
| Hexachlorobenzene | 2 U | 9 U | 2 U | 180 U |
| Pentachlorophenol | 9 U | 45 U | 12 U | 880 U |
| Phenanthrene | 2 U | 9 U | 2 U | 180 U |
| Anthracene | 2 U | 9 U | 2 U | 180 U |
| Carbazole | 2 U | 9 U | 2 U | 180 U |
| Di-n-Butylphthalate | 2 U | **6 J** | 2 U | 180 U |
| Fluoranthene | 2 U | 9 U | 2 U | 180 U |
| Pyrene | 2 U | 9 U | 2 U | 180 U |
| Retene | 2 U | 9 U | 2 U | 180 U |
| Butylbenxylphthalate | 2 U | 9 U | 2 U | 180 U |
| 3,3'-Dichlorobenzidine | 4 U | 18 U | 5 U | 360 U |
| Benzo(a)Anthracene | 2 U | 9 U | 2 U | 180 U |
| bis(2-Ethylhexyl)Phthalate | 1 J | **34** | **2 J** | **200** |
| Chrysene | 2 U | 9 U | 2 U | 180 U |
| Di-n-Octyl Phthalate | 2 U | 9 U | 2 U | 180 U |
| Benzo(b)Fluoranthene | 2 U | 9 U | 2 U | 180 U |
| Benzo(k)Fluoranthene | 2 U | 9 U | 2 U | 180 U |
| Benzo(a)Pyrene | 2 U | 9 U | 2 U | 180 U |
| Indeno(1,2,3-cd)Pyrene | 2 U | 9 U | 2 U | 180 U |
| Dibenzo(a,h)Anthracene | 2 U | 9 U | 2 U | 180 U |
| Benzo(ghi)Perylene | 2 U | 9 U | 2 U | 180 U |

Qualifiers:
U - Not detected at detection limit shown.
J - Estimated amount, concentration is below detection limit.
B - Also detected in method blank.

Results of VOA Priority Pollutant Scan - Spokane, 9/88.

| Compound | Blank (ug/L) | Influent (ug/L) | Effluent (ug/L) | Sludge (ug/Kg) |
|---|---|---|---|---|
| Chloromethane | 2.9 U | 2.9 U | 2.9 U | 9.3 U |
| Bromomethane | 0.9 U | 0.9 U | 0.9 U | 7.6 U |
| Vinyl Chloride | 1.1 U | 1.1 U | 1.1 U | 4.9 U |
| Chloroethane | 0.9 U | 0.9 U | 0.9 U | 8.1 U |
| Methylene Chloride | **0.9 JB** | **3.5 B** | **3.2 B** | **12 B** |
| Acetone | 0.6 U | **130** | 0.6 U | 17 U |
| Carbon Disulfide | 2.0 U | 2.0 U | 2.0 U | **6.6** |
| 1,1-Dichloroethene | 1.3 U | 1.3 U | 1.3 U | 1.7 U |
| 1,1-Dichloroethane | 1.1 U | 1.1 U | 1.1 U | 1.5 U |
| Trans-1,2-Dichloroethene | 1.1 U | 1.1 U | 1.1 U | 2.0 U |
| Cis-1,2-Dichloroethene | 1.2 U | 1.2 U | 1.2 U | NA |
| Chloroform | 0.9 U | **2.3** | 0.9 U | 2.7 U |
| 1,2-Dichloroethane | 0.6 U | 0.6 U | 0.6 U | 1.2 U |
| 2-Butanone | 1.0 U | 1.0 U | 1.0 U | 15 U |
| 1,1,1-Trichloroethane | 1.0 U | 1.0 U | 1.0 U | 1.5 U |
| Carbon Tetrachloride | 0.5 U | 0.5 U | 0.5 U | 2.2 U |
| Vinyl Acetate | 1.7 U | 1.7 U | 1.7 U | 7.6 U |
| Bromodichloromethane | 0.2 U | 0.2 U | 0.2 U | 0.7 U |
| 1,2-Dichloropropane | 0.6 U | 0.6 U | 0.6 U | 1.7 U |
| Cis-1,3-Dichloropropene | 0.5 U | 0.5 U | 0.5 U | 4.7 U |
| Trichloroethene | 0.8 U | 0.8 U | 0.8 U | **1.4 J** |
| Dibromochloromethane | 0.9 U | 0.9 U | 0.9 U | 1.7 U |
| 1,1,2-Trichloroethane | 0.3 U | 0.3 U | 0.3 U | 1.7 U |
| Benzene | 0.4 U | 0.4 U | 0.4 U | **1.8 J** |
| cis-1,3-Dichloropropene | 0.6 U | 0.6 U | 0.6 U | 4.4 U |
| 2-Chloroethylvinylether | 1.5 U | 1.5 U | 1.5 U | 6.6 U |
| Bromoform | 0.3 U | 0.3 U | 0.3 U | 6.1 U |
| 4-Methyl-2-Pentanone | 1.8 U | 1.8 U | 1.8 U | 8.6 U |
| 2-Hexanone | 1.3 U | 1.3 U | 1.3 U | 7.8 U |
| Tetrachloroethene | 0.6 U | **0.7 M** | 0.6 U | 1.2 U |
| 1,1,2,2-Tetrachloroethane | 0.6 U | 0.6 U | 0.6 U | 6.6 U |
| Toluene | 0.6 U | **2.4** | 0.6 U | **34** |
| Chlorobenzene | 0.6 U | 0.6 U | 0.6 U | 2.2 U |
| Ethylbenzene | 1.0 U | 1.0 U | 1.0 U | **7.9** |
| Styrene | 0.5 U | 0.5 U | 0.5 U | 2.7 U |
| Total Xylenes | 1.5 U | 1.5 U | 1.5 U | **40** |

Qualifiers:

U - Not detected at detection limit shown.
J - Estimated amount, concentration is below detection limit.
B - Also detected in method blank.

Results of Pesticides/PCB and Metal Priority Pollutant Scans - Spokane, 9/88.

| Compound | Transfer Blank (ug/L) | Influent (ug/L) | Effluent (ug/L) | Sludge (ug/kg wet) |
|---|---|---|---|---|
| 4,4'-DDT | 0.005 U | 0.14 U | 0.008 | 130 U |
| Chlordane | 0.005 U | 0.14 U | 0.005 U | 130 U |
| Gamma-BHC (Lindane) | 0.005 U | 0.14 U | 0.005 U | 130 U |
| Dieldrin | 0.005 U | 0.14 U | 0.005 U | 130 U |
| Endrin | 0.005 U | 0.14 U | 0.005 U | 130 U |
| 4,4'-DDD | 0.005 U | 0.14 U | 0.005 U | 130 U |
| 4,4'-DDE | 0.005 U | 0.14 U | 0.005 U | 130 U |
| Heptachlor | 0.005 U | 0.14 U | 0.005 U | 130 U |
| Aldrin | 0.005 U | 0.14 U | 0.005 U | 130 U |
| Alpha-BHC | 0.005 U | 0.14 U | 0.005 U | 130 U |
| Beta-BHC | 0.005 U | 0.14 U | 0.005 U | 130 U |
| Delta-BHC | 0.005 U | 0.14 U | 0.005 U | 130 U |
| Alpha-Endosulfan | 0.005 U | 0.14 U | 0.005 U | 130 U |
| Heptachlor Epoxide | 0.005 U | 0.14 U | 0.005 U | 130 U |
| Endosulfan Sulfate | 0.005 U | 0.14 U | 0.005 U | 130 U |
| Endrin Ketone | 0.005 U | 0.14 U | 0.005 U | 130 U |
| Toxaphene | 0.045 U | 2.2 U | 0.060 U | 1980 U |
| PCB-1260 | 0.015 U | 37 | 0.020 U | 660 U |
| PCB-1254 | 0.015 U | 0.72 U | 0.020 U | 660 U |
| PCB-1221 | 0.015 U | 0.72 U | 0.020 U | 660 U |
| PCB-1232 | 0.015 U | 0.72 U | 0.020 U | 660 U |
| PCB-1248 | 0.015 U | 0.72 U | 0.020 U | 660 U |
| PCB-1016 | 0.015 U | 0.72 U | 0.020 U | 660 U |
| Beta-Endosulfan | 0.005 U | 0.14 U | 0.005 U | 130 U |
| PCB-1242 | 0.015 U | 0.72 U | 0.020 U | 660 U |
| **Metal** | | | | **(mg/kg dry)** |
| Arsenic | 0.5 U | 1.7 | 0.6 | 13.2 |
| Lead | 1 U | 93.5 | 3.5 | 225 |
| Thallium | 1 U | 1 U | 1 U | 0.05 |
| Silver | 0.2 U | 10.2 | 0.6 | 0.82 |
| Antimony | 2 U | 2 U | 2 U | 1.65 |
| Selenium | 1 U | 1 U | 1 U | 0.3 U |
| Mercury | 0.08 U | 0.22 | 0.08 U | 3.92 |
| Beryllium | 1 U | 1 U | 1 U | 0.26 |
| Cadmium | 5 U | 17 | 5 | 18.2 |
| Chromium | 10 U | 10 U | 10 U | 93.7 |
| Copper | 4 U | 82 | 13 | 424 |
| Nickel | 20 U | 20 U | 20 U | 17.9 |
| Zinc | 12 | 168 | 42 | 1010.5 |

Qualifiers:

U - Not detected at detection limit shown.
J - Estimated amount, concentration is below detection limit.
B - Also detected in method blank.

# APPENDIX 2

## Laboratory Procedure Review Sheet

Discharger: Spokane WTP

Date: 9/20

Discharger representative: Tim Pelton

Ecology reviewer: Pat Hallinan, Marc Heffner, Otis Hampton

### 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? American Sigma

   The compositor should have pre and post-purge cycles unless it is a flow through type. Check if you are unfamiliar with the type being used.

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

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

5. What time does sample collection usually begin? midnight

6. How long does sample collection last? 24 hrs

7. How often are subsamples that make up the composite collected? varies with flow

8. What volume is each subsample? varies with flow - 520ml max

9. What is the final volume of sample collected? ~ 3.5 l

10. Is the composite cooled during collection? yes

11. To what temperature? ✓

    The sample should be maintained at approximately 4 degrees C (SM, p41, #5b: SSM, p2).

12. How is the sample cooled? ✓

    Mechanical refrigeration or ice are acceptable. Blue ice or similar products are often inadequate.

13. How often is the temperature measured? ✓

    The temperature should be checked at least monthly to assure adequate cooling.

14. Are the sampling locations representative? ✓

15. Are any return lines located upstream of the influent sampling location?

    This should be avoided whenever possible.

16. How is the sample mixed prior to withdrawal of a subsample for analysis?

    The sample should be thoroughly mixed. ✓

17. How is the subsample stored prior to analysis? ✓

    The sample should be refrigerated (4 degrees C) until about 1 hour before analysis, at which time it is allowed to warm to room temperature.

18. What is the cleaning frequency of the collection jugs? ✓

    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? should do.

    Rinsing lines with a chlorine solution every three months or more often where necessary is suggested.

## pH Test Review

1. How is the pH measured? ✓

   A meter should be used. Use of paper or a colorimetric test is inadequate and those procedures are not listed in Standard Methods (SM, p429).

2. How often is the meter calibrated? ✓

   The meter should be calibrated every day it is used.

3. What buffers are used for calibration? 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? SM 1485

   Standard Methods or the Ecology handout should be used. ✓

2. How often are BODs run? 3x's a week

   The minimum frequency is specified in the permit.

3. How long after sample collection is the test begun? 8 hrs

   The test should begin within 24 hours of composite sample completion ✓ (Ecology Lab Users Manual, p42). Starting the test as soon after samples are complete is desirable.

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

5. Is the distilled water made with a copper free still?

   Copper stills can leave a copper residual in the water which can be toxic to the test (SSM, p36).

6. Are any nitrification inhibitors used in the test? No What?

   2-chloro-6(trichloro methyl) pyridine or Hach Nitrification Inhibitor 2533 may be used only if carbonaceous BODs are being determined (SM, p527, #4g: SSM, p37).

7. Are the four nutrient buffers of powder pillows used to make dilution water? mix

   If the nutrients are used, how much buffer per liter of dilution water are added? ✓

   1 mL per liter should be added (SM, p527, #5a: SSM, p37).

8. How often is the dilution water prepared? 2-4 a week

   Dilution water should be made for each set of BODs run.

9. Is the dilution water aged prior to use? yes

   Dilution water with nitrification inhibitor can be aged for a week before use (SM, p528, #5b).

   Dilution water without inhibitor should not be aged.

10. Have any of the samples been frozen? No

    If yes, are they seeded?

    Samples that have been frozen should be seeded (SSM, p38).

11. Is the pH of all samples between 6.5 and 7.5? yes

    If no, is the sample pH adjusted?

    The sample pH should be adjusted to between 6.5 and 7.5 with 1N NaOH or 1N $H_2SO_4$ if 6.5 > pH >7.5 if caustic alkalinity or acidity is present (SM, p529, #5e1: SSM, p37).

    High pH from lagoons is usually not caustic. Place the sample in the dark to warm up, then check the pH to see if adjustment is necessary.

    If the sample pH is adjusted, is the sample seeded?

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

12. Have any of the samples been chlorinated or ozonated? yes

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

    How are they dechlorinated? thiosulfate

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

If chlorinated or ozonated, is the sample seeded? yes

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

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

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

14. How are DO concentrations measured? probe

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

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

How frequently is the meter calibrated? once a day

The meter should be calibrated before use. ✓

15. Is a dilution water blank run? yes

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

What is the usual initial DO of the blank? 7.5 - 8.0 ✓

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

What is the usual 5 day blank depletion? 0.0 - 0.2 mg/L ✓

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

16. How many dilations are made for each sample? 3 influent
2 effluent

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

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

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

18. How many bottles are made at each dilution? 1

How many bottles are incubated at each dilution? 1

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

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

19. Is the initial DO of each dilution measured? yes

What is the typical initial DO? 7.5-8.0

The initial DO of each dilution should be measured. It should approximate saturation (see #14). ✓

20. What is considered the minimum acceptable DO depletion after five days? ✓

What is the minimum DO 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, p531, #6: SSM, p41).

21. Are any samples seeded? yes

Which? 2° effluent

What is the seed source? 1° effluent

Primary effluent or settled raw wastewater is the preferred seed. Secondary treated sources can be used for inhibited tests (SM, p528, #5d: SSM, p41). ✓

How much seed is added to each sample? ½ 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, p529, #5d). ✓

How is the BOD of the seed determined? yes ✓

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

22. What is the incubator temperature? ✓

The incubator should be kept at 20 +/- 1 degree C (SM, p531, #5i: SSM, p40, #3).

How is incubator temperature monitored? water bath ✓

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? infrequent

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? ✓

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

23. Are water seals maintained on the bottles during incubation? ✓

Water seals should be maintained to prevent leakage of air during the incubation period (SM, p531, #5i: SSM, p40, #4).

24. Is the method of calculation correct? ✓

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

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

for unseeded samples;

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

for seeded samples;

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

Where:

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

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

**Total Suspended Solids Test Review**

Preparation

1. What reference is used for the TSS test? SM 1995, EPA 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, p95, footnote: SSM, p23)

3. What is the drying oven temperature? ✓

   The temperature should be 103-105 degrees C (SM, p96, #3a: SSM, p23).

4. Are any volatile suspended solids tests run? yes

   If yes, what is the muffle furnace temperature? ✓

   The temperature should be 550+/- 50 degrees C (SM, p98, #3: SSM, p23).

5. What type of filtering apparatus is used? Gelman 3 piece manifold

   Gooch crucibles or a membrane filter apparatus should be used (SM, p95, #2b: SSM, p23). ✓

6. How are the filters pre-washed prior to use? yes

   The filters should be rinsed three times with distilled water (SM, p23, #2: SSM, p23, #2).

   Are the rough or smooth sides of the filters up? rough ✓

   The rough side should be up (SM, p96, #3a: SSM, p23, #1) ✓

   How long are the filters dried? ✓

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

   How are the filters stored prior to use? ✓

   The filters should be stored in a desiccator (Ibid). ✓

7. How is the effectiveness of the desiccant checked?

   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?

   The sample should be measured with a wide tipped pipette or a graduated cylinder.

9. Is the filter seated with distilled water?

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

10. Is the entire measured volume always filtered? yes

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

11. What are the average and minimum volumes filtered?

| | Volume | |
|---|---|---|
| | Minimum | Average |
| Influent | | 50-100ml |
| Effluent | | 100ml |

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

| | Time |
|---|---|
| Influent | |
| Effluent | < one minute |

13. How long is filtering attempted before deciding that a filter is clogged?

    Prolonged filtering can cause high results due to dissolved solids being caught in the filter (SM, p96, #1b). We usually advise a five minute filtering maximum.

14. What do you do when a filter becomes clogged?

    The filter should be discarded and a smaller volume of sample should be used with a new filter.

15. How are the filter funnel and measuring device rinsed onto the filter following sample addition?

Rinse 3x's with approximately 10 mLs of distilled water each time (? ?).

16. How long is the sample dried? ✓

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

17. Is the filter thoroughly cooled in a desiccator prior to weighing? yes

The filter must be cooled to avoid drafts due to thermal ✓ differences when weighing (SM, p97, #3c: SSM, p97 #3c).

18. How frequently is the drying cycle repeated to assure constant filter weight has been reached (weight loss <0.5 mg or 4 percent, whichever is less: SM, p97, #3c)? yes

We recommend that this be done at least once every two months. ✓

19. Do calculations appear reasonable? yes

Standard Methods calculation (SM, p97, #3c).

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

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

**Fecal Coliform Test Review**

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

This review is for the MF technique.

2. Are sterile techniques used? yes

3. How is equipment sterilized? UV + autoclave

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

4. How is sterilization preserved prior to item use? ✓

   Wrapping the items in kraft paper or foil before they are sterilized protects them from contamination (Ibid.).

5. How are the following items sterilized?

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

6. How are samples dechlorinated at the time of collection? ✓

   Sodium thiosulfate (1 mL of 1% solution per 120 mLs (4 ounces) of sample to be collected) should be added to the collection bottle prior to sterilization (SM p856, #2: SSM p68, sampling).

7. Is phosphate buffer made specifically for this test?

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

8. What kind of media is used? ✓

   M-FC media should be used (SM, p896, SSM p66).

9. Is the media mixed or purchased in ampoules?

   Ampoules are less expensive and more convenient for under 50 tests per day (SSM, p65, bottom).

10. How is the media stored? ✓

    The media should be refrigerated (SM, p897, #1a: SSM p66, #5).

11. How long is the media stored? 2 weeks

    Mixed media should be stored no longer than 96 hours (SM, p897, #1a: SSM, p66, #5). Ampoules will usually keep from three to six months -- read ampoule directions for specific instructions.

12. Is the work bench disinfected before and after testing? ✓

This is a necessary sanitization procedure (SM, p831, #1f).

13. Are forceps dipped in alcohol and flamed prior to use? ✓

Dipping in alcohol and flaming are necessary to sterilize the forceps (SM, p889, #1: SSM p73, #4).

14. Is sample bottle thoroughly shaken before the test volume is removed? ✓

The sample should be mixed thoroughly (SSM, p73, #5).

15. Are special procedures followed when less than 20 mLs of sample is to be filtered?

10-30 mLs of sterile phosphate buffer should be put on the filter. The sample should be put into the buffer water and swirled, then the vacuum should be turned on. More even organism distribution is attained using this technique (SM, p890, #5a: SSM P73, #5).

16. Are special procedures followed when less than 1 mL of sample is to be filtered?

Sample dilution is necessary prior to filtration when <1 mL is to be tested (SM, p864, #2c: SSM p69).

17. Is the filter apparatus rinsed with phosphate buffer after sample filtration?

Three 20-30 mL rinses of the filter apparatus are recommended (SM, p891, #5b: SSM, p75, #7).

18. How soon after sample filtration is incubation begun? ✓

Incubation should begin within 20-30 minutes (SM, p897, #2d: SSM p77, #10 note).

19. What is the incubation temperature? ✓

44.5 +/- 0.2 degrees C (SM, p897, #2d: SSM, p75, #9).

20. How long are the filters incubated? ✓

24 +/- 2 hours (Ibid.).

21. How soon after incubation is complete are the plate counts made? ✓

The counts should be made within 20 minutes after incubation is complete to avoid colony color fading (SSM, p77, FC).

22. What color colonies are counted? blue ✓

    The fecal coliform colonies vary from light to dark blue (SM, p897, #2e: SSM, p78).

23. What magnification is used for counting?

    10-15 power magnification is recommended (SM, p898, #2e: SSM, p78).

24. How many colonies blue colonies are usually counted on a plate? ✓

    Valid plate counts are between 20 and 60 colonies (SM, p897, #2a: SSM, p78).

25. How many total colonies are usually on a plate? ✓

    The plate should have <200 total colonies to avoid inhibition due to crowding (SM, p893, #6a: SSM, p63, top).

26. When calculating results, how are plates with <20 or >60 colonies considered when plates exist with between 20 and 60 colonies? ✓

    In this case the plates with <20 or >60 colonies should not be used for calculations (SM, p898, #3: SSM, p78, C&R).

27. When calculating results how are results expressed if all plates have < 20 or > 60 colonies?

    Results should be identified as estimated. ✓

    The exception is when water quality is good and <20 colonies grow. In this case the lower limit can be ignored (SM, p893, #6a: SSM, p78, C&R).

28. How are results calculated? ✓

    Standard Methods procedure is (SM, p893, #6a: SSM, p79):

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