



Investigation into Recurring Toxicity of the Chinnet Company Columbia River Intake

Abstract

Daphnia pulex (water flea) acute bioassays were conducted on samples of Columbia River intake water from the Chinnet Company in Wenatchee, Washington approximately twice monthly between May and June 2001. The impetus for the monitoring program was data collected by Chinnet showing the intake became acutely toxic to *Daphnia* during the spring of 1999 and again in the spring of 2000.

More intensive sampling by the Washington State Department of Ecology during 2001 showed no evidence of toxicity. Chemical data collected during the monitoring program revealed no unusual levels of metals or detections of pesticides, herbicides, or other synthetic organic compounds.

This segment of the Columbia River is on the 1998 303(d) list, based on a Chinnet intake sample failing a water column bioassay in 1995 (*Ceriodaphnia* reproduction). It is recommended that this segment be reassigned to Part 5 of the 2002 proposed 303(d) list: *Undetermined Status*.

Acknowledgments

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Introduction

The Chinet Company is located in Wenatchee on the west bank of the Columbia River, approximately two miles upstream of the Wenatchee River confluence (Figure 1). Chinet produces apple packing trays and related products from recycled newspaper and cardboard.

Effluent testing to comply with the Washington State Department of Ecology (Ecology) Whole Effluent Toxicity (WET) regulation (Chapter 173-205 WAC) showed serious acute toxicity to daphnid water fleas (*Ceriodaphnia dubia*) from the late winter through spring of 1998. As a result, Chinet expanded their testing in 1999 to include the intake water, which is drawn from the Columbia River.

Acute bioassays of the intake with *Daphnia pulex* showed it became acutely toxic during the spring of 1999 and again in the spring of 2000 (Table 1). During these periods, as much as a 10-fold dilution was required to make the samples non-toxic. The toxicity was dramatic, with 75-100% mortality occurring in the first day of exposure. The relationship between intake and effluent toxicity, however, was unpredictable. Chinet completed the toxicity monitoring required in their NPDES permit in February 2001.

The Columbia River at Wenatchee (segment WA-CR-1040) is on the 1998 303(d) list for a water column bioassay failure. The listing is based on an earlier Chinet intake sample collected in July 1995 (CH2M Hill, 1995). In that sample there was a significant effect on reproduction of *Ceriodaphnia dubia* (11 young per adult vs. 19 in the control). Survival of *Ceriodaphnia*, and of *Daphnia pulex* in a companion test, was 100%.

The degree of toxicity observed in Chinet's intake in 1999 and 2000 is rarely encountered in the intakes or effluents of other industries in Washington (WET program database). In view of the potential implications for the Columbia River, the Ecology Water Quality Program proposed more intensive monitoring of the intake. The monitoring was subsequently conducted during the spring of 2001 by the Ecology Environmental Assessment Program.

The goal of the investigation was to identify the cause of the recurring toxicity and determine if it is a localized or river-wide problem. The objectives were to:

- Conduct *Daphnia pulex* acute bioassays of Chinet intake water.
- Conduct *Daphnia pulex* acute bioassays of water from the opposite (east) bank of the Columbia River, in conjunction with the intake sampling.
- Analyze the intake samples for pesticides and metals, and screen for additional non-target organic and inorganic chemicals.
- If and when significant toxicity is found in an intake sample, perform a Toxicity Identification Evaluation (TIE) on that sample.
- Identify the cause of toxicity and source location (west bank or river-wide) through a comparison of results from the acute bioassays, chemistry, TIE, and published data on the toxicity of detected chemicals to *Daphnia*.

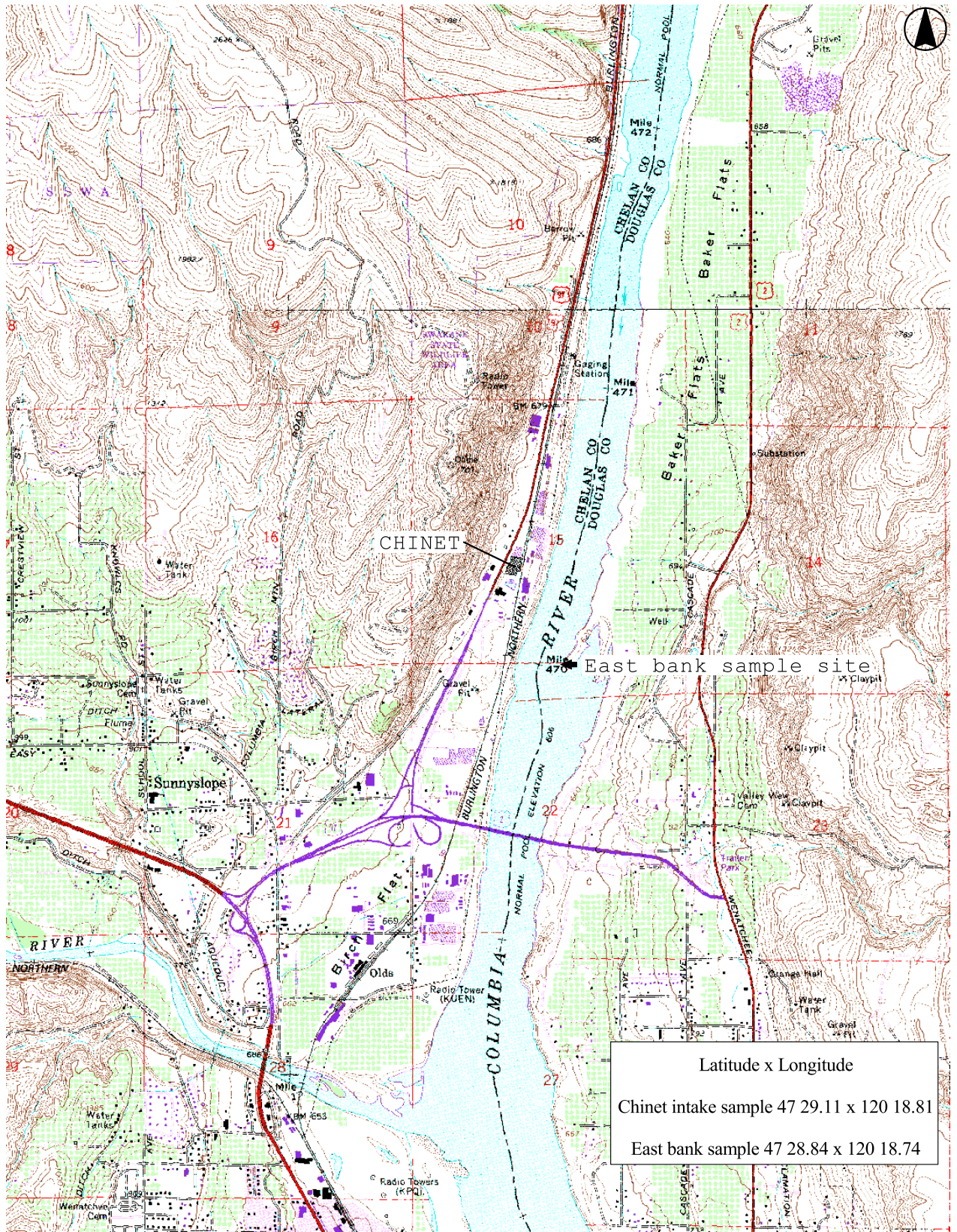


Figure 1. Location of the Chinet Company, Wenatchee, WA

Table 1. Chinnet Columbia River Intake: Acute WET Test Results for *Daphnia pulex* [% survival in 100% river water, NOEC/ LOEC/LC₅₀ in % river water]

| Sample Date | Test Duration | % Survival | NOEC | LOEC | LC ₅₀ |
|------------------|-----------------|------------|------------|-------------|------------------|
| 17-Jul-95 | 48 hours | 100 | 100 | > 100 | -- |
| 22-Feb-99 | 48 hours | 90 | 100 | > 100 | -- |
| 22-Feb-99 | 48 hours | 100 | 100 | > 100 | -- |
| 7-Apr-99 | 24 hours | 25 | 50 | 100 | 78.1 |
| 7-Apr-99 | 48 hours | 0 | 50 | 100 | 63.6 |
| 11-May-99 | 24 hours | 0 | 8.3 | 14.3 | 8.9 |
| 11-May-99 | 48 hours | 0 | 8.3 | 14.3 | 7.5 |
| 20-Mar-00 | 24 hours | 25 | 8.3 | 14.3 | 76.6 |
| 20-Mar-00 | 48 hours | 0 | 50 | 100 | 13.9 |
| 3-Apr-00 | 48 hours | 100 | 100 | > 100 | -- |
| 9-May-00 | 48 hours | 100 | 100 | > 100 | -- |
| 12-Jun-00 | 48 hours | 90 | 100 | > 100 | -- |
| 31-Aug-00 | 48 hours | 85 | 100 | > 100 | -- |
| 13-Sep-00 | 48 hours | 100 | 100 | > 100 | -- |
| 2-Oct-00 | 48 hours | 95 | 100 | > 100 | -- |
| 6-Nov-00 | 48 hours | 95 | 100 | > 100 | -- |
| 4-Dec-00 | 48 hours | 95 | 100 | > 100 | -- |
| 12-Feb-01 | 48 hours | 100 | 100 | > 100 | -- |

Monitoring Program

Water samples were collected on nine occasions between March 6 and June 20, 2001. *Daphnia pulex* acute bioassays were done on samples from the Chinnet intake and from the Columbia River on the opposite bank (Figure 1). The purpose of the opposing bank samples was to rule out a localized source of toxicity upstream of Chinnet (i.e., is the whole river affected?). Additional samples were collected for analysis of pesticides, herbicides, and metals; and screened for other non-target organic and inorganic chemicals. Blue-green algal toxins (microcystins) were also analyzed. Ancillary water quality parameters included temperature, pH, conductivity, hardness, total suspended solids, turbidity, and ammonia.

The trigger for initiating a TIE was to be when the daphnids began dying down to 14% on the first day of the test, as they did in May 1999 and March 2000. Guidance for conducting a TIE can be found in EPA (1991, 1993a,b). Briefly, it involves fractionating the sample to isolate the class of chemicals causing the toxic effect and, potentially, identifying the causative agent.

A Quality Assurance Project Plan was prepared for this study (Johnson, 2001).

Methods

Field

All samples were collected as simple grabs. Chinet intake samples were taken from the same tap used in the company's monitoring program. The east bank samples were taken by wading into the river. pH measurements were obtained with an Orion Model 250A meter. Temperature was measured with a precision thermometer or taken from the pH meter.

Table 2 lists the container, sample size, preservation, and holding time for each parameter. Sample containers were obtained from the CH2M Hill bioassay laboratory in Corvallis, Oregon or the Ecology Manchester Environmental Laboratory. Excess intake sample was collected on each occasion, in the event that a TIE was indicated.

Table 2. Sample Containers, Preservation, and Holding Times for Chinet Investigation

| Analysis | Sample Container | Volume | Preservation | Holding Time |
|-------------------------|--------------------------|------------|--|--------------|
| GC/AED organics | glass / Teflon lid liner | 1 gallon | 4° C | 7 days |
| herbicides | glass / Teflon lid liner | 1 gallon | 4° C | 7 days |
| ICP/MS metals | Teflon | 500 mL | HNO ₃ to pH<2; 4° C | 6 months |
| mercury | Teflon | 500 mL | HNO ₃ to pH<2; 4° C | 28 days |
| total suspended solids | polyethylene | 100 mL | 4° C | 7 days |
| hardness | polyethylene | 125 mL | HNO ₃ to pH<2; 4° C | 6 months |
| conductivity | polyethylene | 500 mL | 4° C | 28 days |
| ammonia | polyethylene | 125 mL | H ₂ SO ₄ to pH<2; 4° C | 28 days |
| blue-green toxins | glass / Teflon lid liner | 40 mL | 4° C | 7 days |
| <i>Daphnia</i> bioassay | polyethylene | 1 gallon | 4° C | 36 hours |
| TIE | polyethylene | 10 gallons | 4° C | unknown |

All samples were put on ice immediately upon collection. The bioassay samples were FedEx'd to CH2M Hill on the day of collection, to arrive the next morning. The chemistry samples were held in a secure cooler at Ecology Headquarters, then transported to Manchester Laboratory the day after collection. Chain-of-custody was maintained.

Laboratory

Table 3 shows the analytical methods used, approximate reporting limits, and laboratories that did the work. More detailed information on how the bioassays were conducted follows below.

Table 3. Analytical Methods, Approximate Reporting Limits, and Laboratories for Chinnet Investigation

| Parameter | Reporting Limit | Units | Method | Laboratory |
|---------------------------------------|-----------------|----------|---------------------------------|-------------|
| OC pesticides | 0.01 - 0.1 | ug/L | GC/AED -EPA 8085 | Manchester |
| OP pesticides | 0.01 - 1 | ug/L | GC/AED -EPA 8085 | Manchester |
| N pesticides | 0.01 - 1 | ug/L | GC/AED -EPA 8085 | Manchester |
| chlorophenoxy herbicides | 0.002 | ug/L | GC/AED -EPA 8085 | Manchester |
| organics screen | -- | -- | GC/AED -EPA 8085 | Manchester |
| Al, Sb, As*, Be, Cd, Cr, Co, Cu, | | | | Manchester |
| Pb*, Mn, Ni, Se*, Ag, V, Zn | 0.1 - 0.5 | ug/L | ICP/MS -EPA 200.8 | Manchester |
| Hg | 0.05 | ug/L | CVAA - EPA 245.7 | Manchester |
| TI | 0.05 | ug/L | GFAA - EPA 279.2 | Manchester |
| total suspended solids | 1 | mg/L | EPA 160.2 | Manchester |
| conductivity | 1 | umhos/cm | SM 2510 | Manchester |
| hardness | 1 | mg/L | SM 2340B | Manchester |
| ammonia | 0.01 | mg/L | EPA 350.1 | Manchester |
| Blue-green microcystins | 0.1 | ug/L | SOP# 04-02-009 | King County |
| <i>Daphnia pulex</i> acute bioassay | na | na | EPA (1993c) & Ecology (1997) | CH2M Hill |
| Toxicity Identification Evaluation | na | na | EPA (1991, 1993a,b) | CH2M Hill |

Note: metals analyzed as total recoverable

*Also analyzed by GFAA in some samples; EPA methods 206.2 (As), 270.2 (Se), and 239.2 (Pb)

The *Daphnia pulex* tests were performed according to *Methods for Measuring the Acute Toxicity of Effluents to Freshwater and Marine Organisms* (EPA, 1993c) and *Laboratory Guidance and Whole Effluent Toxicity Test Review Criteria* (Ecology, 1997). The *Daphnia* were obtained from CH2M Hill's in-house cultures and were less than 24 hours old at test initiation. All organisms tested were maintained during culturing, acclimation, and testing as prescribed by EPA.

Moderately hard reconstituted water with a hardness of 94 mg/L as CaCO₃, alkalinity of 68 mg/L as CaCO₃, and pH of 8.1 – 8.2 was used as dilution water for the tests. The concentrations tested were 8.3, 14.3, 25, 50, and 100 percent sample, with dilution water alone for the control. The tests were run in quadruplicate with five organisms per replicate.

The samples were monitored on arrival for hardness, total residual chlorine, ammonia, and temperature. The tests were monitored at initiation for pH, conductivity, and dissolved oxygen, and every 24 hours thereafter for mortality. Temperature was adjusted prior to test initiation and monitored continuously during the 48-hour test period.

Data Quality

Dissolved oxygen concentrations during the *Daphnia* tests remained at 40 percent saturation or greater and test temperatures remained in the range of 20+/-1 °C. The tests proceeded without interruption or incidence that would have affected the results. Monthly reference toxicant tests conducted using sodium chloride indicated that the test organisms were within their appropriate sensitivity range, based on EPA guidelines. The raw data from the bioassays are available from the author.

Manchester Laboratory prepared written quality assurance reviews on the quality of the chemical data for this project. The reviews include an assessment of sample condition on receipt at the laboratory, compliance with holding times, instrument calibration, procedural blanks, laboratory control samples, standard reference material, matrix spike and matrix spike duplicate recoveries, and duplicate sample analyses. No problems were encountered that compromise the accuracy, validity, or usefulness of the data. The complete quality assurance reviews and chemical data for the metals and organics analyses are provided in Appendix A. Copies of the reviews and complete data for general chemistry parameters are available from the author.

The analysis for blue-green microcystins followed procedures described in the Strategic Diagnostics Inc. SOP for the EnviroGuard Microcystin Plate Kit. The data for all samples were acceptable. Laboratory bench sheets, printouts from the Microplate reader, and control charts are available from the author.

Results

Data showing the general water quality characteristics of the Chinnet intake and opposing bank samples are summarized in Table 4. The intake is located in a deep part of the channel (>20 feet, exact depth uncertain), while the bank sample was taken in approximately 2 feet of water. As a result, water quality conditions were slightly different, with the opposing bank being marginally lower in temperature, pH, and conductivity, but higher in turbidity and suspended solids. Ammonia was detectable on one occasion only, in an intake sample.

Table 4. Water Quality Data: Chinnet Columbia River Intake and Opposite (East) Bank

| Sample Date | Location | Temp. (°C) | pH (S.U.) | Conduct. (umhos/cm) | Turbidity (NTU) | TSS (mg/L) | Ammonia (mg/L) |
|-------------|----------------|------------|-----------|---------------------|-----------------|------------|----------------|
| 6-Mar-01 | Chinnet Intake | 4.6 | 7.0 | 159 | 0.7 | 1 | <0.010 |
| | Opposing Bank | 2.9 | 6.8 | 154 | 1.0 | 2 | <0.010 |
| 19-Mar-01 | Chinnet Intake | 5.9 | 7.9* | 174 | 0.5 | 1 | <0.010 |
| | Opposing Bank | 4.7 | 8.1* | 154 | 0.9 | 2 | <0.010 |
| 3-Apr-01 | Chinnet Intake | 8.9 | 7.5 | 187 | 0.5 | 1 | <0.010 |
| | Opposing Bank | 8.9 | 8.1 | 155 | 1.1 | 6 | <0.010 |
| 16-Apr-01 | Chinnet Intake | 7.5 | 7.4 | 160 | 0.9 | 2 | <0.010 |
| | Opposing Bank | 6.5 | 6.9 | 156 | 1.1 | 2 | <0.010 |
| 30-Apr-01 | Chinnet Intake | na | 8.5* | 158 | 1.3 | 2 | <0.010 |
| | Opposing Bank | 8.6 | 8.2* | 158 | 1.2 | 3 | <0.010 |
| 14-May-01 | Chinnet Intake | 10.8 | 8.4 | 160 | 1.4 | 2 | <0.010 |
| | Opposing Bank | 9.9 | 8.8 | 159 | 1.4 | 3 | <0.010 |
| 29-May-01 | Chinnet Intake | 13.8 | 7.3 | 160 | 0.7 | <1 | <0.010 |
| | Opposing Bank | 12.6 | 8.1 | 147 | 0.9 | 1 | <0.010 |
| 6-Jun-01 | Chinnet Intake | 13.0 | 7.8 | 152 | 0.7 | 1 | <0.010 |
| | Opposing Bank | 12.0 | 8.1 | 145 | 1.1 | 2 | <0.010 |
| 20-Jun-01 | Chinnet Intake | 14.8 | 8.0 | 142 | 0.7 | 2 | 0.42 |
| | Opposing Bank | 13.9 | 8.3 | 138 | 1.1 | 2 | <0.010 |

*laboratory measurement

Metals and organic compounds were only analyzed for the March 6, April 3 (mercury only), April 16 (herbicides only), and June 20 samples (Appendix A). The analyses were limited to the intake, except organics were also analyzed in the opposite bank sample of June 20. Metals concentrations were similar to what has been reported for other parts of the Columbia River drainage. No pesticides, herbicides, or other synthetic organic compounds were detected, other than a trace amount (0.07 ug/L) of an unidentified chlorine-containing compound in the April 3 intake sample.

Blue-green toxins were analyzed for each sampling event, and in both the intake and bank sample. All results were less than the method detection limit (0.1 ug/L). Microcystin concentrations between 0.057 and 0.076 ug/L may have been present in some samples, but the levels were too low to quantify.

Results of the *Daphnia* bioassays are summarized in Table 5. No significant toxicity was observed. Survival averaged over 95% and was either not different from or only slightly lower than the laboratory control. Similar results were obtained for both sides of the river. The NOECs (No Observable Effect Concentration) and LC₅₀s (Lethal Concentration to 50% of test organisms) were all 100% and >100%, respectively (percent river water).

Table 5. Toxicity Monitoring Data: Chinnet Columbia River Intake and Opposite (East) Bank [*Daphnia pulex* survival in 100% river water, NOEC/LC₅₀ in percent river water]

| Sample Date | Test Duration | Chinnet Intake | | | Opposite Bank | | |
|-------------|---------------|----------------|------|------------------|---------------|------|------------------|
| | | % Survival | NOEC | LC ₅₀ | % Survival | NOEC | LC ₅₀ |
| 6-Mar-01 | 48 hours | 100 | 100 | >100 | 100 | 100 | >100 |
| 19-Mar-01 | 48 hours | 100 | 100 | >100 | 100 | 100 | >100 |
| 3-Apr-01 | 48 hours | 95 | 100 | >100 | 90 | 100 | >100 |
| 16-Apr-01 | 48 hours | 100 | 100 | >100 | 100 | 100 | >100 |
| 30-Apr-01 | 48 hours | 85 | 100 | >100 | 90 | 100 | >100 |
| 14-May-01 | 48 hours | 95 | 100 | >100 | 95 | 100 | >100 |
| 29-May-01 | 48 hours | 100 | 100 | >100 | 100 | 100 | >100 |
| 6-Jun-01 | 48 hours | 100 | 100 | >100 | 100 | 100 | >100 |
| 20-Jun-01 | 48 hours | 100 | 100 | >100 | 100 | 100 | >100 |

Recommendations

Although intensive monitoring failed to detect significant toxicity in the Columbia River at the Chinnet facility during 2001, the toxicity episodes of 1999 and 2000 remain a concern. The winter and spring of 2001 were exceptionally dry, so there is a possibility that water quality conditions in the Columbia were not representative of what typically occurs. There is also the possibility of unknown (unpermitted) discharges nearby on the west bank upstream of Chinnet. Discharge practices of current industries may bear closer examination.

River flows were substantially higher when toxicity was observed in 1999/2000 (121,180 – 142,670 cfs) than during the present study (51,250 – 106,680 cfs; 75,930 cfs average). If bank storage contaminants upstream of Chinnet were the source of toxicity, then the water levels in 2001 may have been too low to mobilize them.

We recommend that this segment of the Columbia River be reassigned to Part 5 of the 2002 proposed 303(d) list: *Undetermined Status*. This part of the list is reserved for waterbodies where there are indications of a water quality problem, but where the data are contradictory. That would allow Ecology or other groups to track this issue and pursue opportunities for additional sampling.

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Appendix A

Metals and Organics Data for Chinet Investigation

The appendix is not available electronically. For a printed copy, order this report through the Ecology Publications Office. See page 2 for details.