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Polychlorinated Dibenzo-*P*-Dioxins and Dibenzofurans in Upper Columbia River Suspended Particulate Matter, 1990-1994

by
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Abstract

We analyzed polychlorinated dibenzo-*p*-dioxin (PCDD) and dibenzofuran (PCDF) concentrations in suspended particulate matter (SPM) collected from the Columbia River at Northport during 1990, 1992, 1993, and 1994 autumn low-flow conditions. The primary objective was to document the effects of pollution abatement efforts by the Celgar pulp mill in Castlegar, British Columbia, 46 river miles upstream of Northport. We also estimated PCDD/PCDF loads in the river and compared the 2,3,7,8-tetrachlorodibenzo-*p*-dioxin (2,3,7,8-TCDD) loads to the target for the watershed north of the international boundary as detailed in EPA's total maximum daily load (TMDL) for the Columbia River basin.

Concentrations of 2,3,7,8-tetrachlorodibenzofuran (2,3,7,8-TCDF), the major congener detected, decreased by two orders of magnitude; and the daily 2,3,7,8-TCDF load in Columbia River SPM decreased 300-fold from 1990 to 1994. The decline in 2,3,7,8-TCDF concentrations agreed well with improvements and discharge reductions reported by Celgar. 2,3,7,8-TCDD, the most toxic congener, was not detected in any samples at quantitation limits ranging from 0.4 to 1.8 pg/g. *Maximum possible* 2,3,7,8-TCDD loads in SPM were 0.5 mg/day or less, much lower than the watershed target specified in the TMDL (2.3 mg/day). By estimating dissolved 2,3,7,8-TCDD concentrations, we calculated the total *maximum possible* 2,3,7,8-TCDD loads were about one-half the watershed target in 1990 and 1994, and slightly above the target load in 1992 and 1993.

Toxicity equivalent (TEQ) concentrations, the PCDD/PCDF-associated toxicity expressed as equivalent concentrations of 2,3,7,8-TCDD, decreased seven-fold and the daily TEQ load decreased sixteen-fold from 1990 to 1994. These declines were due mainly to lower 2,3,7,8-TCDF concentrations. Congener profiles changed from year to year, yet aside from 2,3,7,8-TCDF, there was no clear trend in concentrations of SPM-bound PCDDs/PCDFs. We do not have an explanation for the shifting congener patterns but there is little evidence that it was directly related to Celgar discharges.

We recommend additional rounds of SPM collection to verify the findings of this study, check for the presence of 2,3,7,8-TCDD, and determine if sediment resuspension during high-flows contributes significant PCDD/PCDF loads to the Columbia River. Further monitoring should also include analysis for dissolved PCDDs/PCDFs.

Acknowledgments

◊ Sample handling and analysis was placed in the capable hands of Myrna McIntosh, Sally Cull, Debbie Lacroix, Aileen Smith, Karin Fedderson, Stuart Magoon, and Will White of the Ecology Manchester Laboratory, for which we are thankful.

◊ We are also grateful to Jim McLaren of the Celgar Pulp Co. for providing a tour of the Castlegar plant and explaining the many details of pulp production.

◊ Todd Vilen of Triangle Laboratories, Inc. provided valuable information on dioxin analysis.

◊ Larry Goldstein reviewed the report, and Joan LeTourneau formatted the final report.

Introduction

Polychlorinated dibenzo-*p*-dioxins (PCDDs) and dibenzofurans (PCDFs) are compounds formed during combustion of materials containing chlorinated organics, as trace contaminants in chemical production, and as unintended by-products of industrial processes such as chlorine bleaching of wood pulp. Much study and attention has been given to PCDDs/PCDFs in the upper Columbia River and Lake Roosevelt after it was discovered that chlorine-bleaching kraft pulp mills may be a significant source of these chemicals (Amendola, 1987; EPA, 1988). Concerns about the presence of PCDDs/PCDFs in the aquatic environment are due to their toxicity to a variety of organisms, environmental persistence, and tendency to bioaccumulate, especially in fish.

There are 210 PCDD/PCDF congeners, of which 17 are considered toxic. The most toxic and widely studied of these is 2,3,7,8-tetrachlorodibenzo-*p*-dioxin (2,3,7,8-TCDD), often referred to as dioxin. 2,3,7,8-tetrachlorodibenzofuran (2,3,7,8-TCDF) is probably the most important congener in the vicinity of bleached kraft pulp mills due to its pervasiveness and toxicity (one-tenth that of 2,3,7,8-TCDD). Other PCDDs/PCDFs, including 2,3,7,8-TCDD, may be found in the aquatic environment near pulp mills, but generally at much lower concentrations than 2,3,7,8-TCDF.

PCDD/PCDF contamination in the upper Columbia River was first documented by Environment Canada when they reported very high 2,3,7,8-TCDF concentrations in lake whitefish (*Coregonus clupeaformis*) and bottom sediments downstream of the Celgar bleached kraft pulp mill in Castlegar, British Columbia, which has operated on the banks of the Columbia River since 1960 (Figure 1) (Mah *et al*, 1989). The Washington State Department of Ecology (Ecology) responded by conducting a reconnaissance survey to determine if PCDDs/PCDFs were detectable in Lake Roosevelt fish, findings of which indicated that further sampling was warranted (Johnson, 1990).

These surveys provided the impetus for subsequent investigations of PCDDs/PCDFs in the upper Columbia River and Lake Roosevelt (B.C. Ministry of Environment, 1990; Johnson *et al.*, 1991a,b,&c; Boyle *et al.*, 1992; Serdar *et al.*, 1993 & 1994). Most of these investigations have focused on residues in fish in order to assess human health risks. Although a discussion of the fish data is beyond the scope of this report, it is noteworthy that contamination of certain species was at one time substantial enough to elicit consumption advisories on both sides of the border (Kirkpatrick, 1989; Gebbie, 1990).

Ecology first analyzed suspended particulate matter (SPM) from the Columbia River at Northport in 1990 as a way to estimate PCDD/PCDF concentrations in the water column (Johnson *et al.*, 1991c). Because of their tendency to sorb to sediments, PCDDs/PCDFs are readily detectable in SPM compared to analysis of whole water. The SPM data were used in turn to estimate PCDD/PCDF loads to Lake Roosevelt and assist Ecology's Water Quality Program and the U.S Environmental Protection Agency

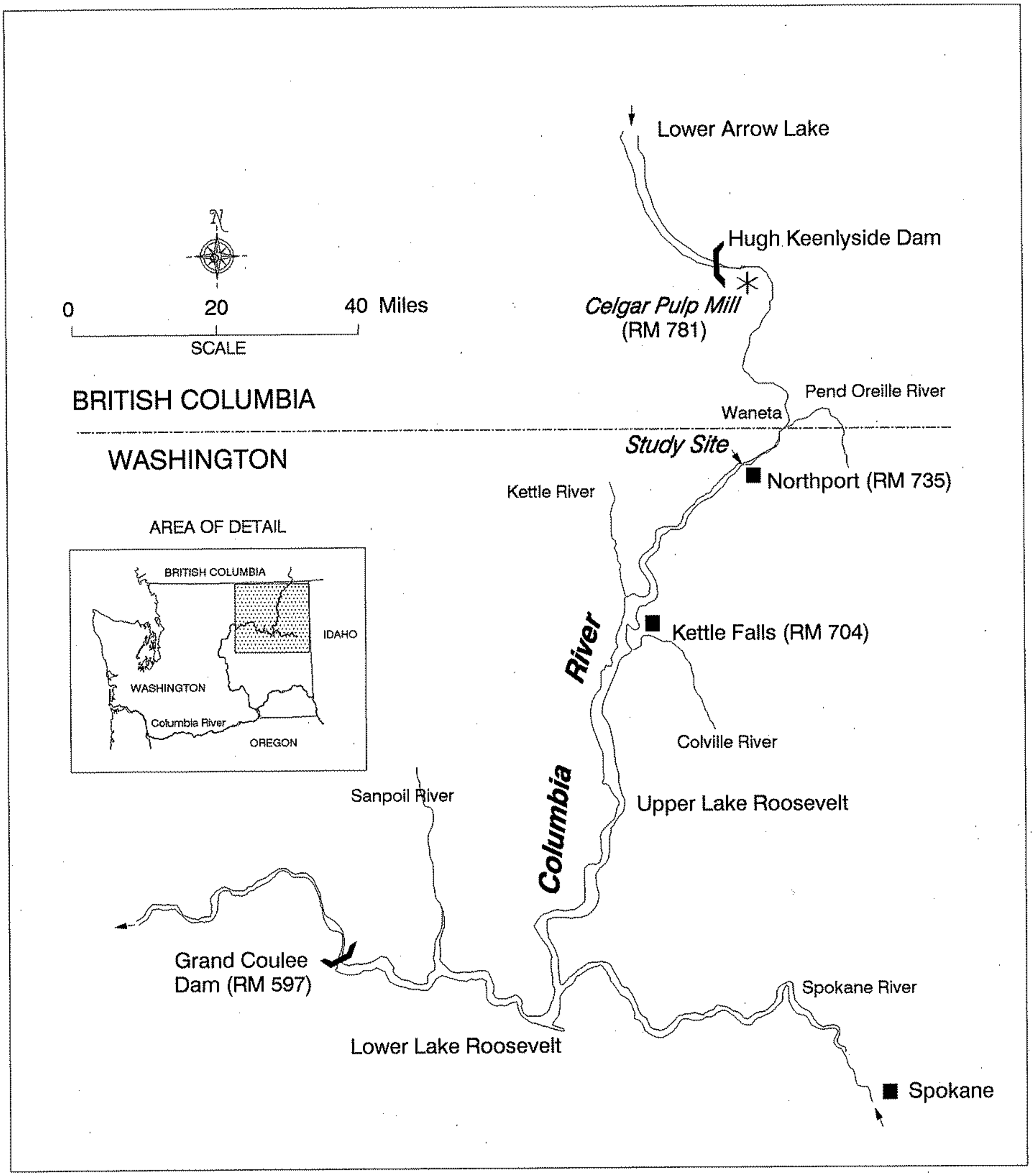

Figure 1. Location of the Northport Study Site and the Upper Columbia River

(EPA) in refining the Columbia River total maximum daily load (TMDL) to limit the discharge of 2,3,7,8-TCDD to the Columbia River basin. The TMDL, finalized by EPA in 1991, identifies a loading capacity or watershed target of 2.31 mg 2,3,7,8-TCDD/day for the Columbia River north of the international boundary (EPA, 1991).

Celgar Expansion and Modernization

In 1991, the Canadian federal government and British Columbia provincial government approved an expansion and modernization plan for the mill to be completed by July 1, 1993. In addition to more than doubling its pulp output to 1200 air-dried tonnes per day, the plan called for process changes and secondary wastewater effluent treatment (previously discharged to the Columbia River untreated) which were expected to reduce PCDDs/PCDFs in Celgar's final effluent to undetectable levels (Celgar Expansion Review Panel, 1991). Celgar also reported more immediate pollution reductions in 1991, including increased substitution of chlorine dioxide for chlorine in pulp bleaching, use of hydrogen peroxide in the delignification process, and removal of recovery boiler scrubber water which contained dibenzofuran, a PCDF precursor.

Ecology's Trend Monitoring Program

With the news of ongoing and planned improvements at Celgar, Ecology decided to conduct long-term monitoring in the upper Columbia River and Lake Roosevelt. EPA provided Ecology with a Clean Lakes grant to initiate contaminant monitoring during 1992-1993. The primary goal of the trend monitoring was to document the effects of pollution controls being implemented by Celgar, as well as the Cominco smelter[1].

To achieve this goal, Ecology decided to build upon the data collected during 1990 as a basis for continued monitoring of PCDDs/PCDFs. Among the sample media analyzed during 1990, SPM and lake whitefish tissues were selected for trend monitoring mainly due to their elevated concentrations of 2,3,7,8-TCDF, and to a lesser extent, 2,3,7,8-TCDD in whitefish. SPM was viewed as a useful gauge of instantaneous PCDDs/PCDFs levels in the water column and concurrent discharges by Celgar. Lake whitefish, on the other hand, were considered useful for longer-term monitoring due to their accumulation of these persistent compounds over time.

[1] Cominco Ltd. operations in Trail, B.C., located on the banks of the Columbia River approximately 11 river miles above the international boundary, include the world's largest integrated lead-zinc smelter and refinery. It is considered to be the major source of metals contamination in the upper Columbia River and upper Lake Roosevelt. Ecology's monitoring program also included collection of data to analyze trends in metals contamination. The metals data are included in Serdar *et al.*, (1993 & 1994) and several forthcoming Ecology reports.

Measurable trends in whitefish 2,3,7,8-TCDF and 2,3,7,8-TCDD concentrations were not expected for several years. However, improvements at Celgar were translated into significant declines of these compounds in muscle tissues between 1990 and 1993 (Serdar *et al.*, 1994). For instance, 2,3,7,8-TCDF in whitefish muscle decreased from a mean concentration of 126 pg/g (parts per trillion) in 1990 to 62 pg/g in 1992, then declined further to 33 pg/g in 1993. There was also a four-fold decrease in 2,3,7,8-TCDD concentrations, with most of the decline occurring between 1990 and 1992. Analysis of whitefish tissues by Ecology was dropped subsequent to 1993 to avoid duplicating part of an EPA survey of PCDDs/PCDFs in Lake Roosevelt fish during 1994.

Results of previous monitoring for PCDDs/PCDFs in the upper Columbia River and Lake Roosevelt have been reported in *Interim Report on Monitoring Contaminant Trends in Lake Roosevelt* (Serdar *et al.*, 1993) and *Contaminant Trends in Lake Roosevelt* (Serdar *et al.*, 1994). A more detailed analysis of samples collected during 1990 may also be found in *PCDDs/PCDFs in Columbia River Suspended Particulate Matter* (Johnson *et al.*, 1991c) and *Polychlorinated Dioxins and -Furans in Lake Roosevelt (Columbia River) Sportfish* (Johnson *et al.*, 1991a).

Objectives

The present report contains results of monitoring PCDDs/PCDFs in SPM during 1990-1994.

The objectives were to:

- Document the effects of pollution controls instituted by Celgar.
- Estimate PCDD/PCDF loads in the Columbia River at Northport and to Lake Roosevelt downstream.
- Compare the 2,3,7,8-TCDD load to the target load for the Columbia River watershed north of the international boundary, as detailed in EPA's TMDL for the Columbia River basin.

Methods

Site Selection

We selected Northport (Figure 2) as a sampling site because it is an accessible, free-flowing reach of the river upstream of the large depositional areas (*i.e.*, PCDD/PCDF sinks) in Lake Roosevelt (Johnson *et al.*, 1991b). At Columbia River mile 735, Northport is approximately 10 river miles below the international border and 46 river miles below Celgar. Samples were collected off a man-made gravel bar extending into the Columbia River from the left bank, near the Northport boat launch.

The reach between Northport and the international border is in a relatively natural state. Aside from a few houses and a two-lane highway, there is little development along the banks. The substrate is sandy, with gravel and cobble-sized material in riffle areas; there is very little fine material (≤0.062 mm) in the sediments (Johnson *et al.*, 1988). The watershed of the border reach is primarily forested, with several small tributary streams. The Pend Oreille River enters the Columbia on the left bank just north of the Canada border, contributing 27% of the mean annual Columbia River flow at the border (Butcher, 1992).

Sample Collection

Sampling of Columbia River SPM in 1990, 1992, 1993, and 1994 was conducted in a nearly identical manner. For all years, a three-day composite sample was collected during low river flow in late September or early October.

River water was pumped from an intake situated in 12-ft deep water (5-ft in 1990) approximately 50 feet offshore in the main current of the river. The depth of the intake was periodically adjusted to 2, 5, and 10-ft depths (1 and 4-ft in 1990) to approximate a depth-integrated sample. The water was delivered via a peristaltic pump to two Alfa-Laval Sedisamp II continuous-flow centrifuges (model 101IL) which extracted SPM from whole water.

To avoid sample contamination, all surfaces coming in contact with the samples were pre-cleaned by scrubbing with Liquinox® detergent, followed by sequential rinses with hot tap water, nitric acid (except 1990), de-ionized water, acetone, and hexane. Tubing and fittings were Teflon® or Teflon-lined except for Silastic® tubing on the peristaltic pump. Nalgene® tubing was used for the intake line in 1994. Centrifuge bowl parts are constructed of high quality stainless steel.

Table 1 compares the flow rate, centrifuge run time, amount of sample obtained, SPM removal efficiency, and Columbia River flow among years. The centrifuge removal efficiency is based on data from laboratory analysis of total suspended solids (TSS) in

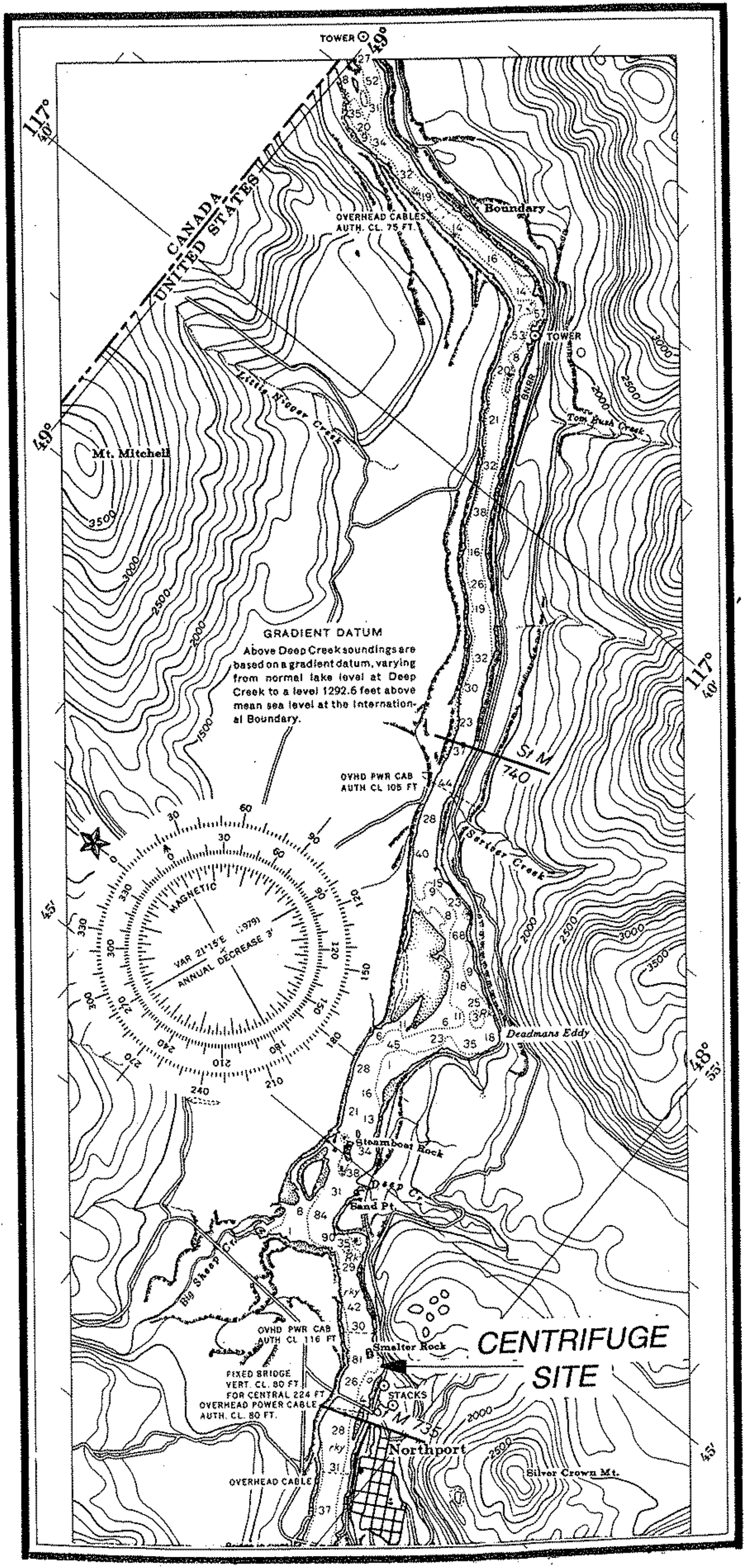

Figure 2. SPM Sampling Site

water samples and from pump-flow measurements. Flow data were obtained from the U. S. Geological Survey NASQAN Station 12399500 (Columbia River @ International Boundary).

Table 1. SPM Sample Collection Parameters, 1990-1994.

	1990 (Oct 9-11)	1992 (Sep 29-Oct 2)	1993 (Oct 5-7)	1994 (Oct 10-12)
Average combined flow rate to centrifuges (L/h)	270	170	170	330
Centrifuge run time (h)	57	59	68	57
Volume of water centrifuged (L)	15,450	10,200	11,540	18,925
Amount of SPM obtained (g, wet)	90	68	81	97
% Solids	26.2	21.2	16.4	16.0
Dry weight of sample (g)	23.6	14.4	13.3	15.5
SPM removal efficiency (%)	76	100	61	unknown
Average flow at border (m^3/s)	1,745	2,662	1,628	1,689

SPM accumulated by the centrifuge was scraped from the centrifuge bowl with a Teflon-coated spatula and placed in amber glass jars specially cleaned for trace organics analysis. Water samples were also collected at eight to fourteen hour intervals for analysis of specific conductance, total organic carbon (TOC), dissolved organic carbon (DOC), TSS, pH, and temperature. Samples for DOC were not analyzed in 1990. Water samples for TOC and DOC analysis were preserved with sulfuric acid to pH<2. DOC samples were filtered with a 0.45 µm extraction disc in the field prior to acidification. Measurements of pH and temperature were done in the field. All samples for laboratory analysis were kept on ice while in the field.

Sample Analysis

SPM samples were analyzed for all 2,3,7,8-substituted PCDDs and PCDFs using isotope dilution, high resolution GC/MS EPA Method 8290 or 1613. SPM samples from 1990, 1992, and 1993 were analyzed using EPA 8290 by: Alta Analytical Laboratory in El Dorado Hills, CA (1990); Enseco California Analytical Laboratory in West Sacramento, CA (1992); and Triangle Laboratories in Research Triangle Park, NC (1993). The 1994 sample was analyzed by Pacific Analytical in Carlsbad, CA using EPA 1613.

Methods 8290 and 1613 have minor differences with respect to the use of internal standards (1613 requires more), reporting of estimated maximum possible concentrations (EMPCs; 1613 does not report these), calibration range (1613 is twice as wide), and laboratory control samples (1613 requires one at the beginning of each instrument run). Their use may be dictated more by laboratory preference than actual performance (personal communications with Dante Bencivengo, Pacific Analytical and Todd Vilen, Triangle Laboratories). We found no evidence to indicate that either method generates biased data or would otherwise preclude data comparability among years.

TOC and percent solids determination in the SPM sample were done by Puget Sound Estuary Program protocols (EPA, 1986a) and EPA Method 160.3, respectively. Analysis of water samples was conducted according to the following EPA methods: specific conductance - EPA 120.1; TOC and DOC - EPA 415.1; and TSS - EPA 160.2.

Data Quality

Complete results of PCDD/PCDF analyses for each year are in the Appendix. Stuart Magoon of the Manchester Environmental Laboratory reviewed all of the PCDD/PCDF data for holding times, method blanks, initial and continuing calibration, internal standard recoveries, isotopic abundance ratios, and matrix spike recoveries.

Overall quality of the data was good. SPM samples collected during 1990 and 1992 were analyzed in duplicate for all seventeen 2,3,7,8-PCDDs/PCDFs to assess precision. The size of the 1994 sample was not sufficient to analyze in duplicate, and the 1993 sample was large enough to conduct duplicate analysis of 2,3,7,8-TCDD and 2,3,7,8-TCDF only. For samples analyzed in duplicate, the relative percent difference (range as a percent of the mean) was generally less than 30%. Duplicate analysis of matrix spikes also showed good precision.

For the most part, analyses of PCDDs/PCDFs were within the method quality control limits and all data are useable as qualified. Qualifiers were included in the following instances:

- Some of the 1992 and 1994 results were below the lower calibration limit and were therefore qualified as estimates (J).
- Analysis of four PCDDs/PCDFs in 1993 did not meet Method 8290 criteria for isotopic abundance ratios. Results were qualified as estimated maximum possible concentrations (EMPCs).
- Two other analytes from the 1993 sample were below practical quantitation limits and were qualified as estimates (J).

- 2,3,4,6,7,8-HxCDF was detected in the 1993 method blank at a concentration equal to 38% of the analytical result. 2,3,7,8-TCDD was detected in the 1994 method blank at a concentration equal to 28% of the analytical result. The presence of these analytes were therefore considered results of laboratory contamination and not native to the samples (UJ).
- The internal standard for OCDD in 1994 was below the acceptable recovery limit (J).

Results

General Water Quality Characteristics of the Columbia River at Northport

Table 2 shows results of water sample analysis. Specific conductance and temperature showed little variation within or among years. The TOC levels in 1990 were about double that in following years, and pH varied substantially among years. While we cannot explain the relatively high TOC in 1990, differences in sampling practices may explain the variations in pH. Higher pH values were obtained during 1990 and 1994 when samples were taken *in situ*, and are more consistent with historical records of pH in the upper Columbia River (USGS, 1980-1991). When samples were taken from the centrifuge intake manifold, as in 1992 and 1993, the pH values were much lower. We conclude that the pressure differential caused by pumping the sample approximately 200 feet horizontally and 20 feet vertically caused changes in dissolved gas concentrations which in turn resulted in lower pH.

Table 2. Summary of Columbia River Water Quality at Northport During SPM Sample Collection (mean ± SD).

	1990 (n=8)	1992 (n=7)	1993 (n=6)	1994 (n=6)
Specific Conductance (µmhos/cm)	148 ± 2	135 ± 2	150 ± 3	140 ± 4
Total Organic Carbon (mg/L)	3.3 ± 0.1	1.3 ± 0	1.9 ± 0.2	1.4 ± 0.1
Dissolved Organic Carbon (mg/L)	na	1.1 ± 0.1	1.9 ± 0.6	1.3 ± 0.1
Total Suspended Solids (mg/L)	2.0 ± 0.5	1.4 ± 0.5	2.2 ± 0.4	0.8 J
pH (s.u., median value)	8.2	7.2	6.9	8.3
Temperature (C)	13.0 ± 0.6	14.7 ± 1.0	14.5 ± 1.0	15.1 ± 0.5

na=not analyzed
J=estimated value

TSS concentrations obtained during 1994 were lower than normal; four of the six water samples had concentrations below the laboratory's reporting limit of 1 mg/L. The 0.8 mg/L value shown in Table 2 was calculated by dividing the dry weight of the SPM sample by the volume of water centrifuged. Since reliable TSS values were not obtained from laboratory analysis, there is no way to assess the centrifuge's

SPM-removal efficiency, which had been estimated to range from 60-100% during previous years of monitoring (Table 1).

As a result of low TSS concentrations in 1994, the estimated daily load of suspended solids in the Columbia River at Northport was also much lower than during previous monitoring. Estimates of daily suspended solids loads during 1990-1993 monitoring had been consistent among years, ranging from 300,000 to 320,000 Kg/day. The daily suspended solids load during the 1994 monitoring was approximately 117,000 Kg/day.

2,3,7,8-PCDD/PCDF Concentrations in Suspended Particulate Matter

PCDD/PCDF concentrations in SPM samples obtained during 1990-1994 are shown in Table 3. The samples collected during 1993 and 1994 had more organic carbon and slightly more moisture than in previous years. This may be due to the relative absence of silt-sized slag particles originating from the Cominco smelter, which had previously been observed as a major component of SPM (Bortleson *et al.*, 1994). The odor and texture of the 1993 and 1994 samples suggest that it contained a greater proportion of biological material than previous samples.

Six PCDDs/PCDFs were detected in the 1990 SPM sample, three in 1992, fourteen in 1993, and nine in 1994. The small number of PCDDs/PCDFs found in the 1992 sample was probably due to relatively high detection limits. Of the 17 PCDD/PCDF congeners analyzed, all but one –1,2,3,7,8,9 HxCDF – were detected in at least one of the SPM samples. OCDD, 1,2,3,4,6,7,8-HpCDD, and 2,3,7,8-TCDF were generally found at the highest concentrations and were the only congeners detected all four years. Other PCDDs/PCDFs were detected at low concentrations (less than 5 pg/g) in all but two instances.

2,3,7,8-TCDF was detected in the 1994 SPM sample at 1.1 pg/g. This represents a 75% decrease from the 1993 concentration and is two orders of magnitude lower than in 1990. 2,3,7,8-TCDD was not detected in SPM during 1990, 1992, or 1993 at quantitation limits ranging from 0.4 to 1.7 pg/g. 2,3,7,8-TCDD was detected in the 1994 SPM sample at 1.8 pg/g, but as mentioned previously, its presence was considered a result of laboratory contamination.

Figure 3 illustrates the variation in congener composition from year to year. OCDD, and to a lesser extent 1,2,3,4,6,7,8-HpCDD, became the predominant congeners during 1992-1994 after 2,3,7,8-TCDF concentrations dropped precipitously from the 1990 level. However, concentrations of these PCDD congeners also dropped dramatically – 20 to 30-fold – between 1993 and 1994.

Table 3. Concentrations of 2,3,7,8-PCDDs/PCDFs in Columbia River SPM at Northport, 1990-1994 (mean ± range; pg/g [parts per trillion], dry weight basis).

Compound	TEF	1990 (n=2)	1992 (n=2)	1993 (n=2)*	1994 (n=1)
% Moisture		73.8	78.8	83.6	84.0
% TOC		7.6	2.2	10.4	12.0
2,3,7,8-TCDD	1	U(0.8-0.9)	U(1.5-1.7)	U(0.4-0.7)	1.8 UJ
1,2,3,7,8-PeCDD	0.5	U(0.5)	U(1.0-1.5)	**0.5** EMPC	**2.3 J**
1,2,3,4,7,8-HxCDD	0.1	U(0.9-1.3)	U(2.5-2.6)	**1.5**	U(0.6)
1,2,3,6,7,8-HxCDD	0.1	U(0.7-1.0)	U(3.2)	**3.0**	U(1.2)
1,2,3,7,8,9-HxCDD	0.1	U(0.8-1.1)	U(2.7)	**3.2 J**	U(0.5)
1,2,3,4,6,7,8-HpCDD	0.01	**11 ± 1**	**31 ± 6 J**	**74**	**3.5 J**
OCDD	0.001	**79 ± 9**	**214 ± 30**	**482**	**16.5 J**
2,3,7,8-TCDF	0.1	**99 ± 22**	**6.2 ± 0.4 J**	**4.4 ± 0.2**	**1.1 J**
1,2,3,7,8-PeCDF	0.05	**1.0 ± 0.1**	U(1.7-1.8)	**0.7 J**	**1.2 J**
2,3,4,7,8-PeCDF	0.5	**1.4 ±0.2**	U(2.5-2.6)	**0.6 EMPC**	U(0.3)
1,2,3,4,7,8-HxCDF	0.1	U(0.2-0.4)	U(2.2-2.6)	**1.3 EMPC**	U(0.5)
1,2,3,6,7,8-HxCDF	0.1	U(0.2-0.3)	U(2.4-2.8)	**0.6**	**1.0 J**
2,3,4,6,7,8-HxCDF	0.1	U(0.2-0.3)	U(2.2-2.5)	1.7 UJ	**0.4 J**
1,2,3,7,8,9-HxCDF	0.1	U(0.3-0.4)	U(2.2-2.6)	U(0.5)	U(0.4)
1,2,3,4,6,7,8-HpCDF	0.01	U(1.1-1.7)	U(1.2-10)	**8.5**	**1.2 J**
1,2,3,4,7,8,9-HpCDF	0.01	U(0.5-0.6)	U(1.0-10)	**0.9 EMPC**	U(0.7)
OCDF	0.001	**4.6 ± 0.7**	U(11-14)	**23.6**	U(1.7)
	TEQ=	10.8	1.1	3.3	1.5

detected PCDDs/PCDFs in **bold** print
TEF=Toxicity Equivalency Factor
*duplicate analysis of 2,3,7,8-TCDD and 2,3,7,8-TCDF only
U=Undetected at or above range of quantitation limits in parentheses
UJ=analyte detected in method blank
EMPC=Estimated Maximum Possible Concentration
J=estimated concentration due to result below calibration range, below practical quantitation limit, or associated internal standard below recovery limit.
TEQ=Toxicity Equivalent, based on detected values

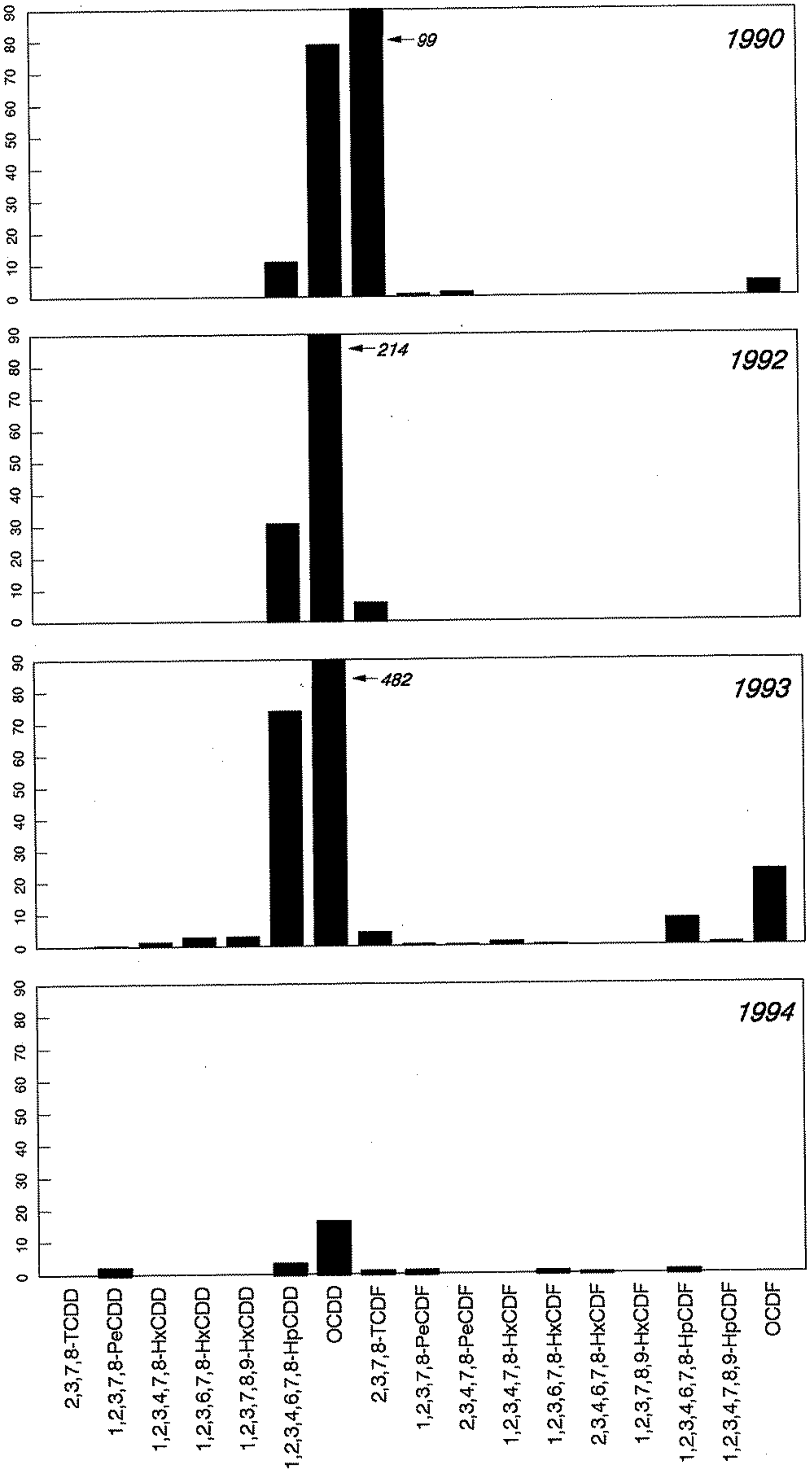

Figure 3. PCDD/PCDF Concentrations in Columbia River SPM at Northport, 1990-1994 (pg/g, dry weight basis).

Toxicity Equivalence

All seventeen PCDDs/PCDFs congeners with chlorine atoms in the 2, 3, 7, and 8 positions (*e.g.* 1,2,3,4,6,7,8-HpCDD) are considered to have a high level of toxicity, with 2,3,7,8-TCDD being the most toxic. An individual congener's toxicity relative to 2,3,7,8-TCDD, or toxicity equivalency factor (TEF), may be used to convert its concentration into an equivalent concentration of 2,3,7,8-TCDD, referred to as a toxicity equivalent (TEQ). In general, congeners with greater chlorine substitution (*e.g.* OCDF) have lower TEFs than less chlorinated congeners (*e.g.* 2,3,7,8-TCDF) (Table 3). Because PCDDs/PCDFs are generally found in mixtures, the TEQ concentration of a sample is expressed as the sum of TEQs for each congener. TEQs have no regulatory basis in water, but may be used to estimate risks associated with exposure to 2,3,7,8-PCDD/PCDF mixtures (Barnes *et al.*, 1989).

TEQs, calculated from PCDDs/PCDFs detected in SPM, declined by an order of magnitude from 1990 to 1992 (10.8 vs. 1.1 pg/g). From 1992 to 1993, TEQ increased three-fold to 3.3 pg/g, then dropped to 1.5 pg/g in 1994. At least some of the dramatic decline in TEQ from 1990 to 1992 was due to the much higher detection limits in 1992 as compared to 1990 (and other years). For instance, if non-detected congeners were used to derive the TEQ using one-half the mean detection limits, TEQs for 1990 and 1992 would be 12.3 and 6.7 pg/g, respectively (4.1 pg/g for 1993 and 2.9 pg/g for 1994).

Perhaps more interesting is the change in compounds responsible for the toxicity, as shown in Figure 4. In 1990 and 1992, the congener 2,3,7,8-TCDF accounted for 92% and 56% of the TEQ, respectively. In 1993, no single PCDD/PCDF accounted for the majority of TEQ (1,2,3,4,6,7,8-HpCDD had the greatest contribution at 22%). 1,2,3,7,8-PeCDD accounted for 75% of the TEQ in the 1994 sample. Again, these changes reflect shifting patterns in the composition and concentrations of PCDDs/PCDFs bound to suspended particulate matter.

2,3,7,8-PCDD/PCDF Loads in the Columbia River at Northport

Daily loads of SPM-bound PCDDs/PCDFs in the Columbia River at Northport were calculated for each year as the product of their concentration in SPM, TSS concentration in water, and the daily Columbia River discharge (Table 4). The PCDD/PCDF loads in Table 4 probably underestimate actual loads because they do not account for dissolved PCDDs/PCDFs. The loads detected in 1994 were generally one-to-two orders of magnitude lower than previous years due to both lower congener concentrations and suspended solids loads in the river. The 2,3,7,8-TCDF load decreased 300-fold between 1990 and 1994.

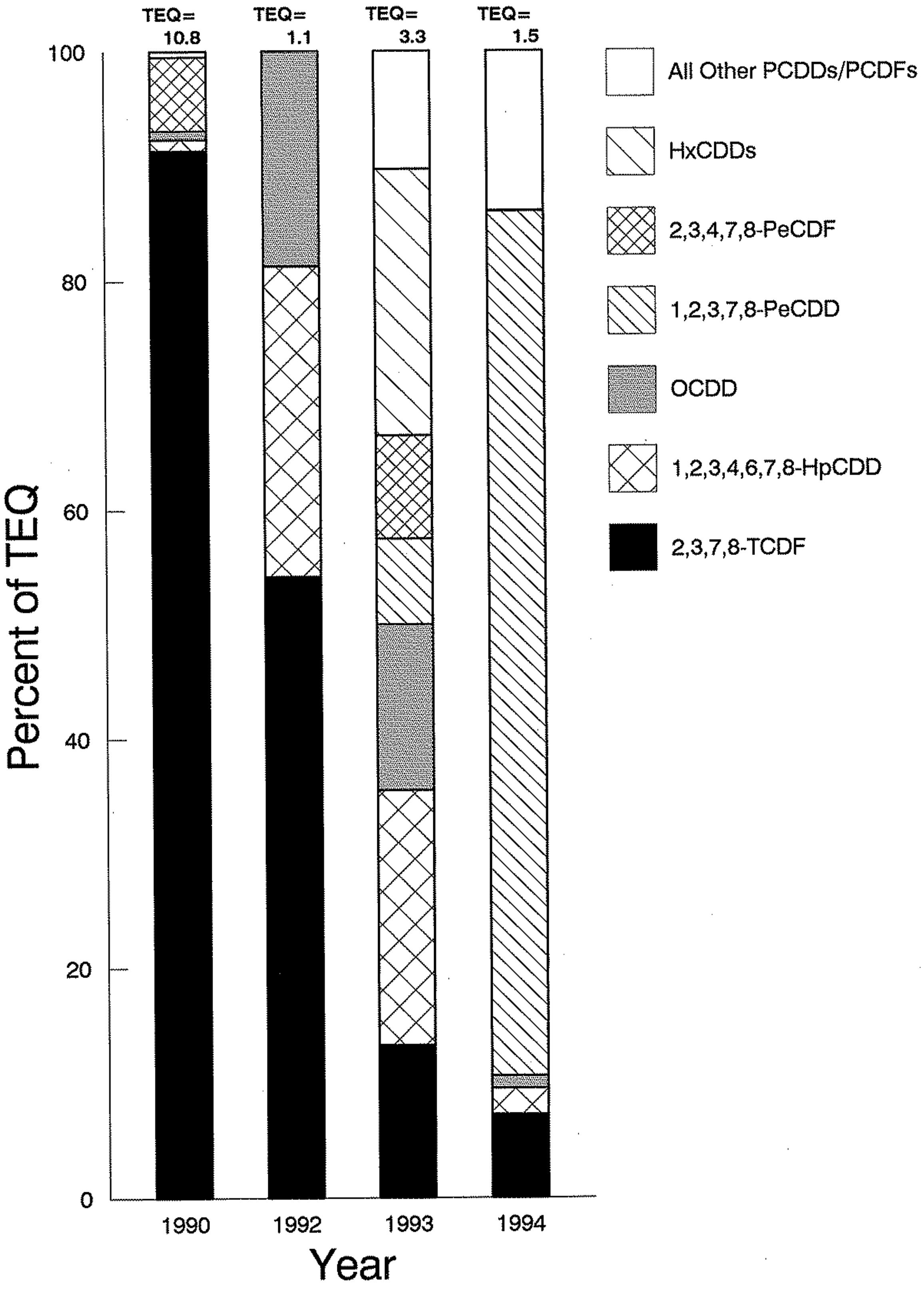

Figure 4. Contribution of PCDD/PCDF Congeners to TEQ

Table 4. Loads of SPM-Bound 2,3,7,8-PCDDs/PCDFs in the Columbia River at Northport, 1990-1994 (mg/day).

Compound	1990	1992	1993	1994
2,3,7,8-TCDD	nd	nd	nd	nd
1,2,3,7,8-PeCDD	nd	nd	0.2	0.3
1,2,3,4,7,8-HxCDD	nd	nd	0.5	nd
1,2,3,6,7,8-HxCDD	nd	nd	0.9	nd
1,2,3,7,8,9-HxCDD	nd	nd	1.0	nd
1,2,3,4,6,7,8-HpCDD	3.3	10	23	0.4
OCDD	24	69	150	1.9
2,3,7,8-TCDF	30	2.0	1.4	0.1
1,2,3,7,8-PeCDF	0.3	nd	0.2	0.1
2,3,4,7,8-PeCDF	0.4	nd	0.2	nd
1,2,3,4,7,8-HxCDF	nd	nd	0.4	nd
1,2,3,6,7,8-HxCDF	nd	nd	0.2	0.1
2,3,4,6,7,8-HxCDF	nd	nd	nd	0.05
1,2,3,7,8,9-HxCDF	nd	nd	nd	nd
1,2,3,4,6,7,8-HpCDF	nd	nd	2.6	0.1
1,2,3,4,7,8,9-HpCDF	nd	nd	0.3	nd
OCDF	1.4	nd	7.3	nd
TEQ=	3.3	0.4	1.0	0.2

nd=not detected
TEQ=Toxicity Equivalent

As mentioned previously, the daily suspended solids load at Northport during 1994 monitoring was approximately 40% of the loads measured 1990-1993. This probably also accounted somewhat for the low 1994 TEQ load. A suspended solids load in 1994 similar to those during 1990-1993 would have yielded a TEQ load of approximately 0.5 mg/day during 1994. The relatively low TEQ load in 1992 is due in part to the higher PCDD/PCDF detection limits reported for the 1992 sample.

Estimation of 2,3,7,8-TCDD Loads

2,3,7,8-TCDD is of special interest because, in addition to being the most toxic congener, it is the only PCDD/PCDF for which water quality criteria have been promulgated (EPA, 1986b). EPA has established a total maximum daily load (TMDL) for 2,3,7,8-TCDD to limit its discharge to the Columbia River basin (EPA, 1991). Based on the harmonic mean flow of the Columbia River at the international border (1.78×10^8 L/day), EPA has determined that the loading capacity or watershed target for the Columbia River north of the border is 2.3 mg/day to avoid surpassing the water quality criterion of 0.013 pg/L (parts per quadrillion).

Since 2,3,7,8-TCDD was not detected in the SPM sample during the four years of monitoring, *maximum possible* SPM or solid phase loads were estimated by substituting quantitation limits for actual concentrations (Table 5). The highest quantitation limits were used for years where ranges are reported (1990, 1992, and 1993). Solid phase loads for all years except 1992 were approximately 0.2 - 0.3 mg/day. The higher load estimate for 1992 was due to a high quantitation limit for 2,3,7,8-TCDD.

Table 5. Estimated *Maximum Possible* 2,3,7,8-TCDD Loads and Whole Water Concentrations in the Columbia River at Northport, 1990-1994.

	1990	1992	1993	1994
Solid Phase 2,3,7,8-TCDD Load (mg/day)	0.3[a]	0.5[a]	0.2[a]	0.2[a]
Dissolved Phase 2,3,7,8-TCDD Load (mg/day)	0.9[b]	2.3[c]	2.3[d]	1.1[b]
Total 2,3,7,8-TCDD Load (mg/day)	1.2	2.8	2.5	1.3
Whole Water Concentration (pg/L)	0.008	0.012	0.018	0.009

[a] Based on quantitation limits, this report

[b] Estimated using K_{oc} of 2.00×10^6

[c] Based on quantitation limits, Bortleson *et al.* (1994)

[d] Based on quantitation limits, Serdar *et al.* (1994)

We also estimated dissolved phase loads for 2,3,7,8-TCDD (Table 5) because a potentially significant fraction is likely dissolved due to the low concentration of suspended solids in the water column. The TMDL and water quality criterion for 2,3,7,8-TCDD are based on whole water, not simply the solid phase.

Maximum possible dissolved 2,3,7,8-TCDD loads for 1992 and 1993 were based on results of sampling at Northport conducted by the U.S.Geological Survey (USGS) during 1992 (Bortleson *et al.*, 1994) and Ecology during 1993 (Serdar *et al.*, 1994). Columns packed with XAD resin were used to concentrate dissolved 2,3,7,8-TCDD in clarified river water. Neither the USGS nor Ecology detected dissolved 2,3,7,8-TCDD at quantitation limits of 0.01 pg/L and 0.016 pg/L, respectively. However, the quantitation limits are useful in estimating *maximum possible* loads, as done for SPM. *Maximum possible* dissolved loads for both 1992 and 1993 were 2.3 mg/day, yielding total (solid + dissolved loads) of 2.8 and 2.5 mg/day, respectively.

Since dissolved phase monitoring was not conducted during 1990 and 1994, we calculated theoretical dissolved loads using the 2,3,7,8-TCDD sorption partition coefficient (K_{oc}). The K_{oc} is a value derived from a hydrophobic compound's equilibrium distribution between sediment and water and normalized to organic carbon. The fraction of 2,3,7,8-TCDD in the dissolved phase can be calculated from the equation:

$$\text{Fraction of dissolved 2,3,7,8-TCDD} = \{1 + (K_{oc} \text{ x Fraction OC in SPM x Fraction SPM in water})\}^{-1}$$

K_{oc} values are obtained experimentally, by observations of partitioning between water and solid phases with known organic carbon content, or they may be calculated using other properties of a compound, such as its relative solubility in octanol and water. Mackay *et al.* (1992) have compiled thirty-three 2,3,7,8-TCDD K_{oc} values from the literature, derived both empirically and theoretically, and ranging from 1.15×10^3 to 3.89×10^7 with a median value of 2.00×10^6. We used the median K_{oc} (2.00×10^6) to calculate dissolved loads.

Total *maximum possible* water column concentrations and daily loads in 1990 and 1994 were about one-half of EPA's water quality criterion and watershed target for the Columbia River north of the international border. In 1992 and 1993, daily *maximum possible* 2,3,7,8-TCDD loads were slightly higher than the target of 2.3 mg/day, although the concentration in water during 1993 was slightly below the criterion of 0.013 pg/L.

Discussion

Pollution Abatement by Celgar

The primary objective of this four-year monitoring program was to document the effects of pollution controls instituted by the Celgar pulp mill. By the time we collected our initial SPM sample in 1990 Celgar had already taken some measures to reduce their discharge of PCDDs/PCDFs, such as terminating the use of defoamers which contained PCDD/PCDF precursors and beginning to substitute chlorine dioxide for chlorine in the bleaching process. Because some of these early process changes were implemented before reliable data were available on PCDDs/PCDFs in Celgar's effluent, it is unclear to what extent they had reduced concentrations prior to 1990.

Figure 5 shows 2,3,7,8-TCDF in Celgar effluent from May 1989 through October 1994. These data, collected and reported by Celgar, indicate that the largest reductions in 2,3,7,8-TCDF discharges occurred during 1991. Celgar (1992) cites these improvements as a result of further refinements in production processes, such as increased chlorine dioxide substitution for chlorine. By the time the modernized mill and secondary treatment system were fully operational in mid-1993, 2,3,7,8-TCDF concentrations in Celgar effluent were no longer detectable at a limit of 2 pg/L (Celgar Pulp Co., 1993 & 1994; Jim McLaren, Environmental Manager, Celgar Pulp Co., personal and written communications).

Decreased concentrations and loads of 2,3,7,8-TCDF in SPM correspond well with reductions achieved by Celgar from 1990 to 1994. Until 1993, 2,3,7,8-TCDF was the most significant congener downstream of Celgar due to its contribution to overall toxicity and consistent detectability – usually at the highest concentrations. 2,3,7,8-TCDD, although more important from a toxicity and regulatory standpoint, was detected in only 15% of effluent samples analyzed by Celgar between May 1989 and October 1994.

Celgar did not begin monitoring for the full suite of 2,3,7,8-PCDDs/PCDFs until September 1992. During the following two years, they reported results on 16 effluent samples, in which OCDD was the most frequently detected (38% of samples), followed by 2,3,7,8-TCDF (19%), 2,3,7,8-TCDD (12%), and OCDF (6%). Four effluent samples were also analyzed between March 1995 and March 1996. The only PCDDs/PCDFs detected were OCDD (in all four samples) and 1,2,3,4,6,7,8-HpCDD (two samples).

There are no documented sources of PCDDs/PCDFs to the study area aside from the Celgar pulp mill. Possible point sources, although probably very small, may include several wood preservation operations and municipal wastewater treatment plants in the Pend Oreille drainage (BCI, 1991-Draft) and along the Columbia River between the border and Hugh Keenlyside Dam (Butcher, 1992; MacDonald Environmental Sciences

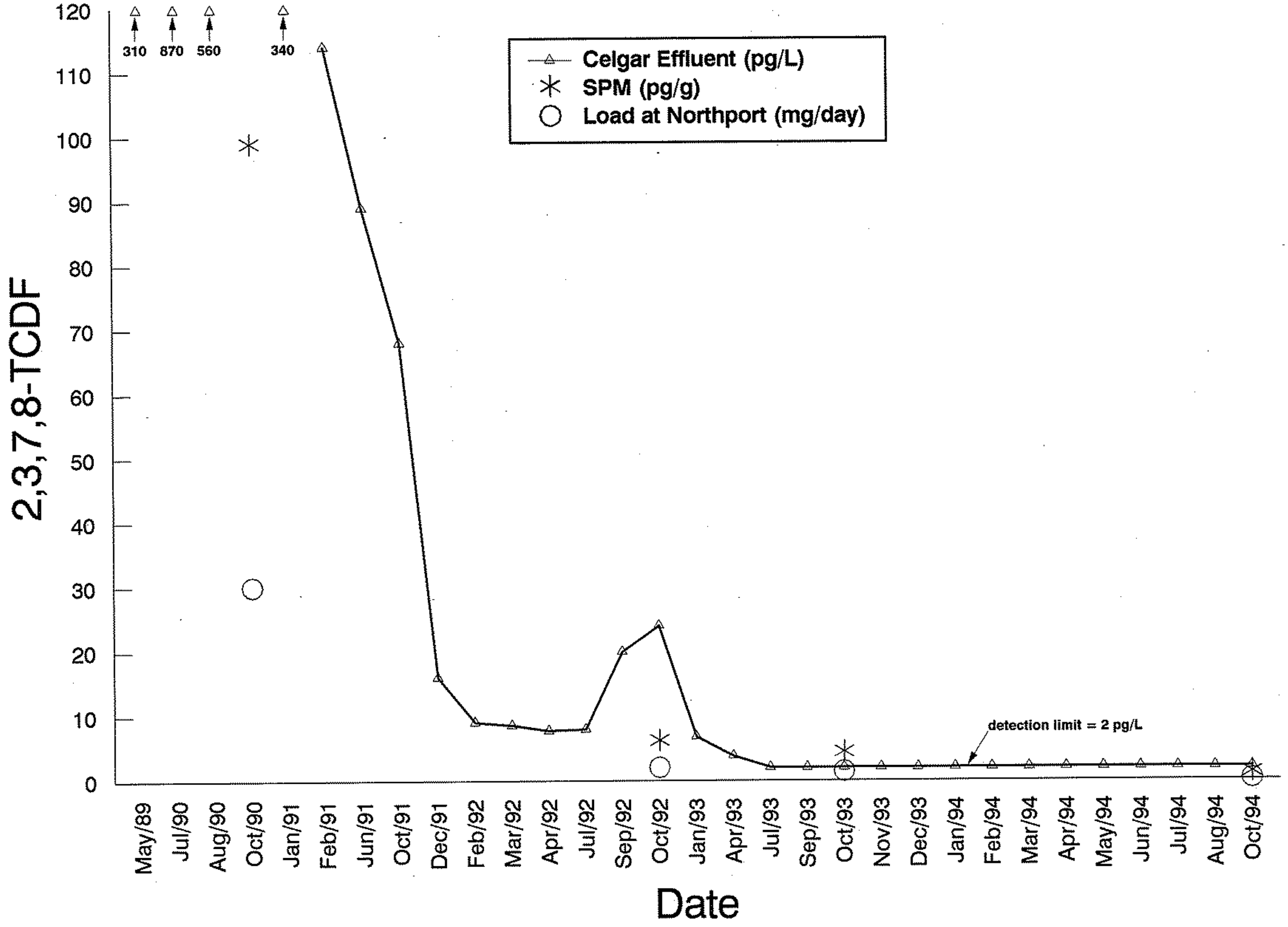

Figure 5. Concentrations of 2,3,7,8-TCDF in Celgar Effluent Plotted with Concentrations in SPM and Loads at Northport

Ltd., 1997-Draft). Celgar is probably not a continuing source of measurable PCDDs/PCDFs, with the possible exception of 1,2,3,4,6,7,8-HpCDD and OCDD.

Although there are no data to indicate a specific source of non-Celgar PCDDs/PCDFs detected in SPM, possibilities may include: 1) resuspension of contaminated bottom sediments between Celgar and Northport; 2) undocumented urban or industrial pollutant sources to the river; and 3) atmospheric deposition in the upper Columbia River basin. The latter possibility is supported by evidence from Arrow Lake upstream of Celgar, sediments of which had OCDD concentrations ranging from 26 to 68 pg/g and lower levels of OCDF (7.5 pg/g), but no other PCDDs/PCDFs at detection limits as low as 0.3 pg/g (Tuominen *et al.*, In Prep). Since there are no known or suspected point-sources of PCDD/PCDFs to Arrow Lake, this suggests their presence is due to atmospheric transport. This hypothesis is consistent with research showing that highly chlorinated PCDDs/PCDFs, especially OCDD, are associated with airborne particulate matter (Czuczwa *et al.*, 1984), and that these chemicals are widely distributed with aquatic sediments as the ultimate sinks (Czuczwa and Hites, 1986).

Comparison to Other Surveys

Environment Canada - 1990-1991

Environment Canada monitored PCDDs/PCDFs in SPM from three Columbia River sites -- one above Celgar and two below -- during October 1990, March 1991, and June 1991 (Table 6)(Tuominen *et al.*, 1994; Tuominen *et al.*, In Prep). Their initial monitoring at Waneta, just upstream of the Pend Oreille River, coincided with our original SPM collection. They also collected SPM from a site approximately 2.4 miles downstream of Celgar several days later (October 14-17, 1990). Unfortunately, low resolution GC/MS was employed for this initial survey, resulting in high detection limits (14-800 pg/g) and only one compound being detected: 2,3,7,8-TCDF at 1,900 pg/g (2.4 RM below Celgar) and at a mean concentration of 878 pg/g (Waneta). The difference in Environment Canada's 2,3,7,8-TCDF results at Celgar and Waneta may be due in part to deposition of sediments in this 30-mile river reach, but more likely reflects dilution from the Kootenay River (assuming no additional 2,3,7,8-TCDF inputs) which increases the Columbia River flow by an annual average of 75% between Celgar and Waneta (Butcher, 1992).

Subsequent monitoring of SPM by Environment Canada in 1991 showed an order of magnitude decrease in 2,3,7,8-TCDF and TEQ concentrations at both the Celgar and Waneta sites. This agrees well with PCDD/PCDF reductions reported by Celgar. The results also show that there is no significant contribution of PCDDs/PCDFs from the Columbia River above Celgar, represented by the Arrow Lake data.

Table 6. Summary of 2,3,7,8-TCDF and TEQ Concentrations in Columbia River SPM Analyzed by Environment Canada and Ecology, 1990-1991 (pg/g, dry weight basis).

Site	October 1990	March 1991	June 1991
Arrow Lake			
2,3,7,8-TCDF	U(4-11)	U(2)	U(1-1.4)
TEQ	0*	0.05*	0.04*
2.4 RM below Celgar			
2,3,7,8-TCDF	1,900	98	107*
TEQ	190	9.9	10.7*
Waneta			
2,3,7,8-TCDF	878*	≤0.4*	≤112*
TEQ	87.8*	0.04*	11.2*
Northport (Ecology)			
2,3,7,8-TCDF	99*	-	-
TEQ	10.8*	-	-

U=Undetected at or above range of quantitation limits in parentheses
TEQ=Toxicity Equivalent, based on detected values
*mean of two results

Although the Environment Canada data generally support results and conclusions from our early study (*e.g.* 2,3,7,8-TCDF as the major contributor to TEQ, substantial reductions in 2,3,7,8-TCDF discharges after 1990), the disagreement in 2,3,7,8-TCDF levels during concurrent monitoring by Ecology and Environment Canada is a confounding item. Tuominen *et al.* (1994) concluded that lower 2,3,7,8-TCDF concentrations obtained by Ecology are a result of incomplete mixing of the Pend Oreille River at Northport. Since the Pend Oreille contributes 27% of the mean annual Columbia River flow at the Canada border, 2,3,7,8-TCDF concentrations in our SPM sample should have been lowered accordingly when compared to Waneta, assuming identical TSS concentrations in the Pend Oreille, no additional 2,3,7,8-TCDF inputs, and complete mixing of the Pend Oreille River at Northport. However, if the Pend Oreille River is not completely mixed with the Columbia River at Northport, it stands to reason that 2,3,7,8-TCDF concentrations would be lowered to a greater degree since the Pend Oreille enters the Columbia at the left bank, the same side from which our samples were collected.

There is no direct evidence that the Pend Oreille and Columbia rivers are not completely mixed at Northport. The 10-mile reach between the confluence and Northport is free-flowing and rapid, often shallow and with a number of torturous bends, all suggesting good if not complete mixing. However, we have not found any empirical data on mixing and the results presented here are not adequate to assess the degree to which these rivers are mixed.

U.S. Geological Survey - 1992

USGS conducted a large-scale survey of sediment quality in Lake Roosevelt during 1992, results of which are reported in Bortleson *et al.* (1994). A portion of this survey involved sampling SPM concurrent with Ecology. Results of their 2,3,7,8-PCDD/PCDF analysis are shown in Table 7.

Table 7. Concentrations 2,3,7,8-PCDDs/PCDFs in Columbia River SPM at Northport Analyzed by the U.S. Geological Survey and Ecology During 1992 (pg/g, dry weight basis).

Compound	USGS-DWIS	USGS-SDPS	Ecology
2,3,7,8-TCDD	U(3.6)	U(3.7)	U(1.5-1.7)
1,2,3,7,8-PeCDD	U(4.4)	U(3.7)	U(1.0-1.5)
1,2,3,4,7,8-HxCDD	U(2.5)	U(4.9)	U(2.5-2.6)
1,2,3,6,7,8-HxCDD	U(8.6)	U(6.3)	U(3.2)
1,2,3,7,8,9-HxCDD	U(3.4)	U(5.4)	U(2.7)
1,2,3,4,6,7,8-HpCDD	**210**	U(37)	**31***
OCDD	**1,300**	**240**	**214***
2,3,7,8-TCDF	U(7.4)	U(9.4)	**6.2***
1,2,3,7,8-PeCDF	U(4.6)	U(3.3)	U(1.7-1.8)
2,3,4,7,8-PeCDF	U(6.7)	U(4.5)	U(2.5-2.6)
1,2,3,4,7,8-HxCDF	U(7.4)	U(5.8)	U(2.2-2.6)
1,2,3,6,7,8-HxCDF	U(4.4)	U(6.3)	U(2.4-2.8)
2,3,4,6,7,8-HxCDF	U(8.0)	U(5.8)	U(2.2-2.5)
1,2,3,7,8,9-HxCDF	U(1.7)	U(6.3)	U(2.2-2.6)
1,2,3,4,6,7,8-HpCDF	**130**	U(18)	U(1.2-10)
1,2,3,4,7,8,9-HpCDF	U(6.1)	U(3.0)	U(1.0-10)
OCDF	**230**	**130**	U(11-14)
TEQ=	4.9	0.4	1.1

detected PCDDs/PCDFs in **bold** print
DWIS=Depth-Width Integrated Sample
SDPS=Short-Duration Point-Source Sample
U=Undetected at or above range of quantitation limits in parentheses
TEQ=Toxicity Equivalent, based on detected values
*mean of two results

Two SPM samples were analyzed for the USGS survey: a depth-width integrated sample (DWIS) collected at Northport during an 8.5-hour period on September 30, and sub-sample of our SPM which was collected during the same 8.5-hour period and designated as a short-duration point source (SDPS) sample. The purpose of this sampling scheme was to determine if PCDD/PCDF data obtained from a point source (*i.e.* from the left bank) were representative of PCDD/PCDF concentrations in the river cross-section.

The USGS samples had much higher concentrations of hepta- and octa-chlorinated congeners than ours, although the OCDD concentration in the SDPS sample agreed well with ours. The DWIS sample also had PCDD/PCDF concentrations at least 75% higher than SPM samples collected from the left bank, with the exception of 2,3,7,8-TCDF. It is possible that elevated PCDD/PCDF concentrations in the DWIS sample were due to Celgar's effluent, and that SPM samples from the left bank were sufficiently diluted by the Pend Oreille River (assuming incomplete mixing) to explain the difference. If this were the case, however, then detectable concentrations (*i.e.* ≥ 7.4 pg/g) of 2,3,7,8-TCDF would be expected in the DWIS since Celgar is probably the only upstream source of measurable 2,3,7,8-TCDF.

There is no clear explanation for the differences in PCDD/PCDF concentrations among these samples. Unfortunately, the USGS data do not support any conclusion about the degree of river mixing and the representativeness of point source data. If anything, these data underscore the high degree of variability associated with monitoring extremely low concentrations of contaminants in a large river.

Ecology Survey of Snake River SPM - 1996

EPA's TMDL for 2,3,7,8-TCDD in the Columbia River specifies three sub-basins for loading targets: the Columbia River above the international boundary, described in the present report; the Willamette River in Oregon; and the Snake River. These watersheds were selected for loading targets because each has a bleached kraft pulp mill which has been responsible for the majority of PCDD/PCDD loading in each basin.

Analysis of pulp mill effluents during a 1988 nationwide survey found that the Potlatch mill loaded 11.0 mg 2,3,7,8-TCDD/day to the Snake River (EPA, 1988 & 1990). The watershed target for the Snake River is 1.18 mg 2,3,7,8-TCDD/day at the mouth, which includes a waste load allocation of 0.39 mg 2,3,7,8-TCDD/day for the Potlatch mill. In addition, the average 2,3,7,8-TCDF load from Potlatch was estimated to be 50 mg/day.

Like Celgar, Potlatch initiated a number of changes, beginning around 1989, to reduce its production and discharge of PCDDs/PCDFs (EPA, 1990; Michael Letourneau, EPA, written communication). In 1996, we analyzed PCDDs/PCDFs in SPM collected from the mouth of the Snake River to determine the load of 2,3,7,8-TCDD and other PCDDs/PCDFs (Serdar and Cubbage, 1997). Five congeners were detected, generally at low concentrations (Table 8). Loads of 2,3,7,8-TCDD and 2,3,7,8-TCDF in 1996 represented reductions of 98% and 99%, respectively, when compared to loads measured during the 1988 survey of Potlatch effluent. Perhaps more notable is that the congener profile of PCDDs/PCDFs detected in the Snake River during 1996 shows many similarities to post-1990 Northport SPM – OCDD and 1,2,3,4,6,7,8-HpCDD as the major congeners, low concentrations of 2,3,7,8-TCDF – possibly signaling a new PCDD/PCDF fingerprint from modernized bleached kraft pulp mills.

Table 8. Concentrations of 2,3,7,8-PCDDs/PCDFs in Snake River SPM During 1996 and Columbia River SPM, 1992-1994 (range; pg/g, dry weight basis).

Compound	Snake R. at Mouth (n=3)	Columbia R. at Northport (n=4)*
2,3,7,8-TCDD	U (0.24-0.32)	U(0.4-1.8)
1,2,3,7,8-PeCDD	U (0.23-0.29)	U(1.0) - **2.3**
1,2,3,4,7,8-HxCDD	U (0.34-0.40)	U(0.6) - **1.5**
1,2,3,6,7,8-HxCDD	U (1.1-1.3)	U(1.2) - **3.0**
1,2,3,7,8,9-HxCDD	U (1.1-1.2)	U(0.5) - **3.2**
1,2,3,4,6,7,8-HpCDD	**22 - 27**	**3.5 - 74**
OCDD	**190 - 230**	**16.5 - 482**
2,3,7,8-TCDF	**2.0**	**1.1 - 6.2**
1,2,3,7,8-PeCDF	U (0.19-0.23)	**0.7 - 1.2**
2,3,4,7,8-PeCDF	U (0.26-0.32)	U(0.3) - **0.6**
1,2,3,4,7,8-HxCDF	U (0.42-0.48)	U(0.5) - **1.3**
1,2,3,6,7,8-HxCDF	U (0.20-0.29)	**0.6 - 1.0**
2,3,4,6,7,8-HxCDF	U (0.53-0.58)	**0.4**
1,2,3,7,8,9-HxCDF	U (0.061-0.12)	U(0.4-2.6)
1,2,3,4,6,7,8-HpCDF	**4.4 - 5.2 J**	**1.2 - 8.5**
1,2,3,4,7,8,9-HpCDF	U (0.29-0.32)	U(0.7) - **0.9**
OCDF	**15 - 18**	U(1.7) - **23.6**
TEQ=	0.7	1.1 - 3.3

*n=5 for 2,3,7,8-TCDD and 2,3,7,8-TCDF

detected PCDDs/PCDFs in **bold** print

U=Undetected at or above range of quantitation limits in parentheses

J=estimated concentration

TEQ=Toxicity Equivalent, based on detected values

Conclusions

There was a significant decrease in the concentration of 2,3,7,8-TCDF bound to Columbia River SPM between 1990 and 1994. The decline in 2,3,7,8-TCDF concentrations was almost certainly due to pollution abatement efforts and process modifications at the Celgar pulp mill in Castlegar, B.C.

Congener profiles in SPM demonstrate substantial variation over time and do not appear to be directly related to discharges from Celgar. Aside from 2,3,7,8-TCDF, there was no clear trend in concentrations of SPM-bound PCDDs/PCDFs from 1990 to 1994.

The daily 2,3,7,8-TCDD load at Northport was well below the EPA TMDL watershed target (2.3 mg/day) during 1990 and 1994 monitoring. Theoretically, 2,3,7,8-TCDD loads may have slightly exceeded the target in 1992 and 1993, but this scenario was based on estimated *maximum possible* loads.

Recommendations

We recommend an additional round of SPM collection at Northport during low-flow conditions to:

- Determine if 2,3,7,8-TCDF concentrations remain low
- Find out if the congener profile has stabilized
- Check for the presence of 2,3,7,8-TCDD

Since the bulk of the PCDD/PCDF loads are apparently in the dissolved phase, analysis of the dissolved fraction should accompany future SPM analysis.

We also recommend collecting SPM during high-flow conditions to assess whether sediment resuspension contributes significant PCDD/PCDF loads to the upper Columbia River and Lake Roosevelt.

Any additional sampling should be accompanied by testing, using dye or other means, to determine the degree to which the Columbia and Pend Oreille rivers are mixed at Northport.

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Appendices

PCDD & PCDF
EPA METHOD 8290

Sample ID: 418250	Date Received: 10/25/90	ICAL ID: 18290CAL
Lab ID: 10065-001-SA	Date Extracted: 10/31/90	QC Lot: LC1031S
Matrix: Sediment	Sample Amount: 10.07 g	Units: pg/g

Compound	Conc.	D.L.	Ratio	S/N Ratio	Qualifier
2,3,7,8-TCDD	ND	0.77			
Total TCDD	ND	0.77			
1,2,3,7,8-PeCDD	ND	0.51			
Total PeCDD	ND	0.51			
1,2,3,4,7,8-HxCDD	ND	0.87			
1,2,3,6,7,8-HxCDD	ND	0.68			
1,2,3,7,8,9-HxCDD	ND	0.75			
Total HxCDD	ND	2.0			
1,2,3,4,6,7,8-HpCDD	9.7		1.04	>10:1	
Total HpCDD	21		1.04	>10:1	
OCDD	65		0.88	>10:1	B
2,3,7,8-TCDF	88		0.80	>10:1	F
Total TCDF	170		0.77	>10:1	
1,2,3,7,8-PeCDF	1.0		1.44	4:1	
2,3,4,7,8-PeCDF	1.2		1.38	5:1	
Total PeCDF	3.8		1.45	5:1	
1,2,3,4,7,8-HxCDF	ND	0.25			
1,2,3,6,7,8-HxCDF	ND	0.19			
2,3,4,6,7,8-HxCDF	ND	0.22			
1,2,3,7,8,9-HxCDF	ND	0.29			
Total HxCDF	ND	0.29			
1,2,3,4,6,7,8-HpCDF	ND	1.1			
1,2,3,4,7,8,9-HpCDF	ND	0.52			
Total HpCDF	2.8		1.10	9:1	
OCDF	3.9		0.91	>10:1	

Analyst:

Reviewer:

PCDD & PCDF
EPA METHOD 8290

Sample ID: 418250
Lab ID: 10065-001-SA

Isotopic Recovery Results

Internal Standard:	% R	Ratio	Qualifier
^{13}C-2,3,7,8-TCDD	83	0.79	
^{13}C-1,2,3,7,8-PeCDD	94	1.57	
^{13}C-1,2,3,6,7,8-HxCDD	79	1.27	
^{13}C-1,2,3,4,6,7,8-HpCDD	71	1.05	
^{13}C-OCDD	65	0.90	
^{13}C-2,3,7,8-TCDF	78	0.80	
^{13}C-1,2,3,7,8-PeCDF	80	1.58	
^{13}C-1,2,3,4,7,8-HxCDF	67	1.27	
^{13}C-1,2,3,4,6,7,8-HpCDF	59	0.45	
Clean-up Recovery Standard:			
^{37}Cl-2,3,7,8-TCDD	77	NA	

Dates Analyzed:

DB-5: 11/08/90 DB-225: 11/12/90 SP-2331: NA

Analyst:

Reviewer: BM

PCDD & PCDF
EPA METHOD 8290

Sample ID: 418250DUP
Lab ID: 10065-001-DUP
Matrix: Sediment

Date Received: 10/25/90
Date Extracted: 10/31/90
Sample Amount: 9.99 g

ICAL ID: 18290CAL
QC Lot: LC1031S
Units: pg/g

Compound	Conc.	D.L.	Ratio	S/N Ratio	Qualifier
2,3,7,8-TCDD	ND	0.91			
Total TCDD	ND	0.91			
1,2,3,7,8-PeCDD	ND	0.54			
Total PeCDD	ND	0.54			
1,2,3,4,7,8-HxCDD	ND	1.3			
1,2,3,6,7,8-HxCDD	ND	1.0			
1,2,3,7,8,9-HxCDD	ND	1.1			
Total HxCDD	ND	2.4			
1,2,3,4,6,7,8-HpCDD	13		1.01	>10:1	
Total HpCDD	27		1.05	>10:1	
OCDD	83		0.87	>10:1	B
2,3,7,8-TCDF	110		0.79	>10:1	F
Total TCDF	240		0.76	>10:1	
1,2,3,7,8-PeCDF	1.1		1.53	4:1	
2,3,4,7,8-PeCDF	1.5		1.43	5:1	
Total PeCDF	4.8		1.42	5:1	
1,2,3,4,7,8-HxCDF	ND	0.35			
1,2,3,6,7,8-HxCDF	ND	0.27			
2,3,4,6,7,8-HxCDF	ND	0.32			
1,2,3,7,8,9-HxCDF	ND	0.42			
Total HxCDF	ND	0.42			
1,2,3,4,6,7,8-HpCDF	ND	1.7			
1,2,3,4,7,8,9-HpCDF	ND	0.56			
Total HpCDF	5.5		1.12	9:1	
OCDF	5.3		0.88	>10:1	

Analyst:

Reviewer: OM

PCDD & PCDF
EPA METHOD 8290

Sample ID: 418250DUP
Lab ID: 10065-001-DUP

Isotopic Recovery Results

Internal Standard:	% R	Ratio	Qualifier
^{13}C-2,3,7,8-TCDD	96	0.77	
^{13}C-1,2,3,7,8-PeCDD	107	1.60	
^{13}C-1,2,3,6,7,8-HxCDD	82	1.25	
^{13}C-1,2,3,4,6,7,8-HpCDD	90	1.05	
^{13}C-OCDD	62	0.92	
^{13}C-2,3,7,8-TCDF	81	0.80	
^{13}C-1,2,3,7,8-PeCDF	88	1.57	
^{13}C-1,2,3,4,7,8-HxCDF	72	0.52	
^{13}C-1,2,3,4,6,7,8-HpCDF	59	0.44	
Clean-up Recovery Standard:			
^{37}Cl-2,3,7,8-TCDD	84	NA	

Dates Analyzed:

DB-5: 11/08/90
DB-225: 11/12/90
SP-2331: NA

Analyst:

Reviewer:

PCDD & PCDF
MATRIX SPIKE

Sample ID: 418250MS
Lab ID: 10065-001-MS
Matrix: Sediment

Date Received: 10/25/90
Date Extracted: 10/31/90
Sample Amount: 10.00 g

ICAL ID: 18290CAL
QC LOT: LC1031S
Units: pg/g

COMPOUND	AMOUNT SPIKED	SAMPLE (pg/g)	MS (pg/g)	MS (%)
2,3,7,8-TCDD	20	ND	18.2	91
1,2,3,7,8-PeCDD	20	ND	17.5	88
1,2,3,4,7,8-HxCDD	50	ND	48	96
1,2,3,6,7,8-HxCDD	50	ND	48	96
1,2,3,7,8,9-HxCDD	50	ND	45	90
1,2,3,4,6,7,8-HpCDD	50	12	55	86
OCDD	100	65	144	79
2,3,7,8-TCDF	20	108	131	118
1,2,3,7,8-PeCDF	20	1.6	20	92
2,3,4,7,8-PeCDF	50	1.5	21	98
1,2,3,4,7,8-HxCDF	50	ND	37	74
1,2,3,6,7,8-HxCDF	50	ND	56	112
2,3,4,6,7,8-HxCDF	50	ND	48	96
1,2,3,7,8,9-HxCDF	50	ND	48	96
1,2,3,4,6,7,8-HpCDF	50	1.7	50	100
1,2,3,4,7,8,9-HpCDF	50	ND	58	117
OCDF	100	5.3	97	91

Analyst:

Reviewer: OM

PCDD & PCDF
MATRIX SPIKE

Lab ID: 10065-001-MS

Internal Standard Recoveries:

COMPOUND	MS %REC	QUALIFIER
^{13}C-2,3,7,8-TCDD	100	
^{13}C-1,2,3,7,8-PeCDD	111	
^{13}C-1,2,3,6,7,8-HxCDD	87	
^{13}C-1,2,3,4,6,7,8-HpCDD	93	
^{13}C-OCDD	65	
^{13}C-2,3,7,8-TCDF	77	
^{13}C-1,2,3,7,8-PeCDF	89	
^{13}C-1,2,3,4,7,8-HxCDF	95	
^{13}C-1,2,3,4,6,7,8-HpCDF	92	

Clean-up Recovery:

^{37}Cl-2,3,7,8-TCDD	89

Dates Analyzed:

DB-5: 11/08/90 DB-225: 11/12/90 SP-2331: NA

Analyst:

Reviewer:

PCDD & PCDF
EPA METHOD 8290

METHOD BLANK
Lab ID: 10065-001-MB
Matrix: Sediment

Date Received: NA
Date Extracted: 10/31/90
Sample Amount: 10.00 g

ICAL ID: I8290CAL
QC Lot: LC1031S
Units: pg/g

Compound	Conc.	D.L.	Ratio	S/N Ratio	Qualifier
2,3,7,8-TCDD	ND	0.82			
Total TCDD	ND	0.82			
1,2,3,7,8-PeCDD	ND	0.88			
Total PeCDD	ND	0.88			
1,2,3,4,7,8-HxCDD	ND	1.4			
1,2,3,6,7,8-HxCDD	ND	0.89			
1,2,3,7,8,9-HxCDD	ND	0.93			
Total HxCDD	ND	1.4			
1,2,3,4,6,7,8-HpCDD	ND	0.97			
Total HpCDD	ND	2.9			
OCDD	8.9		0.90		C
2,3,7,8-TCDF	ND	0.18			F
Total TCDF	ND	0.54			
1,2,3,7,8-PeCDF	ND	0.81			
2,3,4,7,8-PeCDF	ND	0.81			
Total PeCDF	ND	0.81			
1,2,3,4,7,8-HxCDF	ND	0.27			
1,2,3,6,7,8-HxCDF	ND	0.27			
2,3,4,6,7,8-HxCDF	ND	0.32			
1,2,3,7,8,9-HxCDF	ND	0.39			
Total HxCDF	ND	0.39			
1,2,3,4,6,7,8-HpCDF	ND	0.34			
1,2,3,4,7,8,9-HpCDF	ND	0.45			
Total HpCDF	ND	0.45			
OCDF	ND	0.85			

Analyst: ______

Reviewer:

PCDD & PCDF
EPA METHOD 8290

METHOD BLANK
Lab ID: 10065-001-MB

Isotopic Recovery Results

Internal Standard	% R	Ratio	Qualifier
^{13}C-2,3,7,8-TCDD	98	0.85	
^{13}C-1,2,3,7,8-PeCDD	117	1.57	
^{13}C-1,2,3,4,7,8-HxCDD	90	1.28	
^{13}C-1,2,3,4,6,7,8-HpCDD	79	1.04	
^{13}C-OCDD	67	0.90	
^{13}C-2,3,7,8-TCDF	78	0.80	
^{13}C-1,2,3,7,8-PeCDF	74	1.56	
^{13}C-1,2,3,4,7,8-HxCDF	68	0.52	
^{13}C-1,2,3,4,6,7,8-HpCDF	58	0.44	
Clean-up Recovery Standard:			
^{37}Cl-2,3,7,8-TCDD	84	NA	

Dates Analyzed:

DB-5: 11/08/90

DB-225: 11/12/90

SP-2331: NA

Analyst:

Reviewer:

Centrifuge Blank

TRIANGLE LABORATORIES, INC.
PCDD/PCDF 2378X ANALYSIS (b)

Page 1 of 2
07/18/90

FILE NAME....: S214404
CONCAL.......: S902143
ANALYST......: MC
SAMPLE SIZE..: 924.20 ml
ICAL DATE....: 06/27/90
SPIKE FILE...: SPX2372K

CLIENT ID....: WSDE
SAMPLE ID....: 168405
ANALYSIS DATE: 07/08/90
SAMPLE MATRIX: WATER
SAMPLE ORIGIN: n/a

TLI NUMBER....: 30-116-2
PROJECT NUMBER: 15632
DATE RECEIVED.: 05/02/90
DATE COLLECTED: / /
SHIPMENT NO...: WEYCO

NAME	CONC(ppt)	NUMBER	DL	EMPC	RATIO	RT	FLAGS
2378-TCDD	ND		0.01				—
12378-PeCDD	ND		0.01				—
123478-HxCDD	ND		0.01				—
123678-HxCDD	ND		0.008				—
123789-HxCDD	ND		0.01				—
1234678-HpCDD	ND		0.02				—
OCDD	0.13				0.80	51:46	B
2378-TCDF	ND		0.005				—
12378-PeCDF	ND		0.01				—
23478-PeCDF	ND		0.01				—
123478-HxCDF	ND		0.008				—
123678-HxCDF	ND		0.005				—
234678-HxCDF	ND		0.01				—
123789-HxCDF	ND		0.01				—
1234678-HpCDF	ND		0.008				—
1234789-HpCDF	ND		0.02				—
OCDF	ND		0.05				—
TOTAL TCDD	ND		0.01				—
TOTAL PeCDD	ND		0.01				—
TOTAL HxCDD	ND		0.01				—
TOTAL HpCDD	ND		0.02				—
TOTAL TCDF	ND		0.005				—
TOTAL PeCDF	ND		0.01				—
TOTAL HxCDF	ND		0.008				—
TOTAL HpCDF	ND		0.01				—

X237_RPT rev:3.03

7/18/90

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07/18/90

TRIANGLE LABORATORIES, INC.
PCDD/PCDF 2378X ANALYSIS (b) QA/QC SUMMARY

FILE NAME....: S214404
CONCAL.......: S902143
ANALYST......: MC
SAMPLE SIZE..: 924.20 ml
ICAL DATE....: 06/27/90
SPIKE FILE...: SPX2372K

CLIENT ID....: WSDE
SAMPLE ID....: 168405
ANALYSIS DATE: 07/08/90
SAMPLE MATRIX: WATER
SAMPLE ORIGIN: n/a

TLI NUMBER....: 30-116-2

PROJECT NUMBER: 15632
DATE RECEIVED.: 05/02/90
DATE COLLECTED: / /
SHIPMENT NO...: WEYCO

SURROGATE RECOVERY SUMMARY (TYPE B)

NAME	CONC (ppt)	% REC.	RATIO	RT	FLAGS
37Cl-TCDD	1.9	85.6		31:12	___
13C12-PeCDF 234	1.7	78.7	1.57	36:06	___
13C12-HxCDF 478	1.5	71.4	0.52	40:13	___
13C12-HxCDD 478	2.0	92.3	1.20	41:22	Q
13C12-HpCDF 789	1.7	77.1	0.42	47:02	___

ALTERNATE STANDARDS RECOVERY SUMMARY (TYPE B)

NAME	CONC (ppt)	% REC.	RATIO	RT	FLAGS
13C12-HxCDF 789	1.9	85.6	0.51	42:20	___
13C12-HxCDF 234	2.0	91.5	0.50	41:10	Q

INTERNAL STANDARDS RECOVERY SUMMARY

NAME	CONC (ppt)	% REC.	RATIO	RT	FLAGS
13C12-2378-TCDF	1.6	71.7	0.78	30:22	___
13C12-2378-TCDD	1.5	67.9	0.81	31:11	___
13C12-PeCDF 123	1.4	62.5	1.49	35:08	___
13C12-PeCDD 123	1.7	78.1	1.48	36:35	___
13C12-HxCDF 678	1.2	57.5	0.52	40:23	___
13C12-HxCDD 678	1.7	77.6	1.17	41:30	___
13C12-HpCDF 678	1.3	58.1	0.42	44:49	___
13C12-HpCDD 678	1.4	64.1	1.00	46:21	___
13C12-OCDD	0.98	22.5	0.86	51:44	V

X237_RPT rev:3.03

7/18/90

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State of Washington Department of Ecology
Manchester Environmental Laboratory
7411 Beach Dr. East Port Orchard WA. 98366

Data Review
January 5, 199~~2~~3

Project: **Lake Roosevelt**

Sample: 408560

Laboratory: Enseco California Analytical Laboratory 66398

By: Stuart Magoon SM

Case Summary

This sample was received at the Manchester Environmental Laboratory on October 12,1992, and transported to Enseco CAL October 13, 1992 for Polychlorinated Dibenzodioxin/furan (PCDD/PCDF) analysis.

These samples were prepared and analyzed according to EPA method 8290.

These analyses were reviewed for qualitative and quantitative accuracy, validity, and usefulness.

There is no need to assimilate the "dilution factor" or "sample wt/vol" into the final values reported; these calculations have already been figured into the reported values.

Note that results are reported on an as received basis.

Enseco uses an "ND" in conjunction with a detection limit instead of reporting a detection limit with a "U" qualifier.

The sample tag listed a date of September 30, 1992 as the collection date, where as the chain-of-custody lists October 2, 1992. The chain-of-custody date was used as the collection date.

PCDD/PCDF

Holding Times:

EPA method 8290 recommends holding times of thirty days (30) from the date of collection to the date of extraction and forty five (45) days total from collection to analysis.
These samples were analyzed within EPA method 8290 recommended holding times .

Method Blank:

No Dioxins/Furans were detected in the associated method blank.

Initial Calibration:

The five point initial calibration curves demonstrated good linearity with all relative standard deviations (RSD) within the 20 percent upper limit.
The isotopic ratios for all peaks used for identification were with +/- 15 percent of the theoretical value.

Continuing Calibration:

All the continuing calibration standards were within the RSD limit of 20% for the target analytes and 30% for the isotopically labeled compounds.

All the ion abundance ratios were within +/- 15% of the theoretical value.

Internal Standard Recoveries:

Internal standard recoveries are specified for the TCDD/TCDF only and all are above the lower limit of 20%.

Isotopic abundance ratios:

Every PCDD/PCDF isomer detected in this sample set met the isotopic abundance ratios criteria for positive identification.

Matrix Spikes (MS/MSD):

Matrix spike and spike duplicate recoveries and precision data are reasonable and acceptable, no QC limits have been established.

Summary:

This data is acceptable for use with the additional data qualifiers where appropriate ("J" qualifier added to several results due to quantitations performed outside of the calibration range).

California Analytical
Laboratory

Enseco
A Corning Company

November 11, 1992
Lab ID: 066398

Stuart Magoon
Washington State Department of Ecology
7411 Beech Dr. East
Port Orchard, WA 98366

Dear Mr. Magoon:

Enclosed is the report for the PCDD/PCDF and Total Organic Carbon analyses of your one soil sample for your Lake Roosevelt Trend Monitoring Project received at Enseco Cal Lab on 15 October 1992 under chain-of-custody.

Detection limits are reported on a sample specific basis and all results are recovery corrected per the isotope dilution technique for dioxin/furan analyses. The method blank is a laboratory-generated sample which assesses the degree to which laboratory operations and procedures cause false-positive analytical results for your samples.

For pulp and paper industry samples, test methods for chlorinated dioxin/furan analyses will follow NCASI Technical Bulletin 551 unless otherwise noted. Pulp and sludge samples are air dried and prepared per this method. All results for these analyses, including detection limits, are reported on a dry weight basis.

All other solid and waste samples are reported on an "as received" basis, i.e., no correction is made for moisture content, unless the method requires or the client requests that such correction be made.

Results are on the attached data sheets.

If you have any questions, please feel free to call.

Sincerely,

for
Eric W. Redman
Senior Scientist
Advanced Technology Group

Kathleen A. Gill
Kathleen A. Gill
Program Administrator

kaf

Enseco Incorporated
2544 Industrial Boulevard
West Sacramento, California 95691
916/372-1393 Fax: 916/372-7768

SAMPLE DESCRIPTION INFORMATION
for
Washington State Department of Ecology

Lab ID	Client ID	Matrix	Sampled Date	Sampled Time	Received Date
066398-0001-SA	408560	SOIL	02-oct-92 ~~30 SEP 92~~	~~09:00~~	15 OCT 92
066398-0001-DU	408560	SOIL	~~30 SEP 92~~	~~09:00~~	15 OCT 92
066398-0001-MS	408560	SOIL	~~30 SEP 92~~	~~09:00~~	15 OCT 92
066398-0001-SD	408560	SOIL	~~30 SEP 92~~	~~09:00~~	15 OCT 92
066398-0001-MB	Method Blank	SOIL			15 OCT 92

POLYCHLORINATED DIOXINS/FURANS
ISOMER SPECIFIC ANALYSIS
Method 8290

Client Name: Washington State Department of Ecology
Client ID: Method Blank
Lab ID: 066398-0001-MB
Matrix: SOIL
Authorized: 19 OCT 92

Sampled: NA
Prepared: 01 NOV 92

Received: NA
Analyzed: 06 NOV 92

Sample Amount 5.0 G
Column Type DB-5

Parameter	Result	Units	Detection Limit	Data Qualifiers
Furans				
TCDFs (total)	ND	pg/g	0.22	
2,3,7,8-TCDF	ND	pg/g	0.22	
PeCDFs (total)	ND	pg/g	0.32	
1,2,3,7,8-PeCDF	ND	pg/g	0.23	
2,3,4,7,8-PeCDF	ND	pg/g	0.32	
HxCDFs (total)	ND	pg/g	0.52	
1,2,3,4,7,8-HxCDF	ND	pg/g	0.52	
1,2,3,6,7,8-HxCDF	ND	pg/g	0.31	
2,3,4,6,7,8-HxCDF	ND	pg/g	0.28	
1,2,3,7,8,9-HxCDF	ND	pg/g	0.29	
HpCDFs (total)	ND	pg/g	1.5	
1,2,3,4,6,7,8-HpCDF	ND	pg/g	1.5	
1,2,3,4,7,8,9-HpCDF	ND	pg/g	0.22	
OCDF	ND	pg/g	1.1	
Dioxins				
TCDDs (total)	ND	pg/g	0.17	
2,3,7,8-TCDD	ND	pg/g	0.17	
PeCDDs (total)	ND	pg/g	1.0	
1,2,3,7,8-PeCDD	ND	pg/g	0.18	
HxCDDs (total)	ND	pg/g	0.25	
1,2,3,4,7,8-HxCDD	ND	pg/g	0.20	
1,2,3,6,7,8-HxCDD	ND	pg/g	0.25	
1,2,3,7,8,9-HxCDD	ND	pg/g	0.21	
HpCDDs (total)	ND	pg/g	0.24	
1,2,3,4,6,7,8-HpCDD	ND	pg/g	0.24	
OCDD	ND	pg/g	2.4	

(continued on following page)

ND = Not detected
NA = Not applicable

Reported By: Brett Bordelon Approved By: Andre Algazi

POLYCHLORINATED DIOXINS/FURANS
ISOMER SPECIFIC ANALYSIS (CONT.)
Method 8290

Client Name: Washington State Department of Ecology
Client ID: Method Blank
Lab ID: 066398-0001-MB
Matrix: SOIL Sampled: NA Received: NA
Authorized: 19 OCT 92 Prepared: 01 NOV 92 Analyzed: 06 NOV 92

Sample Amount 5.0 G
Column Type DB-5

	% Recovery
13C-2,3,7,8-TCDF	100
13C-2,3,7,8-TCDD	100
13C-1,2,3,7,8-PeCDF	99
13C-1,2,3,7,8-PeCDD	91
13C-1,2,3,4,7,8-HxCDF	86
13C-1,2,3,6,7,8-HxCDD	98
13C-1,2,3,4,6,7,8-HpCDF	102
13C-1,2,3,4,6,7,8-HpCDD	108
13C-OCDD	103

ND = Not detected
NA = Not applicable

Reported By; Brett Bordelon Approved By: Andre Algazi

Enseco
A Corning Company

POLYCHLORINATED DIOXINS/FURANS
ISOMER SPECIFIC ANALYSIS
Method 8290

Client Name: Washington State Department of Ecology
Client ID: 408560
Lab ID: 066398-0001-SA
Matrix: SOIL
Authorized: 19 OCT 92

Sampled: ~~30 SEP 92~~ 02 OCT 92
Prepared: 01 NOV 92

Received: 15 OCT 92
Analyzed: 06 NOV 92

Sample Amount 5.0 G
Column Type DB-5

Parameter	Result	Units wet weight	Detection Limit	Data Qualifiers
Furans				
TCDFs (total)	7.0	pg/g	--	W
2,3,7,8-TCDF	1.4 J	pg/g	--	1
PeCDFs (total)	ND	pg/g	0.56	
1,2,3,7,8-PeCDF	ND	pg/g	0.39	
2,3,4,7,8-PeCDF	ND	pg/g	0.56	
HxCDFs (total)	ND	pg/g	1.2	
1,2,3,4,7,8-HxCDF	ND	pg/g	0.55	
1,2,3,6,7,8-HxCDF	ND	pg/g	0.59	
2,3,4,6,7,8-HxCDF	ND	pg/g	0.54	
1,2,3,7,8,9-HxCDF	ND	pg/g	0.56	
HpCDFs (total)	ND	pg/g	2.2	
1,2,3,4,6,7,8-HpCDF	ND	pg/g	2.2	
1,2,3,4,7,8,9-HpCDF	ND	pg/g	0.21	
OCDF	ND	pg/g	3.0	
Dioxins				
TCDDs (total)	ND	pg/g	0.32	
2,3,7,8-TCDD	ND	pg/g	0.32	
PeCDDs (total)	ND	pg/g	1.0	
1,2,3,7,8-PeCDD	ND	pg/g	0.32	
HxCDDs (total)	ND	pg/g	1.3	
1,2,3,4,7,8-HxCDD	ND	pg/g	0.54	
1,2,3,6,7,8-HxCDD	ND	pg/g	0.68	
1,2,3,7,8,9-HxCDD	ND	pg/g	0.57	
HpCDDs (total)	17	pg/g	--	
1,2,3,4,6,7,8-HpCDD	7.8 J	pg/g	--	2
OCDD	52	pg/g	--	

(continued on following page)

ND = Not detected
NA = Not applicable

Reported By: Brett Bordelon

Approved By: Andre Algazi

The cover letter is an integral part of this report.

POLYCHLORINATED DIOXINS/FURANS
ISOMER SPECIFIC ANALYSIS (CONT.)
Method 8290

Client Name: Washington State Department of Ecology
Client ID: 408560
Lab ID: 066398-0001-SA
Matrix: SOIL
Authorized: 19 OCT 92
Sampled: ~~30 SEP 92~~ 02 OCT 92
Prepared: 01 NOV 92
Received: 15 OCT 92
Analyzed: 06 NOV 92

Sample Amount 5.0 G
Column Type DB-5

	% Recovery
13C-2,3,7,8-TCDF	102
13C-2,3,7,8-TCDD	107
13C-1,2,3,7,8-PeCDF	102
13C-1,2,3,7,8-PeCDD	95
13C-1,2,3,4,7,8-HxCDF	90
13C-1,2,3,6,7,8-HxCDD	96
13C-1,2,3,4,6,7,8-HpCDF	110
13C-1,2,3,4,6,7,8-HpCDD	110
13C-OCDD	109

Note w : MPC - Maximum Possible Concentration.

Note 1 : Result values are between the target detection limit and the lower calibration limit.

Note 2 : Result values are between the target detection limit and the lower calibration limit.

ND = Not detected
NA = Not applicable

Reported By: Brett Bordelon
Approved By: Andre Algazi

The cover letter is an integral part of this report.

POLYCHLORINATED DIOXINS/FURANS
ISOMER SPECIFIC ANALYSIS
Method 8290

Client Name: Washington State Department of Ecology
Client ID: 408560 (duplicate analysis)
Lab ID: 066398-0001-DU
Matrix: SOIL
Sampled: ~~30 SEP 92~~ 02 OCT 92
Received: 15 OCT 92
Authorized: 19 OCT 92
Prepared: 01 NOV 92
Analyzed: 06 NOV 92

Sample Amount 5.0 G
Column Type DB-5

Parameter	Result	Units	Detection Limit	Data Qualifiers
		wet weight		
Furans				
TCDFs (total)	5.1	pg/g	--	W
2,3,7,8-TCDF	1.2 J	pg/g	--	1
PeCDFs (total)	ND	pg/g	0.54	
1,2,3,7,8-PeCDF	ND	pg/g	0.37	
2,3,4,7,8-PeCDF	ND	pg/g	0.54	
HxCDFs (total)	ND	pg/g	0.50	
1,2,3,4,7,8-HxCDF	ND	pg/g	0.47	
1,2,3,6,7,8-HxCDF	ND	pg/g	0.50	
2,3,4,6,7,8-HxCDF	ND	pg/g	0.46	
1,2,3,7,8,9-HxCDF	ND	pg/g	0.47	
HpCDFs (total)	ND	pg/g	2.2	
1,2,3,4,6,7,8-HpCDF	ND	pg/g	0.26	
1,2,3,4,7,8,9-HpCDF	ND	pg/g	2.2	
OCDF	ND	pg/g	2.4	
Dioxins				
TCDDs (total)	ND	pg/g	0.36	
2,3,7,8-TCDD	ND	pg/g	0.36	
PeCDDs (total)	ND	pg/g	1.4	
1,2,3,7,8-PeCDD	ND	pg/g	0.22	
HxCDDs (total)	ND	pg/g	0.95	
1,2,3,4,7,8-HxCDD	ND	pg/g	0.55	
1,2,3,6,7,8-HxCDD	ND	pg/g	0.68	
1,2,3,7,8,9-HxCDD	ND	pg/g	0.58	
HpCDDs (total)	12	pg/g	--	
1,2,3,4,6,7,8-HpCDD	5.4 J	pg/g	--	2
OCDD	39	pg/g	--	

(continued on following page)

ND = Not detected
NA = Not applicable

Reported By: Brett Bordelon
Approved By: Andre Algazi

POLYCHLORINATED DIOXINS/FURANS
ISOMER SPECIFIC ANALYSIS (CONT.)
Method 8290

Client Name: Washington State Department of Ecology
Client ID: 408560
Lab ID: 066398-0001-DU
Matrix: SOIL Sampled: ~~30 SEP 92~~ 02 OCT 92 Received: 15 OCT 92
Authorized: 19 OCT 92 Prepared: 01 NOV 92 Analyzed: 06 NOV 92

Sample Amount 5.0 G
Column Type DB-5

	% Recovery
13C-2,3,7,8-TCDF	92
13C-2,3,7,8-TCDD	95
13C-1,2,3,7,8-PeCDF	91
13C-1,2,3,7,8-PeCDD	84
13C-1,2,3,4,7,8-HxCDF	78
13C-1,2,3,6,7,8-HxCDD	84
13C-1,2,3,4,6,7,8-HpCDF	99
13C-1,2,3,4,6,7,8-HpCDD	99
13C-OCDD	98

Note w : MPC - Maximum Possible Concentration.

Note 1 : Result values are between the target detection limit and the lower calibration limit.

Note 2 : Result values are between the target detection limit and the lower calibration limit.

ND = Not detected
NA = Not applicable

Reported By: Brett Bordelon Approved By: Andre Algazi

The cover letter is an integral part of this report.
Rev 230787

POLYCHLORINATED DIOXINS/FURANS
QUALITY CONTROL SUMMARY
Method 8290

Client Name: Washington State Department of Ecology
Client ID: 408560 Matrix Spike
Lab ID: 066398-0001-MS
Matrix: SOIL   Sampled: ~~30 SEP 92~~ 02-OCT-92   Received: 15 OCT 92
Authorized: 15 OCT 92   Prepared: 01 NOV 92   Analyzed: 06 NOV 92

Sample Amount: 5.0 g
Column Type: DB-5

Parameters	pg/g Found in Sample	pg/g Spiked	pg/g Found in MS Sample	% Recovery *
Furans				
2,3,7,8-TCDF	1.4	40	41	~~98~~ 99
1,2,3,7,8-PeCDF	ND	100	100	100
2,3,4,7,8-PeCDF	ND	100	85	85
1,2,3,4,7,8-HxCDF	ND	100	100	100
1,2,3,6,7,8-HxCDF	ND	100	101	101
1,2,3,7,8,9-HxCDF	ND	100	106	106
2,3,4,6,7,8-HxCDF	ND	100	105	105
1,2,3,4,6,7,8-HpCDF	ND	100	103	103
1,2,3,4,7,8,9-HpCDF	ND	100	102	102
OCDF	ND	200	180	90
Dioxins				
2,3,7,8-TCDD	ND	40	37	91
1,2,3,7,8-PeCDD	ND	100	92	92
1,2,3,4,7,8-HxCDD	ND	100	88	88
1,2,3,6,7,8-HxCDD	ND	100	95	95
1,2,3,7,8,9-HxCDD	ND	100	97	97
1,2,3,4,6,7,8-HpCDD	7.8	100	102	94
OCDD	52	200	230	89

(continued on following page)

* = Percent recoveries are calculated prior to rounding.
ND = Not Detected
NA = Not Applicable

Reported by: Brett Bordelon   Approved by: Andre Algazi

The cover letter is an integral part of this report.

POLYCHLORINATED DIOXINS/FURANS
QUALITY CONTROL SUMMARY (cont'd)
Method 8290

Client Name: Washington State Department of Ecology
Client ID: 408560 Matrix Spike
Lab ID: 066398-0001-MS
Matrix: SOIL   Sampled: ~~30 SEP 92~~ 02-Oct-92   Received: 15 OCT 92
Authorized: 15 OCT 92   Prepared: 01 NOV 92   Analyzed: 06 NOV 92

Sample Amount: 5.0 g
Column Type: DB-5

	% Recovery
13C-2,3,7,8-TCDF	93
13C-1,2,3,7,8-PeCDF	94
13C-1,2,3,4,7,8-HxCDF	92
13C-1,2,3,4,6,7,8-HpCDF	83
13C-2,3,7,8-TCDD	76
13C-1,2,3,7,8-PeCDD	83
13C-1,2,3,6,7,8-HxCDD	88
13C-1,2,3,4,6,7,8-HpCDD	97
13C-OCDD	100

ND = Not Detected
NA = Not Applicable

Reported by: Brett Bordelon   Approved by: Andre Algazi

The cover letter is an integral part of this report.

Enseco
A Corning Company

POLYCHLORINATED DIOXINS/FURANS
QUALITY CONTROL SUMMARY
Method 8290

Client Name: Washington State Department of Ecology
Client ID: 408560 Spike Duplicate
Lab ID: 066398-0001-SD
Matrix: SOIL
Sampled: ~~30 SEP 92~~ 02 OCT 92
Received: 15 OCT 92
Authorized: 15 OCT 92
Prepared: 01 NOV 92
Analyzed: 06 NOV 92

Sample Amount: 5.0 g
Column Type: DB-5

Parameters	pg/g Found in Sample	pg/g Spiked	pg/g Found in SD Sample	% Recovery *	RPD
Furans					
2,3,7,8-TCDF	1.4	40	41	~~98~~ 99	0.0
1,2,3,7,8-PeCDF	ND	100	101	101	1.0
2,3,4,7,8-PeCDF	ND	100	84	84	1.2
1,2,3,4,7,8-HxCDF	ND	100	102	102	2.0
1,2,3,6,7,8-HxCDF	ND	100	102	102	1.0
1,2,3,7,8,9-HxCDF	ND	100	108	108	1.9
2,3,4,6,7,8-HxCDF	ND	100	103	103	1.9
1,2,3,4,6,7,8-HpCDF	ND	100	106	106	2.9
1,2,3,4,7,8,9-HpCDF	ND	100	110	111	7.5
OCDF	ND	200	190	93	5.4
Dioxins					
2,3,7,8-TCDD	ND	40	38	95	2.7
1,2,3,7,8-PeCDD	ND	100	94	94	2.2
1,2,3,4,7,8-HxCDD	ND	100	87	87	1.1
1,2,3,6,7,8-HxCDD	ND	100	95	95	0.0
1,2,3,7,8,9-HxCDD	ND	100	94	94	3.1
1,2,3,4,6,7,8-HpCDD	7.8	100	99	92	3.0
OCDD	52	200	220	82	4.4

(continued on following page)

* = Percent recoveries are calculated prior to rounding.
ND = Not Detected
NA = Not Applicable

Reported by: Brett Bordelon

Approved by: Andre Algazi

The cover letter is an integral part of this report.

POLYCHLORINATED DIOXINS/FURANS
QUALITY CONTROL SUMMARY (cont'd)
Method 8290

Client Name: Washington State Department of Ecology
Client ID: 408560 Spike Duplicate
Lab ID: 066398-0001-SD
Matrix: SOIL
Sampled: ~~30 SEP 92~~ 02 oct 92
Received: 15 OCT 92
Authorized: 15 OCT 92
Prepared: 01 NOV 92
Analyzed: 06 NOV 92

Sample Amount: 5.0 g
Column Type: DB-5

	% Recovery
13C-2,3,7,8-TCDF	104
13C-1,2,3,7,8-PeCDF	104
13C-1,2,3,4,7,8-HxCDF	90
13C-1,2,3,4,6,7,8-HpCDF	120
13C-2,3,7,8-TCDD	107
13C-1,2,3,7,8-PeCDD	93
13C-1,2,3,6,7,8-HxCDD	103
13C-1,2,3,4,6,7,8-HpCDD	123
13C-OCDD	125

ND = Not Detected
NA = Not Applicable

Reported by: Brett Bordelon

Approved by: Andre Algazi

The cover letter is an integral part of this report.

Total Organic Carbon (TOC)-Walkley-Black

Method 29-3.52

Client Name: Washington State Department of Ecology
Matrix: SOIL Received: 15 OCT 92
Units: %Carbon Authorized: 19 OCT 92

Lab ID	Client ID	Result	Reporting Limit	Date Prepared	Date Analyzed
066398-0001-SA	408560	2.2	0.010	NA	03 NOV 92

ND = Not detected
NA = Not applicable

Reported By: Darlene Ogburn Approved By: Flo Burbano

The cover letter is an integral part of this report.
Rev 230787

Received 1/12/94

State of Washington Department of Ecology
Manchester Environmental Laboratory
7411 Beach Dr. East Port Orchard WA. 98366

Data Review
December 27, 1993

Project: **Lake Roosevelt**

Samples: 418380 (suspended sediment)

Laboratory: Triangle Laboratories Inc. 25912

By: Stuart Magoon SM

Case Summary for Tetra through Octa chlorodibenzo-p-dioxin and furan
(PCDD and PCDF)

Data from these analyses were reviewed for qualitative and quantitative accuracy, validity, and usefulness. These samples were prepared and analyzed according to EPA method 8290.

The results are reported in Pico grams per gram (pg/g) which is parts per trillion dry weight.

Triangle Laboratories "EPMC" flag should be considered synonymous with the Manchester Laboratory "UJ" qualifier.

Triangle Laboratories use of the "E" flag can be ignored, if it was significant then the associated result has been qualified with a "J".

There is a number reported for each analyte that appears in one of three columns. If the number appears in the column labeled "Conc. (ppt)" then this analyte has been detected at the level reported. If the number appears in the column labeled "DL" then the analyte was not detected and the number is the detection limit at or above which the analyte was not detected (there should be an "ND", short for not detected, that appears in the "Conc. (ppt)" column). If there is a number in the column labeled "EMPC" then this means that the analyte was not detected at or above the estimated concentration listed; all of these values have been qualified with a "UJ" .

Triangle Laboratories has developed their own data "flags". The definitions of these "flags" are described in the Case Narrative. The "flags" are to be considered separate from "data qualifiers". Flags are added by the laboratory performing the analysis, usually the analyst. Qualifiers are added by the data reviewer as part of addressing the usability of the data. Generally the flags signal the reviewer to access the results and determine what to do about the fact that flags were added. For your reporting purposes the "flags" should not be considered part of the final result. The qualifiers, however, are to be considered part of the final result.

Suspended Sediment

Holding times:

EPA method 8290 recommends holding times of thirty days (30) from the date of collection to the date of extraction and forty five (45) days total from collection to analysis. The method also states "PCDDs and PCDFs are very stable in a variety of matrices, and holding times under the conditions listed in Section 6.4 may be as high as a year for certain matrices.
Considering the environmental persistence of dioxin and furans no qualifiers have been added due to holding times.

Method Blank:

Several analytes were detected in the sediment method blank. **Octachlorodibenzodioxin (OCDD), was detected in this blank at 9.2 ng/kg (parts per trillion).** OCDD was also detected in the sediment sample. The OCDD detected in the sediment sample was much greater than five times the amount detected in the method blank. The "B" flag was used to indicate that some of the OCDD detected in this sample may be due to laboratory contamination. The contribution of HpCDD from potential laboratory contamination should be considered insignificant; no data qualification was warranted.

EMPC (estimated maximum possible contamination) results for the blank were given for **Heptachlorodibenzodioxin (HpCDD),** 1,2,3,4,6,7,8-**Heptachloro- dibenzofuran** (1,2,3,4,6,7,8-**HpCDF), Octachlorodibenzofuran (OCDF). These analytes** were also detected in the sample. However, the amounts detected in the sample were considerably higher and the possible contribution from laboratory contamination should be considered negligible.

2,3,4,6,7,8 Hexachlorodibenzofuran (2,3,4,6,7,8-HxCDF) was the only analyte detected in both the method blank and the sample where the amounts were similar. The 2,3,4,6,7,8-HxCDF sample result has been qualified with a "UJ" because the detection of this analyte in the sample is most likely due to laboratory contamination.

Calibration:

The calibration standards were within the relative standard deviation (RSD) limit of 20% for the target analytes and 30% for the isotopically labeled compounds.
All the ion abundance ratios were within +/- 15% of the theoretical value.

Internal Standard Recoveries:

All Internal standard recoveries were above the lower limit of 20%.

Isotopic abundance ratios:

Every dioxin and furan isomer reported as detected met the isotopic abundance ratios criteria for positive identification.

Summary:

This data is acceptable for use. Sample results for 1,2,3,7,8,9-HxCDD and 1,2,3,7,8-PeCDF have been qualified with a "J" to indicate that these two results should be considered estimates; Poor peak resolution ("PR" flag) may have biased the amounts high.

The EMPC values are analogous to a result with a "UJ" qualifier.

TRIANGLE LABS

CASE NARRATIVE

Sediment.

Analysis of Samples for the Presence of Polychlorinated Dibenzo-*p*-Dioxins and Dibenzofurans by High-Resolution Gas Chromatography / High-Resolution Mass Spectrometry

METHOD 8290

Date :	NOVEMBER 30, 1993
Client ID :	WASHINGTON STATE DEPARTMENT OF ECOLOGY
P.O. Number :	
TLI Project Number	25912

Rev. 10/21/93

Triangle Laboratories of RTP, Inc.
801 Capitola Drive
Durham, NC 27713
919-544-5729
P.O. Box 13485
Research Triangle Park, NC 27709
Fax # 919-544-5491

Overview

One sediment sample was received from WASHINGTON STATE DEPT. OF ECOLOGY in good condition November 2, 1993 and stored in a refrigerator at 4°C. The Hazardous Waste Client Services Manager, Lorri White, requested more sample from the client because there was not enough to obtain adequate detection limits. The client sent additional sample on November 17, 1993. The sample was extracted and analyzed according to procedures described in the Triangle Laboratories Data User Manual (Rev. 12/92-LLW-7-AH-2/93). Any particular difficulties encountered during the sample handling by Triangle Labs will be discussed in the QC Remarks section below. Results reported relate only to the items tested.

Quality Control Samples

A laboratory method blank -- identified as the TLI Sediment Blank -- was prepared along with the sample.

QC Remarks

The release of this particular set of WASHINGTON STATE DEPT. OF ECOLOGY analytical data by Triangle Labs was authorized by the Quality Control Chemist who has reviewed each sample data package individually following a series of inspections/reviews. When applicable, general deviations from acceptable QC requirements are identified below and comments are made on the effect of these deviations upon the validity and reliability of the results. Please consult Triangle Laboratories' Data User Manual for further details. Specific QC issues associated with this particular project are:

***Sample Preparation Laboratory*:** The two aliquots of sediment received on November 2 and 17, 1993 were composited into one sample and extracted. A 1.0 gram aliquot was taken for the Total Organic Carbon subcontract analysis. This sample was very wet (83% moisture) and greenish-brown. There was only 29.59 grams wet weight (i.e. 5.0 dry weight) to extract after percent moisture was measured using a 1-1.5 gram aliquot.

***Mass Spectrometry*:** None

***Data Review*:** Despite the small sample size, detection limits were well below 1 ppt due to good instrument sensitivity.

Other Comments:
Any analytes found in the TLI Blank are detected at a level equal to or less than the Target Detection Limit. This level of contamination is acceptable as per TLI Guidelines. OCDD is not subject to blank contamination criteria as per TLI Guidelines.

Sample Calculations:

Analyte Concentration

The concentration or amount of any analyte is calculated using the following expression.

$$C(\delta) = \frac{A_\delta * Q_\beta}{A_\beta * RRF(\delta) * W}$$

where:

$C(\delta)$ is the concentration or amount of a given analyte,

A_δ is the integrated current for the characteristic ions of the analyte,

A_β is the integrated current of the characteristic ions of the corresponding internal standard,

Q_β represents the amount of internal standard added to the sample before extraction,

$RRF(\delta)$ is the mean analyte relative response factor from the initial calibration (ICal) and,

W is the sample weight or volume

Detection Limits

The detection limit reported for a target analyte that is not detected or presents an analyte response that is less than 2.5 times the background level is calculated by using the following expression. The area of the analyte is replaced by the noise level measured in a region of the chromatogram clear of genuine GC signals multiplied by an empirically determined factor. The detection limits represent the maximum possible concentration of a target analyte that could be present without being detected.

$$DL(\delta) = \frac{2 * 2.5 * (F * H) * Q_\beta}{A_\beta * RRF(\delta) * W}$$

where:

$DL(\delta)$ is the estimated detection limit for a target analyte,

2.5 is the minimum response required for a GC signal,

F is an empirical number that approximates the area to height ratio for a GC sign al. This number is 5 for the DB-5 column and 3.5 for the DB-225 column,

H is the height of the noise,

A_{β} is the integrated current of the characteristic ions of the corresponding internal standard,

Q_{β} represents the amount of internal standard added to the sample before extraction,

$RRF_{(\delta)}$ is the mean analyte relative response factor from the initial calibration (Ical) and,

W is the sample weight or volume

Other sample calculations may be found in Triangle Laboratories' Data User Manual.

Data Flags

A "B" flag is used to indicate that an analyte has been detected in the laboratory method blank as well as in an associated field sample. This flag denotes possible contribution of background laboratory contamination to the concentration or amount of that analyte detected in the field sample. Under Triangle Laboratories of RTP guidelines, a laboratory blank is acceptable if one of the following conditions is satisfied: 1) the tetra through hepta CDD/CDF levels are all below the target detection limits (TDLs), 2) the analyte levels found are all below 1/3 the theoretical method detection limit (TMQL), or 3) the contamination levels are less than 5% of the levels detected in the associated field samples. If these conditions are satisfied or if the blank is unable to be reextracted, the interpretation of the contamination levels relative to the samples should be as follows: 1) analyte quantitations should be considered valid if the level of blank contamination is less than five percent of the level detected in the field sample, 2) analyte quantitations should be considered estimated if the analyte level in the sample is five to twenty times the level of the analyte in the blank, or 3) analytes whose level in a sample is the same as or less than five times the level detected in the associated blank should be considered present likely due to laboratory contamination and not native to the sample.

An "E" flag is used to indicate that a PCDF peak has eluted at the same time as the associated diphenyl ether (DPE) and that the DPE peak intensity is ten percent or more of the PCDF peak intensity. Total PCDF values are flagged "E" if the total DPE contribution to the total PCDF value is greater than ten percent. All PCDF peaks that are significantly influenced by the presence of DPE peaks are quantitated with EMPC values, regardless of the isotopic abundance ratio. These EMPC values are most likely overestimated due to the DPE contribution to the peak area.

An "I" flag is used to indicate labeled standards have been interfered with on the GC column by coeluting, interferent peaks. The interference may have caused the standard's area to be overestimated. All quantitations relative to this standard, therefore, may be underestimated.

A "PR" flag is used to indicate that a GC peak is poorly resolved. This resolution problem may be seen as two closely eluting peaks without a reasonable valley between the peak tops, overly broad peaks, or peaks whose shapes vary greatly from a normal distribution. The concentrations or amounts reported for such peaks are most likely overestimated.

A "Q" flag is used to indicate the presence of QC ion instabilities caused by quantitative interferences. Affected analytes may be overestimated or underestimated as a result of this interference. A peak is flagged "Q" only if it is affected by a QC ion deviation greater than 20% full scale as determined relative to the labeled standard against which it is quantitated. Total PCDD/PCDF quantitations will be flagged "Q" if the interferences affect ten percent or more of the total PCDD/PCDF peak areas.

An "RO" flag is used to indicate that a labeled standard has an ion-abundance ratio that is outside of the acceptable QC limits, most likely due to a coeluting interference. This may have caused the percent recovery of the standard to be overestimated. All quantitations versus this standard, therefore, may be underestimated.

A "U" flag is used to indicate that a specific (2,3,7,8-substituted) isomer cannot be resolved from a large, coeluting interferent GC peak. The specific isomer is reported as not detected as a valid concentration/amount cannot be determined. The calculated detection limit, therefore, should be considered an underestimated value.

A "V" flag is used to indicate that, although the percent recovery of a labeled standard may be below a specific QC limit, the signal-to-noise ratio of the peak is greater than 10:1. The standard is considered reliably quantifiable. All quantitations derived from the standard are considered valid as well.

By our interpretation, the analytical data in this project are valid based on the guidelines of EPA Method 8290 (Rev. 0) and Triangle Laboratories' Method 8290 Data User Manual. Any specific QC concerns or problems have been discussed in the QC REMARKS section with emphasis on their affect on the data. Should WASHINGTON STATE DEPT. OF ECOLOGY have any questions or comments regarding this data package, please feel free to contact Lorri White, Hazardous Waste Client Services Manager, at (919) 544-8352.

For Triangle Laboratories of RTP, Inc.,

Report Preparation

Diane H. Williford

Diane H. Williford

Report Preparation Chemist

Quality Control

Stuart R. Jefferys

Report Preparation Chemist

Total pages in report: 478

TL-RTP Project: **25912**
Client Sample: **418380**

Method 8290 PCDD/PCDF Analysis (b)
Analysis File: **S935774**

Client Project:	**LAKE ROOSEVELT TREND MONITOR.**				
Sample Matrix:	**SEDIMENT**	Date Received:	**11/17/93**	Spike File:	**SPX2372S**
TLRTP ID:	*	Date Extracted:	**11/20/93**	ICAL:	**SF5N163**
		Date Analyzed:	**11/24/93**	CONCAL:	**S935761**
Sample Size:	**29.588 g**	Dilution Factor:	**n/a**	% Moisture:	**83.1**
Dry Weight:	**5.000 g**	Blank File:	**S935773**	% Lipid:	**n/a**
GC Column:	**DB-5**	Analyst:	**ML**	% Solids:	**16.9**

Analytes	Conc. (ppt)	DL	EMPC	Ratio	RT	Flags
2,3,7,8-TCDD	ND	0.4				—
1,2,3,7,8-PeCDD	EMPC		0.48 UJ Sm			—
1,2,3,4,7,8-HxCDD	1.5			1.17	34:08	—
1,2,3,6,7,8-HxCDD	3.0			1.33	34:13	—
1,2,3,7,8,9-HxCDD	3.2 J			1.39	34:31	PR
1,2,3,4,6,7,8-HpCDD	74.0			1.01	37:29	B
1,2,3,4,6,7,8,9-OCDD	482			0.84	41:04	B
2,3,7,8-TCDF	4.2			0.66	25:44	—
1,2,3,7,8-PeCDF	0.70 J			1.73	29:52	PR
2,3,4,7,8-PeCDF	EMPC		0.65 UJ Sm			—
1,2,3,4,7,8-HxCDF	EMPC		1.3 UJ Sm			—
1,2,3,6,7,8-HxCDF	0.59			1.16	33:32	—
2,3,4,6,7,8-HxCDF	Sm ~~1.7~~	1.7 UJ		1.13	34:01	B,PR
1,2,3,7,8,9-HxCDF	ND	0.5				—
1,2,3,4,6,7,8-HpCDF	8.5			0.96	36:26	B
1,2,3,4,7,8,9-HpCDF	EMPC		0.91 UJ Sm			—
1,2,3,4,6,7,8,9-OCDF	23.6			0.92	41:16	B

Totals	Conc. (ppt)	Number	DL	EMPC	Flags
Total TCDD	4.2	2			—
Total PeCDD	0.78 J	1		4.6	—
Total HxCDD	34.9	6		35.4	—
Total HpCDD	165	2			—
Total TCDF	12.9	7		26.5	E
Total PeCDF	7.5	4		11.4	
Total HxCDF	14.7	6		16.0	—
Total HpCDF	29.2	3		29.9	—

WASHINGTON STATE DEPT. OF ECOLOGY

Method 8290 PCDD/PCDF Analysis (b)
Analysis File: **S935774**

L-RTP Project: **25912**
Client Sample: **418380**

Internal Standards	Conc. (ppt)	% Recovery	Ratio	RT	Flags
$^{13}C_{12}$-2,3,7,8-TCDF	279	69.7	0.78	25:43	—
$^{13}C_{12}$-2,3,7,8-TCDD	327	81.7	0.79	26:29	—
$^{13}C_{12}$-1,2,3,7,8-PeCDF	394	98.5	1.60	29:52	—
$^{13}C_{12}$-1,2,3,7,8-PeCDD	473	118	1.56	30:56	—
$^{13}C_{12}$-1,2,3,6,7,8-HxCDF	306	76.6	0.51	33:32	—
$^{13}C_{12}$-1,2,3,6,7,8-HxCDD	350	87.5	1.21	34.13	—
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF	350	87.5	0.43	36:25	—
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD	425	106	1.01	37:27	—
$^{13}C_{12}$-1,2,3,4,6,7,8,9-OCDD	1060	133	0.86	41:03	—

Surrogate Standards (Type B)	Conc. (ppt)	% Recovery	Ratio	RT	Flags
$^{37}Cl_4$-2,3,7,8-TCDD	30.6	76.6		26:30	—
$^{13}C_{12}$-2,3,4,7,8-PeCDF	431	108	1.56	30:36	—
$^{13}C_{12}$-1,2,3,4,7,8-HxCDF	319	79.8	0.50	33:25	—
$^{13}C_{12}$-1,2,3,4,7,8-HxCDD	366	91.5	1.18	34:08	—
$^{13}C_{12}$-1,2,3,4,7,8,9-HpCDF	384	96.0	0.50	37:57	—

Alternate Standards (Type B)	Conc. (ppt)	% Recovery	Ratio	RT	Flags
$^{13}C_{12}$-1,2,3,7,8,9-HxCDF	361	90.1	0.50	34:47	—
$^{13}C_{12}$-2,3,4,6,7,8-HxCDF	340	84.9	0.50	34:01	—

Recovery Standards	Ratio	RT	Flags
$^{13}C_{12}$-1,2,3,4-TCDD	0.81	26:17	—
$^{13}C_{12}$-1,2,3,7,8,9-HxCDD	1.21	34:31	—

Quality Control Notes:
* TLI SAMPLE ID 73-14-1 AND 72-79-1

Data Reviewer: DMW 11/30/93

X237_PSR v:1.06; LARS 5.10.01

Printed: 18:13 11/30/93

2,3,7,8-TCDF confirmation

WASHINGTON STATE DEPT. OF ECOLOGY

TL-RTP Project: **25912**
Client Sample: **418380**

Method 8290 TCDD/TCDF Analysis (DB-225)
Analysis File: **W296003**

Client Project:	LAKE ROOSEVELT TREND MONITOR.				
Sample Matrix:	SEDIMENT	Date Received:	11/17/93	Spike File:	SPC2NF2S
TLRTP ID:	*	Date Extracted:	11/20/93	ICAL:	WF29113
		Date Analyzed:	11/26/93	CONCAL:	W932958
Sample Size:	29.588 g	Dilution Factor:	n/a	% Moisture:	83.1
Dry Weight:	5.000 g	Blank File:	NA	% Lipid:	n/a
GC Column:	DB-225	Analyst:	ML	% Solids:	16.9

Analytes	Conc. (ppt)	DL	EMPC	Ratio	RT	Flags
2,3,7,8-TCDD	ND	0.7				—
2,3,7,8-TCDF	4.5			0.78	19:36	—

Totals	Conc. (ppt)	Number	DL	EMPC	Flags
Total TCDD	4.1	1		5.9	—
Total TCDF	29.4	11		37.8	E

Internal Standards	Conc. (ppt)	% Recovery	Ratio	RT	Flags
$^{13}C_{12}$-2,3,7,8-TCDF	337	84.2	0.80	19:34	—
$^{13}C_{12}$-2,3,7,8-TCDD	277	69.3	0.81	18:24	—

Surrogate Standard (Type B)	Conc. (ppt)	% Recovery	Ratio	RT	Flags
$^{37}Cl_4$-2,3,7,8-TCDD	29.8	74.5		18:25	—

Recovery Standard	Ratio	RT	Flags
$^{13}C_{12}$-1,2,3,4-TCDD	0.79	18:38	—

Quality Control Notes:
TLI SAMPLE ID 73-14-1 AND 72-79-1

Data Reviewer: ______________ 11/30/93

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WASHINGTON STATE DEPT. OF ECOLOGY

TL-RTP Project: **25912**
Client Sample: **TLI SEDIMENT BLANK**

Method 8290 PCDD/PCDF Analysis (b)
Analysis File: **S935773**

Client Project:	n/a			Spike File:	SPX2372S
Sample Matrix:	SEDIMENT	Date Received:	/ /	ICAL:	SF5N163
TLRTP ID:	TLI BLANK	Date Extracted:	11/20/93	CONCAL:	S935761
		Date Analyzed:	11/24/93		
Sample Size:	5.000 g	Dilution Factor:	n/a	% Moisture:	n/a
Dry Weight:	n/a	Blank File:	S935773	% Lipid:	n/a
GC Column:	DB-5	Analyst:	ML	% Solids:	n/a

Analytes	Conc. (ppt)	DL	EMPC	Ratio	RT	Flags
2,3,7,8-TCDD	ND	0.5				—
1,2,3,7,8-PeCDD	ND	0.7				—
1,2,3,4,7,8-HxCDD	ND	0.9				—
1,2,3,6,7,8-HxCDD	ND	0.8				—
1,2,3,7,8,9-HxCDD	ND	0.8				—
1,2,3,4,6,7,8-HpCDD	EMPC		0.83 UJ			—
1,2,3,4,6,7,8,9-OCDD	9.2			0.82	41:04	—
2,3,7,8-TCDF	ND	0.4				—
1,2,3,7,8-PeCDF	ND	0.5				—
2,3,4,7,8-PeCDF	ND	0.5				—
1,2,3,4,7,8-HxCDF	ND	0.6				—
1,2,3,6,7,8-HxCDF	ND	0.5				—
2,3,4,6,7,8-HxCDF	0.64			1.18	34:01	—
1,2,3,7,8,9-HxCDF	ND	0.8				—
1,2,3,4,6,7,8-HpCDF	EMPC		0.40 UJ			—
1,2,3,4,7,8,9-HpCDF	ND	0.9				—
1,2,3,4,6,7,8,9-OCDF	EMPC		2.4 UJ			—

Totals	Conc. (ppt)	Number	DL	EMPC	Flags
Total TCDD	ND		0.5		—
Total PeCDD	ND		0.7		—
Total HxCDD	ND		0.9		—
Total HpCDD	0.92	1		1.8	—
Total TCDF	ND		0.4		—
Total PeCDF	ND		0.5		—
Total HxCDF	0.63	1			—
Total HpCDF	1.3	1		2.2	—

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WASHINGTON STATE DEPT. OF ECOLOGY

TL-RTP Project: **25912**
Client Sample: TLI SEDIMENT BLANK

Method 8290 PCDD/PCDF Analysis (b)
Analysis File: S935773

Internal Standards	Conc. (ppt)	% Recovery	Ratio	RT	Flags
$^{13}C_{12}$-2,3,7,8-TCDF	277	69.3	0.76	25:43	—
$^{13}C_{12}$-2,3,7,8-TCDD	304	76.1	0.80	26:30	—
$^{13}C_{12}$-1,2,3,7,8-PeCDF	357	89.3	1.56	29:52	—
$^{13}C_{12}$-1,2,3,7,8-PeCDD	466	116	1.50	30:56	—
$^{13}C_{12}$-1,2,3,6,7,8-HxCDF	314	78.6	0.51	33:32	—
$^{13}C_{12}$-1,2,3,6,7,8-HxCDD	373	93.3	1.20	34:13	—
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF	377	94.2	0.44	36:25	—
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD	436	109	1.00	37:27	—
$^{13}C_{12}$-1,2,3,4,6,7,8,9-OCDD	986	123	0.87	41:03	—

Surrogate Standards (Type B)	Conc. (ppt)	% Recovery	Ratio	RT	Flags
$^{37}Cl_4$-2,3,7,8-TCDD	27.7	69.2		26:30	—
$^{13}C_{12}$-2,3,4,7,8-PeCDF	360	90.0	1.53	30:36	—
$^{13}C_{12}$-1,2,3,4,7,8-HxCDF	313	78.3	~~0.50~~	~~33:25~~	—
$^{13}C_{12}$-1,2,3,4,7,8-HxCDD	360	90.0	1.18	34:08	—
$^{13}C_{12}$-1,2,3,4,7,8,9-HpCDF	409	102	0.43	37:57	—

Alternate Standards (Type B)	Conc. (ppt)	% Recovery	Ratio	RT	Flags
$^{13}C_{12}$-1,2,3,7,8,9-HxCDF	331	82.8	0.48	34:47	—
$^{13}C_{12}$-2,3,4,6,7,8-HxCDF	336	84.1	0.51	34:01	—

Recovery Standards	Ratio	RT	Flags
$^{13}C_{12}$-1,2,3,4-TCDD	0.79	26:18	—
$^{13}C_{12}$-1,2,3,7,8,9-HxCDD	1.18	34:31	—

Data Reviewer: Stuart J for DHW 11/30/93

State of Washington Department of Ecology
Manchester Environmental Laboratory
7411 Beach Dr. East Port Orchard WA. 98366

Data Review
December 5, 1994

Project: **Lake Roosevelt**

Samples: 428090 (suspended sediment)

Laboratory: Pacific Analytical Inc.

By: Stuart Magoon

Case Summary for Tetra through Octa chlorodibenzo-p-dioxin and furan
(PCDD and PCDF)

Data from these analyses were reviewed for qualitative and quantitative accuracy, validity, and usefulness. These samples were prepared and analyzed according to EPA method 1613.

The results are reported in Nanograms grams per Kilogram (NG/KG) which is parts per trillion dry weight.

There is a number reported for each analyte that appears in one of two columns. If the number appears in the column labeled "CONCENTRATION FOUND" then this analyte has been detected at the level reported. If the number appears in the column labeled "DETECTION LIMIT" then the analyte was not detected and the number is the detection limit at or above which the analyte was not detected. (there should be a "U" qualifier associated with this number).

Suspended Sediment

Holding times:

EPA method 1613 states that samples analysis is to be performed within forty days (40) from extraction. There is no mention of a holding time from collection to extraction in method 1613. Dioxins/furans are very stable in a variety of matrices, and holding times under the conditions listed in Section 9, should insure minimal degradation for many weeks. EPA method 1613 B (in review status) states a one year holding time from collection to extraction.

Considering the environmental persistence of dioxin and furans no qualifiers have been added due to holding times.

Method Blank:

TCDD was detected in the method blank. The on column amounts detected in the method blank and the sample were 0.26 and 0.63 picograms respectively. Since the amount detected in the sample was only 2.4 times higher than the amount detected in the method blank, the TCDD detected in the sample may be due in part or solely to laboratory contamination. Since there is no way to know for sure whether or not the TCDD detected in the sample is native or due to contamination, the TCDD sample result has been qualified with a "UJ". According to Table 2 in method 1613, the minimum allowable contamination for TCDD in a method blank is 0.5 picograms per micoliter. Since the amount of TCDD detected in the method blank was below 0.5 picograms, no corrective action was taken. There was also insufficient sample to perform the duplicate analysis as requested, without elevating the detection limits. The TCDD detected in the method blank has been qualified with a "J" , because it was detected below the lowest calibration point, and should be considered an estimate.

Calibration:

The calibration standards were within the relative standard deviation (RSD) limit of 20%. All the ion abundance ratios were within the established QC limits listed in Table 3A.

Internal Standard Recoveries:

All Internal standard recoveries were within the specified recovery range of 25 - 150%, with one exception. The Carbon 13 OCDD recovery in the sample was 21%. Due to the low isotope recovery of C_{13} OCDD, the OCDD detected in this sample has been qualified as an estimate ("J" qualifier"), and the undetected OCDF has been qualified with a "UJ".

Isotopic abundance ratios:

Every dioxin and furan isomer reported as detected met the isotopic abundance ratios criteria for positive identification.

Summary:

Every dioxin/furan detected in this sample with the exception of TCDD and OCDD was detected below the lowest calibration point, and has therefore been qualified as an estimate ("J" qualifier added to the result). This data is acceptable for use as amended.

PACIFIC ANALYTICAL
6349 Paseo Del Lago, Suite102
Carlsbad, CA 92009
619-438-3100

November 28, 1994

Stuart Magoon
WA State Dept. of Ecology
Manchester Laboratory
7411 Beach Drive East
Port Orchard WA 98366-8204

Subject: Analysis of Dioxins and Furans by EPA Method 1613.

Samples: 428090

Please find enclosed the data package for the one soil sample received on October 28,1994. The amount extracted was 6.84 grams dry weight. The final extract volume was 20 ul. The sample was analyzed for Dioxins and Furans by EPA Method 1613 with the addition of estimated detection limits.

The estimated detection limits are flag with a "U" and calculated by taking the noise height at each analyte retention time. Confirmation analysis was run for 2,3,7,7-TCDF.

If you have any questions please call.

submitted by
Dante Bencivengo

USEPA - ITD

FORM 1A
PCDD/PCDF ANALYSIS DATA SHEET
Use for Sample and Blank Results

EPA SAMPLE NO.

428090

Lab Name: Pacific Analytical, Inc. Episode No.:

Contract No.: SAS No.: Lab Sample ID: A5801

Matrix (aqueous/solid/leachate): SOLID Sample Wt/Vol: 41.0 g or mL: G

Sample Receipt Date: 10/28/94 Initial Calibration Date: 08/27/94

Ext. Date: 11/02/94 Shift: 0800 Instrument ID: VG#5

Analysis Date: 11/11/94 Time: 1630 GC Column ID: DB5 60M

Extract Volume (uL): 20 Sample Data Filename: A5801

Injection Volume (uL): 1 Blank Data Filename: B0350

Dilution Factor: 1.0 Cal. Ver. Data Filename: IC11E11C

Concentration Units (pg/L or ng/Kg dry weight): NG/KG %Solids: 16.7

ANALYTE	CONCENTRATION FOUND	DETECTION LIMIT	ION ABUND. RATIO (1)	RRT (1)
2378-TCDD	1.84 UJ		0.82	1.001
12378-PeCDD	2.33 J		1.59	1.000
123478-HxCDD		0.61 U		
123678-HxCDD		1.23 U		
123789-HxCDD		0.52 U		
1234678-HpCDD	3.53 J		1.04	1.000
OCDD	16.52 J		0.93	1.000
2378-TCDF	1.11 J		0.88	1.001
12378-PeCDF	1.24 J		1.68	1.001
23478-PeCDF		0.32 U		
123478-HxCDF		0.50 U		
123678-HxCDF	1.00 J		1.20	1.001
123789-HxCDF	0.44 J		1.21	1.000
234678-HxCDF		0.40 U		
1234678-HpCDF	1.21 J		0.92	1.000
1234789-HpCDF		0.68 U		
OCDF		1.74 UJ		
Total TCDD	1.84 UJ			
Total PeCDD	3.68 J			
Total HxCDD	0.41 J			
Total HpCDD	6.74 J			
Total TCDF	1.63 J			
Total PeCDF	2.08 J			
Total HxCDF	2.31 J			
Total HpCDF	1.21 J			

(1) Contract-required limits for RRTs and ion abundance ratios are specified in Tables 2 and 3A, respectively, Method 1613.

USEPA - ITD

FORM 1B
PCDD/PCDF CONFIRMATION ANALYSIS DATA SHEET

EPA SAMPLE NO.

428090

Lab Name: Pacific Analytical, Inc.   Episode No.:

Contract No.:   SAS No.:   Lab Sample ID: A5801C

Matrix (aqueous/solid/leachate): SOLID   Sample Wt/Vol: 41.0 g or mL: G

Sample Receipt Date: 10/28/94   Initial Calibration Date: 09/10/94

Ext. Date: 11/02/94 Shift: 0800   Instrument ID: VG#5

Analysis Date: 11/23/94 Time: 1112   GC Column ID: Rtx-200

Extract Volume (uL): 20   Sample Data Filename: A5801C

Injection Volume (uL): 1   Blank Data Filename: B0350C

Dilution Factor: 1.0   Cal. Ver. Data Filename: IC11D23C

Concentration Units (pg/L or ng/Kg dry weight): NG/KG   %Solids: 16.7

ANALYTE	CONCENTRATION FOUND	DETECTION LIMIT	ION ABUND. RATIO (1)	RRT (1)
2378-TCDF	0.91		0.77	1.002

(1) Contract-required limits for RRTs and ion abundance ratios are specified in Tables 2 and 3A, respectively, Method 1613.

USEPA - ITD

FORM 2
PCDD/PCDF LABELED COMPOUND AND CLEANUP STANDARD RECOVERIES

EPA SAMPLE NO.

428090

Lab Name: Pacific Analytical, Inc. Episode No.:

Contract No.: SAS No.: Lab Sample ID: A5801

Matrix (aqueous/solid/leachate): SOLID Sample Wt/Vol: 41.0 g or mL: G

Sample Receipt Date: 10/28/94 Initial Calibration Date: 08/27/94

Ext. Date: 11/02/94 Shift: 0800 Instrument ID: VG#5

Analysis Date: 11/11/94 Time: 1630 GC Column ID: DB5 60M

Extract Volume (uL): 20 Sample Data Filename: A5801

Injection Volume (uL): 1 Blank Data Filename: B0350

Dilution Factor: 1.0 Cal. Ver. Data Filename: IC11E11C

Concentration Units (pg/L or ng/Kg dry weight): NG/KG %Solids: 16.7

	SPIKE CONC.	CONC. FOUND	R(%) (1)	ION ABUND. RATIO (2)	RRT (2)
LABELED COMPOUNDS					
13C-2378-TCDD	290	220	76	0.77	1.009
13C-12378-PeCDD	290	400	140	1.62	1.195
13C-123478-HxCDD	290	320	110	1.29	0.988
13C-123678-HxCDD	290	260	90	1.36	0.991
13C-1234678-HpCDD	290	140	48	1.02	1.092
13C-OCDD	580	120	21 *	0.91	1.214
13C-2378-TCDF	290	250	86	0.80	0.979
13C-12378-PeCDF	290	240	83	1.64	1.153
13C-23478-PeCDF	290	470	160	1.60	1.181
13C-123478-HxCDF	290	220	76	0.53	0.966
13C-123678-HxCDF	290	290	100	0.54	0.969
13C-123789-HxCDF	290	220	76	0.53	1.007
13C-234678-HxCDF	290	270	93	0.52	0.985
13C-1234678-HpCDF	290	190	66	0.42	1.059
13C-1234789-HpCDF	290	130	45	0.43	1.108
CLEANUP STANDARD					
37Cl4-2378-TCDD	29	27	93		1.001

* below QC limits of 25 - 150%

USEPA - ITD

FORM 1A
PCDD/PCDF ANALYSIS DATA SHEET
Use for Sample and Blank Results

EPA SAMPLE NO.

BLANK

Lab Name: Pacific Analytical, Inc. Episode No.:

Contract No.: SAS No.: Lab Sample ID: B0350

Matrix (aqueous/solid/leachate): SOLID Sample Wt/Vol: 10.0 g or mL: G

Sample Receipt Date: Initial Calibration Date: 08/27/94

Ext. Date: 11/02/94 Shift: 0800 Instrument ID: VG#5

Analysis Date: 11/11/94 Time: 1341 GC Column ID: DB5 60M

Extract Volume (uL): 20 Sample Data Filename: B0350

Injection Volume (uL): 1 Blank Data Filename: B0350

Dilution Factor: 1.0 Cal. Ver. Data Filename: IC11E11C

Concentration Units (pg/L or ng/Kg dry weight): NG/KG %Solids: 100.0

ANALYTE	CONCENTRATION FOUND	DETECTION LIMIT		ION ABUND. RATIO (1)	RRT (1)
2378-TCDD	0.52 J Sn			0.76	1.001
12378-PeCDD		0.63	U		
123478-HxCDD		0.31	U		
123678-HxCDD		0.35	U		
123789-HxCDD		0.35	U		
1234678-HpCDD		0.63	U		
OCDD		0.95	U		
2378-TCDF		0.37	U		
12378-PeCDF		0.47	U		
23478-PeCDF		0.19	U		
123478-HxCDF		0.31	U		
123678-HxCDF		0.36	U		
123789-HxCDF		0.66	U		
234678-HxCDF		0.33	U		
1234678-HpCDF		0.55	U		
1234789-HpCDF		0.49	U		
OCDF		0.52	U		
Total TCDD	0.52 J Sn				
Total PeCDD		0.63	U		
Total HxCDD		0.35	U		
Total HpCDD		0.63	U		
Total TCDF		0.37	U		
Total PeCDF		0.19	U		
Total HxCDF		0.33	U		
Total HpCDF		0.49	U		

(1) Contract-required limits for RRTs and ion abundance ratios are specified in Tables 2 and 3A, respectively, Method 1613.

USEPA - ITD

FORM 2
PCDD/PCDF LABELED COMPOUND AND
CLEANUP STANDARD RECOVERIES

EPA SAMPLE NO.

BLANK

Lab Name: Pacific Analytical, Inc. Episode No.:

Contract No.: SAS No.: Lab Sample ID: B0350

Matrix (aqueous/solid/leachate): SOLID Sample Wt/Vol: 10.0 g or mL: G

Sample Receipt Date: Initial Calibration Date: 08/27/94

Ext. Date: 11/02/94 Shift: 0800 Instrument ID: VG#5

Analysis Date: 11/11/94 Time: 1341 GC Column ID: DB5 60M

Extract Volume (uL): 20 Sample Data Filename: B0350

Injection Volume (uL): 1 Blank Data Filename: B0350

Dilution Factor: 1.0 Cal. Ver. Data Filename: IC11E11C

Concentration Units (pg/L or ng/Kg dry weight): NG/KG %Solids: 100.0

	SPIKE CONC.	CONC. FOUND	R(%) (1)	ION ABUND. RATIO (2)	RRT (2)
LABELED COMPOUNDS					
13C-2378-TCDD	200	140	70	0.80	1.009
13C-12378-PeCDD	200	140	70	1.63	1.194
13C-123478-HxCDD	200	190	95	1.29	0.988
13C-123678-HxCDD	200	200	100	1.29	0.991
13C-1234678-HpCDD	200	120	60	1.09	1.093
13C-OCDD	400	130	32	0.87	1.214
13C-2378-TCDF	200	150	75	0.81	0.979
13C-12378-PeCDF	200	140	70	1.69	1.152
13C-23478-PeCDF	200	160	80	1.65	1.181
13C-123478-HxCDF	200	150	75	0.53	0.967
13C-123678-HxCDF	200	200	100	0.52	0.969
13C-123789-HxCDF	200	140	70	0.50	1.007
13C-234678-HxCDF	200	190	95	0.52	0.984
13C-1234678-HpCDF	200	140	70	0.45	1.059
13C-1234789-HpCDF	200	130	65	0.41	1.108
CLEANUP STANDARD					
37C14-2378-TCDD	20	15	75		1.001

Date: 04/20/95

To: Dave Serdar

From: Stuart Magoon [redacted]

Subject: Dioxin Blank for Lake Roosevelt sample 428090.

As per our discussion, I had Pacific Analytical re-analyze the method blank associated with Lake Roosevelt sample 428090. 2,3,7,8-TCDD was detected at a very low level in this method blank. Even though the level of 2,3,7,8-TCDD detected in this method blank, was below the level specified for re-analysis, any 2,3,7,8-TCDD detected is cause for concern. Since there is no sample remaining for re-analysis our options, to determine whether or not the 2,3,7,8-TCDD detected in the sample is the result of laboratory contamination or native to the sample, are limited. It was decided to re-analyze the method blank in hopes that the contamination may not have come form the blank extract, but somehow was introduced into the process during the analysis.

Unfortunately 2,3,7,8-TCDD was detected at the same concentration in the re-analysis of the method blank associated with sample 428090. Since 2,3,7,8-TCDD was detected in the sample at a level less than five times that detected in the method blank there is no way to know for certain whether or not the 2,3,7,8-TCDD detected in the sample is native to the sample or the result of contamination. Pacific Analytical did state that it is uncommon to see 2,3,7,8 TCDD in their method blanks.

SM/SM