Polychlorinated Biphenyl and Chlorinated Pesticide Content of Wastewater Suspended Solids Data Summary Report

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## 1. INTRODUCTION

The Fraser River Action Plan, a six-year basin-wide program, was initiated in 1991 to assess the condition of the river (FRAP, 1992). Determining the quality and quantity of wastewater discharges and estimating contaminant loadings was a component of Environmental Protection Branch's contribution to the program.

Crispin et al., 1995 reported that, even today, many years after restrictions of PCB manufacture and use were introduced, cities continue to act as sources of airborne PCBs to surrounding areas. Congeners 28 and 52 dominated the urban air mixture. Coplanar and ortho-substituted PCBs have been reported to represent an important group of halogen containing anthropogenic compounds which occur in trace amounts in sewage sludges (Berset and Holzer, 1996). In most surface waters colloidal associated contaminants (such as PCBs) may be the dominant species (Baker et al., 1986). Non-polar organic contaminants such as polycyclic aromatic hydrocarbons (PAHs) and polychiorinated biphenyls (PCBs), are bound by particles in the water and tend to accumulate in sediments (Kukkonen and Landrum, 1996).

Rogers et al., 1990 reported that in one of the two years of sampling, the presence of Arochlor 1254 in eulachon gonads was detected at a concentration which reportedly might impact upon the spawning success of some fish. The concurrent presence of both PCBs and organochlorine pesticides in various environmental compartments has been frequently documented (Oliver and Niiml, 1988, Hofelt and Shea, 1997).

This report includes the results a limited number of centrifuge-concentrated suspended solids samples which were collected over 1993 to 1995, in an effort to more completely characterize a variety of wastewater sources for PCBs and other organic contaminants (Figure 1 and 2). These contaminants might be expected to be largely found associated with the organically rich and fine suspended matter in some wastewaters.

## 2. SUSPENDED SOLIDS COLLECTION

An Envirodat Sedisamp System II Model 100IL (modified Alfa-Laval MAB103B) continuous-flow centrifuge was used to collect a concentrated suspended solids sample at the point of effluent discharge into the receiving environment. The centrifuge was operated at 4L/minute and long enough to collect a $300-500 \mathrm{~g}$ (wet weight) sample . Centrifuge operation, clean-up procedures and sample handling procedures are reported in detail elsewhere (Mitchell, 1994).

On four occasions, a 50L sample of centrifuge-clarified effluent sample (three pulp mills and one sewage treatment plant) was also collected for PCB analysis.

Figure 1: Location of Wastewater Sources for Suspended Solids



The samples were passed through a solid phase extraction (XAD-2 resin) system described by Sekela et al., 1995.

## 3. ANALYTICAL METHODS FOR SUSPENDED SOLIDS AND XAD-2 COLUMN SAMPLES

### 3.1 Suspended Solids PCBs and Organochlorine Pesticides

The wastewater sources sampled along with the respective PCB/pesticide analytical methods are listed in Table 1 for: Nwood (Northwood Pulp Mill), Canfor (Canadian Forest Products Pulp Mill), QRP (Quesnel River Pulp Mill), Cboo (Cariboo Pulp Mill), Wey (Weyerhaeuser Pulp Mill), Landsdowne (Prince George Central STP), Annacis and Lulu (GVRD STPs) and Clark Drive (GVRD Combined Sewer Overflow to Burrard Inlet).

### 3.1.1 AXYS (Axys Analytical Services) CL-SL-01Ner. 1 (CL-S-01Ner.1)

All samples were spiked with 13C-labelled surrogates (hexachlorobenzene, gamma BHC, p,p'-DDE, p,p'-DDT, mirex, PCB 101, PCB 180 and PCB 209) prior to analysis (Appendix A1). An additional surrogate standard solution containing 13C-labeled coplanar PCBs (PCB 77, PCB 126 and PCB 169) was added to samples for the analysis of coplanar PCBs. Samples were solvent extracted (1:1 dichloromethane:methanol followed by dichloromethane) on a shaker table and the final extracts were cleaned up and fractionated into two fractions (F1+F2 and F3) on a Florisil column. The first fraction (called F1+F2) was split gravimetrically and one half analyzed by HRGC/LRMS for PCBs as individual congeners, Aroclors and non-polar to moderately polar chlorinated pesticides. The other half of the F1+F2 fraction underwent additional cleanup and an additional GC/MS run for the analysis of coplanar PCBs. The F3 fraction was analyzed for the most polar chlorinated pesticides by HRGC/ECD.

### 3.1.2 AXYS (Axys Analytical Services) CL-S-01/Ner. 2

All samples were spiked with surrogate standards (tetrchloro-m-xylene, 13Clabeled PCBs $(77,126,169,209)$ and per-deuterated alpha-endosulphan prior to analysis (Appendix A2). Samples were solvent extracted with 1:1 dichloromethane:methanol followed by dichloromethane. The final extracts were separated into three fractions on a Florisil column. A combined F1+F2 fraction was analyzed by GC/MS for PCBs, PCB congeners, and non-polar and moderately polar chlorinated pesticides. An additional chromatographic cleanup step of F1+F2 on a carbon/Celite 545 column isolated the coplanar PCBs which were analyzed by HRGC/HRMS. The F3 fraction was analyzed for the most polar chlorinated pesticides by GC/ECD.

### 3.2 XAD-2 Column PCBs .

The centrifuge-clarified 50 L samples were analyzed by AXYS using method CL-C-05Ner.1. The XAD-2 samples were collected at Nwood (Northwood Pulp Mill), Cboo (Cariboo Pulp Mill), Wey (Weyerhaeuser Pulp Mill) and Annacis (GVRD STP).

Table 1: Wastewater Sources and PCB/Pesticide Methods for Suspended Solids

| Sample Source | Method | Sample Source | Method | Sample Source | Method |
| :---: | :---: | :---: | :---: | :---: | :---: |
| PULP MILLS |  | MUNICIPAL SEWAGE** |  | COMBINED SEWER |  |
| Nwood |  |  |  | OVERFLOW** |  |
| $\begin{gathered} \text { 04/11/93 } \\ 25 / 10 / 94^{*} \end{gathered}$ | CL-S-01/Ver. 1 | $\begin{aligned} & \text { Landsdowne } \\ & 03 / 11 / 93 \end{aligned}$ | CL-SL-01/Ver. 1 | Clark Drive | CL-SL-01Ner 1 |
| Canfor | CL-SL-01/Ner. 1 | Annacis 03/03/94* | CL-SL-01/Ver. 1 |  | CL-SL-OTVE. 1 |
| $\begin{aligned} & 05 / 11 / 93 \\ & 23 / 11 / 93 \end{aligned}$ | CL-SL-01/Ner. 1 | $\begin{aligned} & \text { O3/03/94** } \\ & 22 / 08 / 95 \end{aligned}$ | CL-S-01Ner. 2 |  |  |
| QRP |  | 21/11/95 | CL-S-01/Ver. 2 |  |  |
| 01/11/93 | CL-SL-01/Ve | Lulu |  |  |  |
| $\begin{array}{\|l\|} \hline \text { Cboo } \\ 02 / 11 / 93^{*} \end{array}$ | CL-SL-01/Ver. 1 | 12/05/94 |  |  |  |
| Wey 08/11/93* | CL-SL-01/Ver. 1 |  |  |  |  |

* 50 L centrifuge-clarified effluent sample also collected using XAD-2 2 for PCB analysis. ** Pesticides in addition to PCBs analyzed.


## 4. RESULTS

The individual sample PCB results (Aroclor, coplanar, congener) are reported in Appendix B1 and the organochlorine pesticide results in Appendix B2. The PCB surrogate recovery results for the individual wastewater samples are reported in Appendix C1. The PCB spiked matrix sample recoveries are reported in Appendix C2. Sample surrogate recoveries for the organochlorine pesticides are reported in Appendix C3 and for the spiked samples in Appendix C4. The surrogate recoveries and spiked matrix samples did not indicate anything unusual, other than a low d4-alpha-Endosulphan recovery of $33 \%$ for the Annacis November 21, 1995 sample (Appendix C3).

The data represent surrogate recovery corrected results but which have not been corrected for the procedural blank results. The procedural blank results are also included with Appendix B1 and B2. There was nothing in the procedural blank results that indicated that any of the sample results would be seriously affected and required correction.

The suspended solids contaminant loading was calculated (dry weight contaminant concentration $\mathbf{x}$ the daily loading of suspended solids) and is presented as either ug/d or $\mathrm{mg} / \mathrm{d}$. The whole effluent loading was calculated from the effluent concentration and the daily effluent volume discharged. For the CSO results, the loading is expressed on a daily ( 24 h ) basis for comparative purposes only. The actual CSO annual loading, which depends upon the actual number of hours of discharge per year and varies annually with rainfall, would be the most appropriate comparison with the annual loading of the other wastewaters. This would of course require a large enough data set to adequately represent the whole year, which the results of this report do not.

## 5. DISCUSSION

The highly organic nature of the suspended solids discharged from the wastewaters tested is clearly evident and ranged from a low of $\sim 25 \%$ for the CSO to as high $\sim 50 \%$ for the pulp mills (Table 2). These discharges represent major sources of particulate carbon into the Fraser system.

Table 2: Daily Loading of Carbon Associated with Suspended Solids*


[^0]
### 5.1 Polychlorinated Biphenyls

### 5.1.1 PCB Aroclors and Coplanars

For the three Aroclors tested, with GC-LRMS, none were detected in the pulp mill solids. In the one case where GC-HRMS was utilized (Nwood, 25/10/94), Aroclor 1242, 1254 and 1260 were detected and loading were calculated as 127 $\mathrm{mg} / \mathrm{d}, 70 \mathrm{mg} / \mathrm{d}$ and $12 \mathrm{mg} / \mathrm{d}$ respectively (Table 3). For the municipal wastewaters, Aroclors 1242 and 1254 were consistently present using GCLRMS. For a major STP such as Annacis, the average ( $n=3$ ) daily loading associated with the suspended solids fraction was estimated to be in the order of $1070 \mathrm{mg} / \mathrm{d}$ Aroclor 1242 and $1130 \mathrm{mg} / \mathrm{d}$ Arolcor 1254. All three Arolcors were detected in the combined sewer overflow (CSO) sample, with a 24-h discharge loading of greater than $1000 \mathrm{mg} / \mathrm{d}$ for each contaminant.

For the one case where a suspended solids sample and a 50 L centrifugeclarified municipal sewage effluent sample for solid phase extraction were collected concurrently, Aroclor 1242 was detected in both fractions. The total Aroclor 1242 loading (solids fraction + XAD-2 fraction) was estimated to be approximately $1250 \mathrm{mg} / \mathrm{d}$, of which $47 \%$ was associated with the XAD-2 fraction. Aroclor 1254 was not detected in the XAD-2 fraction and in this case appeared to be totally associated with the suspended solids fraction (Table 3).

Coplanar \#77 was detected in five of the six bleached kraft pulp mill suspended solids samples but, was not identified in the single chemical-thermo mechanical mill sample (QRP). The loading (range: $69 \mathrm{ug} / \mathrm{d}$ to $532 \mathrm{ug} / \mathrm{d}$ ) was similar to that of the main STP serving the City of Prince George (307 ug/d) (Table 3). For a major STP such as Annacis, the average ( $n=3$ ) daily loading was estimated to be in the order of $3300 \mathrm{ug} / \mathrm{d}$. Coplanar \#77 was detected in both the suspended solids and XAD-2 samples at two of the pulp mills with approximately $70 \%$ of the total loading associated with the solids. For the Annacis STP, in the one case were both fractions were analyzed, approximately $86 \%$ of the coplanar \#77 loading was associated with the solids.

Coplanar \#126 detected in both the pulp mill and municipal STP solids fraction but not in the XAD-2 fraction. The individual pulp mill loadings were similar to the Prince George STP, whereas, the Annacis STP loading was an order of magnitude greater. Coplanar \#169 was only detected in the municipal wastewater samples (Table 3).

Table 3: Estimated Loading of Aroclors 1242, 1254, and 1260 and Coplanars \#77, \#126 and \# 169 Associated with Suspended Solids from Various Wastewater Sources

| TSS Source/PCB | Aroclor 1242 (mg/d) | Aroclor 1254 (mg/d) | $\begin{gathered} \text { Aroclor } 1260 \\ (\mathrm{mg} / \mathrm{d}) \end{gathered}$ | $\begin{aligned} & \hline \text { Coplanar \#77 } \\ & \text { (ug/d). } \end{aligned}$ | $\begin{gathered} \text { Coplanar\#126 } \\ \text { (ug/d) } \\ \hline \end{gathered}$ | $\begin{gathered} \hline \begin{array}{c} \text { Coplanar \#169 } \\ \text { (ug/d) } \end{array} \\ \hline \hline \end{gathered}$ |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| PULP MILLS |  |  |  |  |  |  |
| Nwood | $<$ | < | $<$ | < | $<$ | < |
|  |  |  |  |  | \%\%\%\%** |  |
| \#. $_{25 / 10 / 94^{*}}$ | 127 | 70 | 12 | 394 | < | < |
| Canfor | $<$ | $<$ | $<$ | 448 | 49 | $<$ |
| $\begin{aligned} & 05 / 11 / 93 \\ & 23 / 11 / 93 \end{aligned}$ | $<$ | $<$ | $<$ | 373 | 48 | < |
| QRP 01/11/93 | $<$ | $<$ | $<$ | $<$ | $<$ | $<$ |
| Cboo | $<$ | < | < | 69 | 11 | $<$ |
|  | \%\%\%\%凶\%\%\% |  |  |  |  | \% $\sim$, |
| Wey | < | < | < | 532 | 53 | < |
| 08/11/93 |  |  |  |  |  |  |
| MUNICIPAL SEWAGE |  |  |  |  |  |  |
| Landsdowne 03/11/93 | 74 | 128 | $<$ | 307 | 64 | 4 |
| Annacis |  |  |  | 3039 | 608 | 43 |
| 03/03/94 |  | \%\%\%\% |  |  |  |  |
| $\underset{22 / 08 / 95}{204 \%}$ | 1803 | 1494 | 464 | 5152 . | 361 | 36 |
| $\begin{aligned} & 22 / 08 / 95 \\ & 21 / 11 / 95 \end{aligned}$ | 748 | 810 | 287 | 1745 | 231 | 31 |
| Lulu 12/05/94 | 503 | 373 | $<$ | 838 | NDR | < |
| COMBINED SEWER OVERFLOW**** |  |  |  |  |  |  |
| 06/04/94 | 1356 | 1971 | 1484 | 5299 | 721 | 360 |

* Arociors analyzed by GC-HRMS on this instance, otherwise all other Aroclor analyses are GC-LRMS


### 5.1.2 PCB Congeners

Congeners were not generally detected in the pulp mill solids samples using GCLRMS but were evident in the one sample tested by GC-HRMS (Table 4). Due to some differences in the reported congeners, depending on the analytical method, an identical comparison between the various wastewaters wasn't possible. However, for some of those congeners that were reported consistently in all of the samples, the municipal discharges appear to be an obvious source relative to pulp mills (Table 4).

### 5.2 Organochlorine Pesticides - Municipal Wastewaters

Three organochlorine pesticides were common to all six samples and included hexachchlorobenzene, gamma HCH and p,p'-DDE (Table 5). Alphaendosulphan, dieldrin and methoxychlor were identified in five of the six samples. In terms of the number of positive identifications, the largest number of pesticides were identified in the CSO sample.

## 6. SUMMARY

The results of this study demonstrate the presence of a variety of contaminants associated with the suspended solids fraction of various wastewater sources. The suspended solids from these sources have also been shown to be high in organic content. Although the data base is not large, municipal wastewater's appear to be a major source of PCBs discharged to the receiving environment, relative to pulp mills.

Coplanar \#77 appeared to be ubiquitous to the majority of the wastewater's sampled. However, the origin of contaminants such as coplanar \#77 into the various wastewater streams is not easily ascertained. Brown et al., 1995 concluded that based on available PCB congener distribution data, that combustion processes rather than Aroclor releases or Aroclor photolyses, are the most likely source of environmental coplanar PCBs.

The initial dispersion of these contaminants in the receiving environment will be largely influenced by the settleability characteristics of the solids. The shortest path and thus their initial availability to aquatic organisms would, likely be through organisms that filter their food sources from the water column.
Table 4: Estimated Loading of Select PCB Congeners Associated with Suspended Solids from Various Wastewater Sources


[^1]** NDR values not included. na $=$ not analyzed.

Table 5 : Estimated Loading of Organochlorine Pesticides Associated with Suspended Solids from Municipal Wastewater Sources

| Source/Pesticide (mg/d) | $\begin{gathered} \hline \text { Landsdowne } \\ 03 / 11 / 93 \\ \hline \end{gathered}$ | $\begin{aligned} & \text { Annacis } \\ & 03 / 03 / 94 \end{aligned}$ | $\begin{aligned} & \hline \text { Annacis } \\ & 22 / 08 / 95 \end{aligned}$ | Annacis 21/11/95 | $\begin{gathered} \text { Lulu } \\ 12 / 05 / 94 \end{gathered}$ | $\begin{gathered} \text { Clark CSO } \\ 06 / 04 / 94 \end{gathered}$ |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: |
|  |  | \%\% | \% |  | 尔的 | 61 |
| alpha HCH |  |  |  | 65 | 3.8 | 14 |
| beta HCH |  |  | 149 |  | 59 | 59 |
| gamma HCH |  |  |  | \%\%\%3V\%\% |  |  |
| Heptachlor |  |  |  |  |  |  |
| Aldrin |  |  |  |  |  |  |
| Oxychlordane |  |  |  |  | 10. | 22 |
| trans-Chlordane |  |  | 36 | 34 | 15 - | 86 |
| cis-Chlordane |  |  | 26 | 26 | 19 | 158 |
| o, ${ }^{\prime}$ '-DDE |  |  | 26 |  |  | 38 |
| p, p'-DDE |  |  |  | \%\% |  | \% \% |
| trans-Nonachlor |  |  | 23 | 26 | 55 | 63 |
| cis-Nonachlor |  |  |  |  |  | 29 |
| o,p'-DDD |  |  | 59 |  | 130 | 27 |
| P. P'-DDD |  |  |  |  |  | 196 |
| o,p'-DDT |  |  |  |  |  |  |
| p,p'-DDT |  |  |  | 196 | , | 1014 20 |
| Mirex |  |  |  |  |  |  |
| Heptachlor Epoxide alpha-Endosulphan | 0.6 | 18 | 103 |  | 29 | 56 |
| Dieldrin | 4.4 | 63 | 34 |  | 41 | 122 |
| Endrin |  |  |  |  |  |  |
| Methoxychlor | 7.8 | 144 | 165 |  | 628 | 406 |

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## APPENDIX A1

## ANALYSIS OF POLYCHLORINATED BIPHENYLS AND CHLORINATED PESTICIDES IN SLUDGE SAMPLES

All samples were spiked with 13C-labelled surrogates (hexachlorobenzene, gamma BHC, p,p'-DDE, p,p'-DDT, mirex, PCB 101, PCB 180 and PCB 209) prior to analysis. An additional surrogate standard solution containing 13C-labelled coplanar PCBs (PCB 77, PCB 126 and PCB 169) was added to samples for the analysis of coplanar PCBs. Sludge samples were solvent extracted on a shaker table and the final extracts were cleaned up and fractionated into two fractions (F1+F2 and F3) on a Florisil column. The first fraction (called F1+F2) was split gravimetrically and one half analyzed by HRGC/LRMS for PCBs as individual congeners, Aroclors and non-polar to moderately polar chlorinated pesticides. The other half of the F1+F2 fraction underwent additional column cleanup and an additional GC/MS run for the analysis of coplanar PCBs. The F3 fraction was analyzed for the most polar chlorinated pesticides by HRGC/ECD.

## 1. Extraction

A homogenized subsample (approximately 10 g wet) was spiked with aliquots of the surrogate standard solutions. Samples were extracted by shaking first with dichloromethane:methanol and then with dichloromethane. The extracts were decanted, combined, washed with solvent extracted water and dried over anhydrous sodium sulphate. The extract was concentrated to a small volume by rotary evaporation prior to column cleanup.

## 2. Column Chromatography For Pesticides and PCBs

The concentrated extract was applied to a Florisil column for cleanup and fractionation. If The first fraction was eluted with hexane, followed by 85:15 hexane:dichloromethane and the eluates combined (F1+F2). The F1+F2 extract was gravimetrically split in half. One half was prepared for analysis by high resolution gas chromatography with low resolution (quadrupole) mass spectrometric analysis for PCBs, pesticides and individual PCB congeners. The other half of the F1+F2 fraction underwent cleanup procedures for coplanar PCBs.

One half of the F1+F2 fraction and the F3 fraction were each concentrated to a small volume and transferred to microvials. An aliquot of recovery standard solution ( ${ }^{13}$ C-labelled PCB 153) was added to each prior to instrumental analysis.

## 3. $\mathrm{GC} / \mathrm{MS}$ Analysis Of PCBs and Pesticides

The F1+F2 fraction was analyzed for PCB congeners, Aroclors and non-polar to moderately polar chlorinated pesticides using a Finnigan INCOS 50 mass spectrometer equipped with a Varian 3400 GC, a CTC autosampler and a DG 10 data system running Incos 50 (Rev 9) software. Chromatographic separation of PCBs and pesticides was achieved with a 60 metre DB-5 chromatography column ( 0.25 mm i.d., $0.10 \mu \mathrm{~m}$ film thickness). The MS was operated in the El mode at unit mass resolution and in the MID (Multiple lon Detection) mode acquiring at least two characteristic -ions for each target analyte and surrogate standard.
4. Extract Cleanup for the Analysis of Coplanar PCBs

The second half of the $F 1+F 2$ fraction was applied to a carbon/celite column for cleanup. The carbon/celite , column was eluted with 1:1 cyclohexane:dichloromethane (discarded) followed by ethylacetate (discarded). The coplanar PCBs were eluted with 1:1 toluene:ethylacetate (retained). This fraction was evaporated to just dryness and redissolved in hexane.

The extract was applied to a basic alumina column (10 g) for further clean up. The alumina column was eluted with hexane (discarded) followed by 5:95 dichloromethane:hexane (retained). The extract was concentrated, transferred to a microvial and an aliquot of recovery standard ( ${ }^{13}$-labelled PCB 153) added prior to GC/MS analysis for coplanar PCBs.

## 5. Instrumental Analysis for Coplanar PCBs

At Axys' convenience the extracts were analyzed on a VG 70 SE high resolution mass spectrometer system. The mass spectrometer was equipped with a Hewlett Packard gas chromatograph, a CTC autosampler and a VAX workstation. A 60 metre DB-5 chromatography column ( 0.25 mm i.d., $0.25 \mu \mathrm{~m}$ film thickness), used for the GC separation, was coupled to the MS source. The MS was operated at unit mass resolution in the selected ion recording (SIR) mode, acquiring two characteristic ions for each target analyte and surrogate standard.
6. GC/ECD Analysis for Polar Pesticides (F3)

Polar chlorinated pesticides in F3 were analyzed by HRGC7ECD using a Hewlett Packard 5890 gas chromatograph, equipped with a 60 metre ( 0.25 mm id, $0.10 \mu \mathrm{~m}$ film thickness) DB5 Durabond fused silica capillary column, a ${ }^{63} \mathrm{Ni}$ electron capture detector and a CTC autosampler.

## APPENDIX AZ

ANALYSIS OF PESTICIDES, PCBs, PCB CONGENERS AND COPLANAR PCBS

## Summary

All samples were spiked with surrogate standards (tetrachloro-mxylene, ${ }^{13} \mathrm{C}$-labelled PCBs (77, 126, 169, 209) and per-deuterated. alpha-endosulphan) prior to analysis. Samples were solvent extracted. The final extracts were separated into three fractions on a Florisil column. A combined F1+F2 fraction was analyzed by GC/MS for PCBS, PCB congeners, and non-polar and moderately polar chlorinated pesticides. An additional chromatographic cleanup step of F1+F2 on a carbon/Celite 545 column isolated the coplanar PCBS which were analyzed by HRGC/HRMS. The F3 fraction was analyzed for the most polar chlorinated pesticides by GC/ECD.

## 1. EXTRACTION PROCEDURE

A homogenized sample was spiked with an aliquot of surrogate standards. The sample was extracted by shaking with 1:1 dichloromethane:methanol followed by dichloromethane. The extracts were combined, washed with solvent-extracted distilled water and dried over anhydrous sodium sulphate. The extract was concentrated and activated copper added to.remove sulphur. The extract was ready for cleanup and separation on a Florisil column.

## 2. CHROMATOGRAPHIC CLEANUP PROCEDURES

The extract was loaded onto a Florisil column which was eluted with hexane (F1) followed by 15:85 dichloromethane:hexane (F2). The eluates were combined (F1+F2, retained). The column was eluted with $1: 1$ dichloromethane:hexane (F3, retained). Each fraction was concentrated, transferred to " an autosampler vial and recovery standards added (4,4'-dibromooctafluorobiphenyl and PCB 204 to $\mathrm{F} 1+\mathrm{F} 2,{ }^{13} \mathrm{C}$-labelled PCB 153 to F 3 ). The extracts were ready for instrumental analysis.

The extracts required additional chromatographic cleanup on a gel permeation column to remove interferences observed in the GC/MS and GC/ECD chromatograms.

## Cleanup of Coplanar PCBs

After the GC/MS analysis of F1+F2, the extract was loaded onto a carbon/Celite column and eluted with $1: 1$ cyclohexane:dichloromethane (discarded) followed by ethyl acetate (discarded). The column was then eluted with 1:1 toluene: ethyl acetate (retained). The extract was concentrated and applied to an alumina column. The column was eluted with hexane (discarded) followed by $1: 1$ dichloromethane:hexane (retained), Recovery standard ( ${ }^{13} \mathrm{C}-$ PCB 153) was added. The extract was ready for analysis by HRGC/HRMS.

## 3. INSTRUMENTAL ANALYSIS

GC/MS
GC/MS analysis of F1+F2 for PCB/pesticides was carried out using a Finnigan INCOS 50 mass spectrometer equipped with a Varian 3400 GC, a CTC autosampler and a DG 10 data system running Incos 50 (Rev 11) software. The MS was operated at unit mass resolution in the MID mode acquiring two characteristic ions for each target analyte and surrogate standard. Chromatographic separation of pesticides and PCB congeners was achieved with a DB-5 chromatography column ( 60 $\mathrm{m}, 0.25 \mathrm{~mm}$ i.d., $0.10 \mu \mathrm{~m}$ film thickness). A splitless/split injection sequence was used.

## GC/ECD Analysis

Chlorinated pesticides in F3 were analyzed using a Hewlett Packard 5890 gas chromatograph, with a ${ }^{63} \mathrm{Ni}$ electron capture detector and a $60 \mathrm{~m} \times 0.25 \mathrm{~mm}, 0.10 \mu \mathrm{~m}$ film DB-5 Durabond Fused Silica capillary column.

HRGC/HRMS Analysis of Coplanar PCBs
HRGC/HRMS analysis of coplanar PCBs was carried out on a VG 70 SE high resolution mass spectrometer equipped with a Hewlett Packard 5890 gas chromatograph, a CTC autosampler and a VAX work station. Data were acquired in the voltage selected Ion Mode (SIM) to enhance sensitivity. Chromatographic separation was achieved with DB-5 capillary chromatography column $(60 \mathrm{~m}, ~ 0.25 \mathrm{~mm}$ i.d $\times 0.1 \mu \mathrm{~m}$ film thickness). A splitless/split injection sequence was used.

## APPENDIX B1


ss = sample size; $\mathrm{pb}=$ procedural blank; NDR $=$ peak detected but did not meet quantification criteria

ss = sample size; $\mathrm{pb}=$ procedural blank; NOR = peak detected but did not meet quantification criteria


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[^2]Appendix: 81 QRP - NOVEMBER 01 1993-BIOSOLIDS PCB RESULT

$\boldsymbol{s s}=$ sample size; $\mathrm{pb}=$ procedural blank; NDR $=$ peak detected but did not meet quantification criteria

ss = sample size; pb = procedural blank; NDR = peak detected but did nọt meet quantification criteria


| Appendox: $\mathbf{B 1}$ | DOWNE | OVEMBE | - BIOS | CB RESL |  |  |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
|  | log Kow | Sofids <br> ( $\mathrm{ng} / \mathrm{g}$ ) | $\begin{aligned} & \text { Solids } \\ & (\mathrm{mg} / \mathrm{d}) \end{aligned}$ |  |  |  |  |
| Aroctor 1242 . | 4.2-5.8 | 87 | 74 |  | . | . |  |
| Aroctor 1254 | 6.1-6.8 | 150 | 128 |  |  |  |  |
| Aroctor 1260 | 6.3-7.5 | $<76$ | - |  |  |  |  |
|  | . | ( $\mathrm{p} / \mathrm{g}$ ) | ( $\mathrm{m} / \mathrm{d}$ ) |  |  |  |  |
|  | 6.5 | 360 | 307 |  |  |  |  |
| PCB *128 (pb 0.8) |  | 75 | 64 |  |  |  |  |
| PCB \# 169 ( 960.53 ) |  | 5.1 | 4 |  |  |  |  |
|  | log Kow | ( $\mathrm{n} / \mathrm{/g}$ ) | (mg/d) | \% | $\log$ Kow | ( g g 9 ) | (mp/d) |
| 8/5 |  | NOR 9.7 |  | 149 |  | 11 | $\theta$ |
| . 15 | 5.3 | NDR 46 |  | 134 | 7.3 | 4:0 |  |
| 19 |  | $<2.3$ |  | 131 |  | 4.0 |  |
| 18 | 5.6 | 4.1 | 4 | 146 |  | 4.1 |  |
| 17 |  | $<2.3$ |  | 153 | 6.9 | - 6.40 | 5 |
| $24 / 27$ |  | 2.3 | 2 | 141 |  | 2.8 |  |
| 18132 |  | 3 | 3 | 130 |  | $<2.5$ |  |
| 26 |  | $<2.2$ |  | 137 |  | 2.5 |  |
| 25. |  | $<2.2$ |  | 138/163/164 |  | 6.5 | 6 |
| 31/28 |  | 12 | 10 | 158 |  | $<2.5$ |  |
| 33 | 5.8 | 5.1 | 4 | 129 | 7.3 | - <2.5 |  |
| 22 |  | 2.3 | 2 | 128 | 7.0 | < 5.6 |  |
| 45 |  | $<2.1$ |  | 156 |  | <6.0 |  |
| 46. |  | <2.1 |  | 157 |  | $<6.0$ |  |
| 52 |  | 11 | 9 | 179 |  | $<2.2$ |  |
| 49 | - | 4.3 | 4 | 176 |  | $<2.2$ |  |
| 47/48 |  | 2.4 | 2 | - 878 |  | $<2.2$ |  |
| 44 | 6 | 7.8 | 7 | 175 |  | $<2.2$ |  |
| 42 |  | $\langle .6$ |  | 187/182 |  | $<2.2$ | - |
| 41/71/64 |  | 8.2 | 7 | 183 | 7.0 | -4.0 |  |
| 40 | 5.6 | < 3.2 |  | 185 |  | $<4.0$ |  |
| 74 |  | 4.1 | 4 | 174 |  | <4.0 |  |
| $70 \times 76$ |  | 11 | 9 | 177 |  | 4.0 |  |
| 65 | 5.8 | 4.6 | 4 | 171 | 8.7 | 4.0 |  |
| 56/60 |  | 2.8 | 2 | - 172 |  | 4.3 |  |
| 95 |  | 12 | 10 | 180 |  | 4.3 | - |
| 01 |  | $\cdots$ - 2.7 |  | 193 |  | 4.3 |  |
| 8489 |  | 5.7 | 5 | 191 |  | $<4.3$ |  |
| 001701 . |  | 16 | 14. | 170/180 |  | 4.3 |  |
| $69$ |  | 5.5 | 5 | 189 |  | $<4.3$ |  |
| $83$ |  | $<2.8$ |  | 201 |  | 4.4 |  |
| 97 |  | 4.2 | 4 | 197 |  | $<7.2$ |  |
| 87 | 6.5 | 6.8 | 6 | 188 |  | 4.2 |  |
| 85 |  | <2.8 |  | 199 |  | $<7.2$ |  |
| $\because 110$ |  | 16. | 14 | 188/203 |  | \$.1 |  |
| 107 |  | $<2.8$ |  | 195 |  | $<5.1$ |  |
| 118 (pb 0.18) |  | 11 | 9 | 194 | 7.4 | $<10$ | . |
| 114 |  | - $<2.9$ | . | 205 |  | $<10$ |  |
| 105 | 6 | 3.7 | 3 | 208 | 8.2 | 4.1 |  |
| 138 |  | $<4.0$ |  | 207 | 7.5 | 4.1 |  |
| $151{ }^{\circ}$ |  | - 4.0 |  | - 200 | 7.2 | 4.1 |  |
| 144/135 |  |  |  | 209 | 8.3 | $<3.7$ |  |

ss = sample size; $\mathrm{pb}=$ procedural blank; NDR = peak detected but did not meet quantification criteria

ss = sample size; $\mathrm{pb}=$ procedural blank, NDR $=$ peak detected but did not meet quantification criteria

ss = sample size; $\mathrm{pb}=$ procedural blank; NDR = peak detected but did nọt meet quantification criteria

| Appendix : 81. | ANNACIS STP | MBER 211 | IOSOLIL | CB RESULTS |  |  |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
|  | log Kow | Solids ( $\mathrm{n} 9 \mathrm{P} / \mathrm{g}$ ) | $\begin{aligned} & \text { Solids } \\ & \text { (mg/d) } \end{aligned}$ |  |  | $\cdots$ |  |
| Aroctor 1242 | - 4.2-5.8 | 24 | 748 |  |  |  |  |
| Arocter 1254 | 6.1-6.8 | 32 | 810 | . |  |  |  |
| Arodor 1260 | 6.3-7.5 | 9.2 | 287 |  |  |  |  |
|  |  | ( $\mathrm{g} / \mathrm{g}$ ) | (ug/d) |  | , | , |  |
| PC8 1777 - pb 2.8 | 6.5 | 56 | 1745 |  |  |  |  |
| PCB \#126 |  | 7.4 | 231 |  |  |  |  |
| PCB $\# 169$ pb NOR 0.6i |  | 1 | 31 |  |  |  |  |
|  | log Kow | ( $\mathrm{O} / \mathrm{s}$ ) | (mg/d) |  | $\log$ Kow | ( $\mathrm{ng} / \mathrm{g}$ ) | (mg/d) |
| 18 | 5.6 | 1.2 | 37 | 105 | 6 | 1.1 | 34 |
| 15/17 |  | 1.5 | 47 | 141/178 |  | 0.44 | 14 |
| 54 | 5.48 | <0.1 |  | 137 |  | $\infty$ ¢ 10 |  |
| 31 |  | 1.5 | 47 | 138/158 |  | 3.00 | 93 |
| $28 . \mathrm{pb}$ NOR 0.08 | 5.8 | 1.5 | 47 | 128/128 |  | 40.18 |  |
| 52 |  | 2 | 62 | 187/182/159 |  | 0.49 | 15 |
| 48 | 6.1 | 1.1 | 34 | 183 |  | 40.19 |  |
| 44 | 8 | 8.7 | 53 | 128 | 7 | 0.3 | 8 |
| 401103 |  | NOR 0.23 |  | 185 | 7 | $<0.16$ |  |
| 61/9474 | . | 1.1 | 34 | \$74181 |  | 0.35 | 11 |
| 88/80\%95 |  | 4.2 | 131 | 202/171/156 |  | 0.48 | 15 |
| 121. |  | $\infty .12$ |  | 173/201 |  | <0.18 |  |
| 56/60 |  | 1.2 | 37 | 180 |  | 0.95 | 30 |
| $90 / 101$ |  | 3 | 93 | 191 |  | <0.18 |  |
| 88/97 |  | 2.1 | 65 | 170 |  | 0.61 | 19 |
| 87 |  | 1.8 | 50 | 199 |  | <0.24 |  |
| 77/154/110 | 6.5 | $\cdots 4.1$ | 128. | 203/196 |  | -0.20 |  |
| 151 |  | NDR 0.44 |  | 189 |  | $<0.22$ |  |
| 135/144 | - | NOR 0.45 |  | 208/185 |  | $<0.23$ |  |
| 149 |  | 2.4 | 65 | 207 | 7.52 | <0.38 |  |
| 118 |  | 2.6 | 81 | 194 | 7.4 | $<0.38$ |  |
| 143 |  | - 0.22 |  | - 205 |  | $<0.38$ | , |
| 114 |  | 40.22 |  | 206 | - 7.2 | <0,38 | $\cdots$ |
| 132/153 |  | 2.3 | 72 |  |  |  |  |


$s s=$ sample size; $p \mathrm{p}=$ procedural blank; NDR = peak detected but did not meet quantification criteria

| Appendix: B1 | K DRIVE | 61994 - | CB | TS |  |  |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
|  | log Kow | Sollds <br> ( $\mathrm{ng} / \mathrm{g}$ ) | Solids (mg/d) |  |  |  |  |
| Arocior 1242 | 4.2-5.8 | 64 | 1356 |  |  |  |  |
| Aroclor 1254 | 6.9-6.8 | 83 | 1971 | - |  |  |  |
| Aroctor 1260 | 6.3 - 7.5 | 70 | 1484 |  |  |  |  |
|  | . | ( $\mathrm{pg} / \mathrm{g}$ ) | ( $4 \mathrm{~g} / \mathrm{d}$ ) |  |  |  | - |
| PCB 517 | 6.5 | 250 | 5299 |  |  |  |  |
| PCB $\$ 126$ |  | 34 | 721 | : |  |  |  |
| PCB \# 169 |  | 17 | 360 |  |  |  |  |
|  | $\log$ Kow | (ng/0) | $(\mathrm{mg} / \mathrm{d})$ | K K k k | log Kow | ( $\mathrm{ng} / \mathrm{g}$ ) | (mg/d) |
| 18 | 5.6 | 4.4 | 87 | 105 | 6 | 2.9 | 61 |
| 15117 |  | 4.2 | 69 | 141/179 |  | 2.50 | 53 |
| 54 | 5.48 | $<1.4$ |  | - 137 |  | 0.87 | 18 |
| 31 |  | 3.4 | 72 | $138 / 158$ |  | 13.00 | 276 |
| 28 | - 5.8 | 4 | 85 | 1291126 |  | 8.50 | 32 |
| 52 |  | 6.5 | 138 | 187/182/159 |  | 4.6 | 97 |
| 49 | 6.1 | 3.5 | 74 | 183 |  | 2.3 | 49 |
| 44 | 6 | 3.2 | 68 | 128 | 7 | 2.8 | 59 |
| 401403 |  | $\infty$ - 67 |  | 185 | 7 | -0.57 |  |
| 81/94/74 |  | 4.7 | 36 | 1741181 |  | 2.8 | 59 |
| O6R80/95 |  | 7.2 | 153 | 202/171/156 |  | 1.8 | 38 |
| 121 |  | $<0.86$ |  | 173/201 |  | $<0.7$ |  |
| 56/60 |  | 2.8 | 59 | 180 |  | 8 | 170 |
| 90101 |  | 7.8 | 165 | 191 | . | $<0.7$ |  |
| 86797 |  | 2.5 | 53 | 170 |  | 3.5 | 74 |
| 87 |  | 4.4 | 93 | 199 | . | 1.8 | 40 |
| 77/154/110 | 6.5 | 13 | 276 | 2031186 |  | 1.5 | 32 |
| 151 |  | 3.4 | 72 | 189 |  | $\infty 0.76$ |  |
| $135 / 144$ | $\cdots$ | 4.6 | 87 | 208/195 |  | $<0.54$ |  |
| 149 |  | 11 | 233 | 207 | 7.52 | 0.76 |  |
| 118 |  | 12 | 254 | 194 | 7.4 | 1.8 | 38 |
| 143 |  | 4.2 |  | - 205 |  | $<0.76$ |  |
| 114 |  | 1.4 | 30 | 206 | 7.2 | 3.2 | 68 |
| 132153 |  | 12 | 254 |  |  |  |  |

$\square$

mosis

| Appendix B2: | Landsdowne | - November 31 | inated Pesticides |
| :---: | :---: | :---: | :---: |
| Pesticide |  | (ng/g dry wt.) | Loading (mg/d) |
| hexachlorobenzene | (pb 0.11) | 4 | 3.4 |
| alpha HCH |  | $<11$ |  |
| beta HCH |  | <22 |  |
| gamma HCH |  | 190 | 162.3 |
| Heptachlor |  | <18 |  |
| Aldrin |  | <5.8 |  |
| Oxychiordane |  | <32 |  |
| trans-Chlordane |  | <3.3 |  |
| cis-Chlordane |  | <3.6 |  |
| o, ${ }^{\prime}$ '-DDE |  | NDR 9.8 |  |
| P, $\mathrm{p}^{\prime}$-DDE | (pb 0.16) | 26 | 22.2 |
| trans-Nonachlor |  | <3.4 |  |
| cis-Nonachlor |  | <4.5 |  |
| 0, ${ }^{\prime}$-DDD |  | $<7.2$ | - |
| p, ${ }^{\text {a }}$-DDD |  | $<8.5$ |  |
| O, ${ }^{\prime}$ '-DDT |  | <14 |  |
| p,p'-DDT |  | <18 |  |
| Mirex |  | $<4.0$ |  |
| Heptachlor Epoxide |  | $<0.48$ |  |
| alpha-Endosulphan |  | 0.75 | 0.6 |
| Dieldrin . |  | 5.1 | 4.4 |
| Endrin |  | <3.2 |  |
| Methoxychlor |  | 9.1 | 7.8 |
| Sample size 1.13 g dry <br> Daily total suspended solids loading $\mathrm{kg} / \mathrm{d}:$ <br> $\mathrm{pb}=$ procedural blank$\quad$ NDR = Peak detected but did not meet quantification criteria |  |  |  |
|  |  |  |  |
|  |  |  |  |






Appendix C1: Surrogate Recovery (\%) for Aroclor and Coplanar PCBs

| TSS Source/PCB | $\begin{aligned} & 13 \mathrm{C}- \\ & \mathrm{HCB} \\ & \hline \end{aligned}$ | $\begin{gathered} 13 \mathrm{C}- \\ \text { PCB } 101 \end{gathered}$ | $\begin{gathered} 13 C- \\ \text { PCB } 180 \end{gathered}$ | $\begin{gathered} 13 C- \\ \text { PCB } 209 \end{gathered}$ | $\begin{gathered} 13 \mathrm{C}- \\ \text { PCB } 77 \end{gathered}$ | $\begin{gathered} 13 \mathrm{C}- \\ \text { PCB } 126 \end{gathered}$ | $\begin{gathered} 13 C- \\ \text { PCB } 169 \end{gathered}$ |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| PULP MILLS |  |  |  |  |  |  | - |
| Nwood | 70 | 76 | 69 | 67 | 54 | 40 | 46 |
| $04 / 11 / 93$ $25 / 10 / 94^{*}$ | 70 | 51 | 57 | 58 | 59 | 52 | 51 |
| Canfor |  |  |  |  | 56 | 46 |  |
| 05/11/93 | 72 | 82 | 76 | 85 | 49 | 40 | 48 |
| 23/11/93 | 92 | 82 |  | 85 |  |  | 48 |
| QRP 01/11/93 | 75 | 77 | 72 | 71 | 5 | 44 | 54 |
| Cboo 02/11/93 | 88 | 82 | 81 | 81 | 70 | 52 | 70 |
| Wey 08/11/93 | 70 | 70 | 73 | 68 | 56 | 46 | 52 |
| MUNICIPAL SEWAGE |  |  |  |  |  |  |  |
| Landsdowne 03/11/93 | 78 | 74 | 86 | 74 | 46 | 38 | 50 |
| Annacis |  |  |  |  |  |  |  |
| 03/03/94 | 89 | 80 | 92 | 52 | 59 | 48 | 37 |
| $\begin{aligned} & 22 / 08 / 95 \\ & 21 / 11 / 95 \end{aligned}$ | - | - | - | 76 | 90 | 68 | 57 |
| Lulu 12/05/94 | - | - | - | 75 | 91 | 96 | 78 |
| COMBINED SEWER OVERFLOW**** |  |  |  |  |  |  |  |
| 06/04/94 | - | - | $\star$ | 90 | 95 | 100 | 98 |

* Aroclors analyzed by GC-HRMS on this instance, otherwise all other Aroclor analyses are GC-LRMS

Appendix C2: Spiked Matrix Samples - PCB Recovery (\%) (1) Coplanars

| PCB Spike | Nov 28/95 | Jan 10/95 | Apr 26/95 | Apr 29/96 |
| :--- | :---: | :---: | :---: | :---: |
| $\# 77$ | 110 | 110 | 97 | 81 |
| $\# 126$ | 91 | 120 | 96 | 85 |
| $\#$ \#169 | 97 | 86 | 85 | 76 |
| Surrogate |  |  |  |  |
| Recovery (\%) |  | 58 |  |  |
| 13C-PCB 77 | 100 | 46 | 63 | 79 |
| 13C-PCB 126 | 100 | 63 | 62 | 76 |
| 13C-PCB 169 | 89 | Lulu94 | Nwood 93 | Nwood 94 |
| Sites | Clark 94 | Canfor 93 |  | Annacis 95 |
|  |  | QRP 93 |  |  |
|  |  | Wey 93 |  |  |
|  |  | Landsdowne 93 |  |  |
|  |  | Annacis 94 |  |  |
|  |  |  |  |  |
|  |  |  |  |  |


| (ii) Aroclors |  |  |  |  |  |  |
| :--- | :---: | :---: | :---: | :---: | :---: | :---: |
| PCB Spike | Jul 15/94 | May 15/95 | Nov 20/95 | Feb 20/96 |  |  |
| Aroclor 1242 | 109 | 100 | 102 | 100 |  |  |
| Aroclor 1254 | 100 | 100 | 126 | 126 |  |  |
| Aroclor 1260 | 98 | 100 | 115 | 115 |  |  |
| Surrogate |  |  |  |  |  |  |
| Recovery (\%) |  |  |  |  |  |  |
| 13C-PCB 101 | 89 | 90 | - | - |  |  |
| 13C-PCB 180 | 100 | 89 | - | - |  |  |
| 13C-PCB 209 | 99 | 63 | 59 | 56 |  |  |
| Sites | Nwood 93 | Nwood 94 | Annacis Aug/95 | Annacis Nov/95 |  |  |
|  | Canfor 93 |  |  |  |  |  |
|  | QRP 93 |  |  |  |  |  |
|  | Wey 93 |  |  |  |  |  |
|  | Landsdowne 93 |  |  |  |  |  |
|  | Annacis 94 |  |  |  |  |  |


| (iii) Congeners |  |  |  |  |
| :---: | :---: | :---: | :---: | :---: |
| PCB Congener Copmpound Spike | Jul 15/94 | May 15/95 | Mar 05/96 | Feb 19/96 |
| 18 | 104 | 130 | 94 | 88 |
| 31/28 | 109 | 130 |  |  |
| 31 |  |  | 111 | 111 |
| 28 |  |  | 83 . | 66 |
| 52 | 103 | 120 | 106 | 97 |
| 95 | 130 | 100 |  |  |
| 66/80/95 |  |  | 117 | 100 |
| 118 | 103 | 110 | 114 | 129 |
| 138/163/164 | 85 |  |  |  |
| 138 |  | 120 |  |  |
| 138/158 |  |  | 129 | 102 |
| 180 | 102 | 100 | 133 | 124 |
| 196 | 94 |  |  |  |
| 196/203 |  | 120 | 122 | 102 |
| Sites | Nwood 93 | Nwood 94 | Annacis Aug/95 | Annacis Nov/95 |
|  | Canfor 93 QRP 93 |  |  |  |
|  | Wey 93 |  |  |  |
|  | Landsdowne 93 |  |  |  |

## Appendix C3 : Sample Surrogate Recovery (\%) for Organochlorine Pesticides

| Pesticide/TSS Sample <br> Source | Landsdowne <br> $03 / 11 / 93$ | Annacis <br> $03 / 03 / 94$ | Annacis <br> $22 / 08 / 95$ | Annacis <br> $21 / 11 / 95$ | Lulu <br> $12 / 05 / 94$ | Clark <br> $06 / 04 / 94$ |
| :--- | :---: | :---: | :---: | :---: | :---: | :---: |
| Tetrachloro-M-xylene | - | - | 51 | 57 | 58 | 64 |
| 13C-gamma BHC | 77 | 80 | - | - | - | - |
| 13C-p,p'-DDE | 72 | 73 | - | - | - | - |
| 13c-p,p'-DDT | 60 | 75 | - | - | - | - |
| 13C-Mirex | 80 | 76 | - | - | - | - |
| d4-alpha-Endosulphan | 120 | 130 | 94 | 33 | 94 | 100 |

Appendix C4: Spiked Sample and Surrogate Recovery (\%) for Organochlorine Pesticides

| Pesticide/TSS Sample Source | $\begin{gathered} \hline \text { Landsdowne } \\ 03 / 11 / 93 \\ \text { Annacis } \\ 03 / 03 / 94 \\ \hline \end{gathered}$ | $\begin{aligned} & \text { Annacis } \\ & \text { 22/08/95 } \end{aligned}$ | Annacis 21/11/95 |
| :---: | :---: | :---: | :---: |
| hexachlorobenzene | 98 | 100 | 111 |
| alpha HCH | 96 | 66 | 62 |
| beta HCH | 109 | 95 | 85 |
| gamma HCH | 98 | 89 | 91 |
| Heptachlor | 121 | 107 | 143 |
| Aldrin | 106 | 111 | 125 |
| Oxychlordane | 110 | 120 | 103 |
| trans-Chlordane | 113 | 103 | 119 |
| cis-Chiordane | 100 | 96 | 119 |
| o,p'-DDE | 95 | . 109 | 100 |
| p, p'-DDE | 94 | 96 | 106 |
| trans-Nonachlor | 102 | 115 | 95 |
| cis-Nonachlor | 100 | 100 | 100 |
| o,p'-DDD | 111 | 79 | 66 |
| p, p'-DDD | 113 | 76 | 63 |
| o,p'-DDT | 86 | - | - |
| p, p'-DDT | 105 | 100 ? | 102 |
| Mirex | 102 | 93 | 111 |
| Heptachlor Epoxide | 98 | 98 | 65 |
| alpha-Endosulphan | 98 | 98 | 90 |
| Dieldrin | 98 | 96 | 69 |
| Endrin | 109 | 109 | 62 |
| Methoxychior | 112 | 116 | 56 |
| 12C-HCB | 83 | - | - |
| 13C-gamma BHC | 96 | - | - |
| 13C-p,p ${ }^{\prime}$-DDE | 90 | - | - |
| 13c-p, p'-DDT | 100 | - | - |
| 13C-Mirex | 95 | - | - |
| Tetrachloro-M-xylene | - | 77 | 54 |
| d4-alpha-Endosulphan | 100 | 74 | 60 |


[^0]:    * Derksen, 1997

[^1]:    * Congeners analyzed by GC-HRMS on this instance, otherwise all other Aroclor analyses are GC-LRMS

[^2]:    ss = sample size; $\mathrm{pb}=$ procedural blank; NDR $=$ peak detected but did not meet quantification criteria

