Green-Duwamish River Watershed

PCB Congener Study: Phase 1

Prepared for

Toxics Cleanup Program
Northwest Regional Office
Washington State Department of Ecology
Bellevue, Washington

Prepared by

Leidos
18912 North Creek Parkway, Suite 101
Bothell, Washington 98011

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Table of Contents

Page

1.0 Introduction 1

2.0 Background 3

3.0 Description of Polychlorinated Biphenyls 7
3.1 Congeners 7
3.2 Homologs 14
3.3 Aroclors 15

4.0 Regulation of Polychlorinated Biphenyls 19
4.1 Relevant Laws and Regulations 19
4.1.1 Toxic Substances Control Act 19
4.1.2 State Laws and Regulations 20

5.0 Sources and Chemistry of Polychlorinated Biphenyls 27
5.1 Sources of Polychlorinated Biphenyls 27
5.1.1 Historical Uses and Sources of Polychlorinated Biphenyls 27
5.1.2 Current Sources (Non-Legacy) 32
5.2 Fate and Transport of Polychlorinated Biphenyls 44
5.2.1 Transport and Partitioning 44
5.2.2 Transformation/Degradation 49

6.0 Environmental Effects of Polychlorinated Biphenyls 53
6.1 Dioxin-Like Versus Non-Dioxin-Like Polychlorinated Biphenyls 53
6.1.1 Dioxin-Like Polychlorinated Biphenyls 53
6.1.2 Non-Dioxin-Like Polychlorinated Biphenyls 55
6.2 Modes of Polychlorinated Biphenyl Toxicity 55
6.3 Toxicity Summary 60

7.0 Chemical Analysis of Polychlorinated Biphenyls 61
7.1 Measurement of Polychlorinated Biphenyls 61
7.1.1 Screening Methods 61
7.1.2 Analysis of Aroclors 62
7.1.3 Analysis of Congeners 63
7.1.4 Analysis of Homologs 66
7.2 Data Comparisons and Translation 67
7.3 Laboratory and Reporting Issues 74
7.3.1 Laboratory Issues 75
7.3.2 Data Interpretation and Reporting 79

8.0 Summary of Available Data 87
8.1 Data Acquisition 87
8.2 Data Sets Included in Polychlorinated Biphenyl Congener Database 87
8.2.1 Lower Duwamish Waterway Remedial Investigation/Feasibility Study 88
8.2.2 East Waterway Supplemental Remedial Investigation/Feasibility Study 90
8.2.3 Lower Duwamish Waterway and Green River Contaminant Loading Studies 91
8.2.4 Studies Conducted by King County 92

8.2.5 Storm Drain System Studies 96
8.2.6 Other Fish/Shellfish Studies 97
8.2.7 Other Sediment Studies....102
8.2.8 Soil/Other Solids Data 110
8.3 Data Sets Not Included in the Polychlorinated Biphenyl Congener Database 110
8.4 Summary of Available Data....111
8.5 Recommendations for Data Management....123
8.5.1 Require Use of an Ecology-Approved Laboratory 123
8.5.2 Recommend a Standard Column for Polychlorinated Biphenyl Congener Analyses .123
8.5.3 Use Naming Conventions 123
8.5.4 Develop Standardized Data Codes....123
8.5.5 Develop Blank Contamination Decision Rules 124
8.5.6 Other Quality Assurance/Quality Control Issues....124
8.5.7 Develop Standard Data Output Formats 125

9.0 References....127

Appendices

Appendix A Lower Duwamish Waterway Source Control Bulk Atmospheric Deposition Study Polychlorinated Biphenyl Congener Profiles

Appendix B List of Co-eluting Congers in Green-Duwamish Data Set by Study Identifier

Appendix C Delaware River Basin Commission Guidance

Tables

Page

Table 3-1. List of PCB Congeners 8
Table 3-2. PCB Homolog Groups 15
Table 3-3. Homolog Group Composition of PCB Aroclors by Weight Percent 17
Table 4-1. Applicable Federal and Washington State Criteria for PCBs in Environmental Media…….. 21
Table 5-1. Historical Uses of PCB Aroclors 28
Table 5-2. PCB Congeners in Concrete Joint Material Samples 32
Table 5-3. PCB Congener Concentrations in Traffic Paint 35
Table 5-4. PCB Congener Concentrations in Silicone Rubber 36
Table 5-5. PCB Congener Concentrations in Motor Vehicle Fluids 38
Table 5-6. PCB Congener Concentrations in Asphalt-Related Products 39
Table 5-7. PCB Congener Concentrations in Other Materials 40
Table 5-8. Average PCB Emissions for Steady State Diesel Engine Tests 42
Table 5-9. Chemical Properties of PCB Homolog Groups 45
Table 5-10. Estimated Half-Lives of Representative PCB Congeners in Environmental Media ... 48

Table 6-1. TEFs of Dioxin-Like PCBs in Fish, Birds, and Mammals 54
Table 7-1. Comparison of PCB Sediment Analyses by Low- and High-Resolution Methods 67
Table 7-2. Laboratories Accredited by Washington State to Perform EPA Method 1668C.... 76
Table 8-1. Summary of Available PCB Congener Data – Green-Duwamish Watershed..... 112
Table 8-2. Data Qualifier Codes 124

Figures

Page

Figure 2-1. Green River and Major Tributaries 5
Figure 2-2. Lower Duwamish Waterway Source Control Areas 6
Figure 3-1. PCB Structure Showing Possible Chlorine Positions 7
Figure 3-2. Homolog Composition of "Early" and "Late" Aroclor 1254..... 16
Figure 3-3. Graphical Illustration of Aroclor Composition by Homolog Groups 18
Figure 5-1. Congener Profile of Aroclor 1016..... 28
Figure 5-2. Congener Profile of Aroclor 1242..... 29
Figure 5-3. Congener Profile of Aroclor 1248a..... 29
Figure 5-4. Congener Profile of Aroclor 1248g..... 29
Figure 5-5. Congener Profile of Aroclor 1254a..... 30
Figure 5-6. Congener Profile of Aroclor 1254g..... 30
Figure 5-7. Congener Profile of Aroclor 1260..... 30
Figure 5-8. Homolog Composition of PCB Aroclors 31
Figure 5-9. Frequency of Detection of PCB Congeners in Commercial Products 42
Figure 5-10. Relationship Between K_{ow} and Chlorine Content 45
Figure 7-1. McFarland and Clarke (1989) 36 Priority Congeners..... 66
Figure 7-2. Total PCBs by High-Resolution Congeners and Aroclor Analyses..... 68
Figure 7-3. Total PCBs by High-Resolution Congeners and Kaplan-Meier-Adjusted Low Resolution Homolog Analyses 69
Figure 7-4. Total PCBs in LDW Fish and Crab Tissue Samples – Comparison of Sums of Congeners and Sums of Aroclors 70
Figure 7-5. Total PCBs in Surface Sediment Samples – Comparison of Sums of Congeners and Sums of Aroclors 70
Figure 7-6. Total PCBs in East Waterway Fish and Crab Samples – Comparison of Sums of Congeners and Sums of Aroclors 71
Figure 7-7. Total PCBs in East Waterway Clam and Crab Samples – Comparison of Sums of Congeners and Sums of Aroclors 71
Figure 7-8. Total PCBs in LDW Storm Drain Samples – Comparison of Sums of Congeners and Sums of Aroclors 72
Figure 7-9. Conversion of EPA 1668C Congener Values to Aroclors 73
Figure 7-10. Co-Eluting Congeners by Analytical Column 75

Figure 7-11. PCB Congener Profiles for Base Flow Method Comparison Samples 79
Figure 7-12. Delaware River Basin Commission Method Blank Decision Rules 83
Figure 7-13. Spokane River Method Blank Contamination Decision Rules 84
Figure 8-1. Green-Duwamish Sediment Sampling Locations: PCB Congeners 115
Figure 8-2. Green-Duwamish Surface Water and Suspended Solids Sampling Locations: PCB Congeners 116
Figure 8-3. Green-Duwamish Soil and Other Solids Sampling Locations: PCB Congeners 117
Figure 8-4. Green-Duwamish Tissue Sampling Locations: PCB Congeners 118
Figure 8-5. Green-Duwamish Storm Drain Solids Sampling Locations: PCB Congeners..... 119
Figure 8-6. Green-Duwamish Storm Drain Water Sampling Locations: PCB Congeners..... 120
Figure 8-7. Green-Duwamish Air Deposition Sampling Locations: PCB Congeners..... 121
Figure 8-8. Green-Duwamish Upstream Sampling Locations: PCB Congeners..... 122

Acronyms and Abbreviations

1254-E	early formulation of Aroclor 1254
1254-L	later (1970s) formulation of Aroclor 1254
Ah	aryl hydrocarbon
BCF	bioconcentration factor
BDL	below detection limit
Boeing	The Boeing Company
CFR	Code of Federal Regulations
cm	centimeter
COC	chemical of concern
CSL	Cleanup Screening Level
CSO	combined sewer overflow
CTPS	chlorotriphenylsilane
CYP	cytochrome P450
DCDPS	dichlorodiphenylsilane
DPSDO	diphenylsilanediol
DRBC	Delaware River Basin Commission
DW	dry weight
EADA	emulsified asphalt dust abatement
ECD	electron capture detector
Ecology	Washington State Department of Ecology
EDL	estimated detection limit
EIM	Environmental Information Management
EMAP	Environmental Monitoring and Assessment Program
EMDL	estimated method detection limit
EMPC	estimated maximum possible concentration
EPA	U.S. Environmental Protection Agency
FR	Federal Register
FS	feasibility study
GC	gas chromatography
GC/MS	gas chromatography/mass spectrometry
HRGC	high-resolution gas chromatography
HRMS	high-resolution mass spectrometry
IARC	International Agency for Research on Cancer
IUPAC	International Union of Pure and Applied Chemistry
LDW	Lower Duwamish Waterway
LDWG	Lower Duwamish Waterway Group
LRMS	low-resolution mass spectrometry
µg/kg	micrograms per kilogram
µg/L	micrograms per liter
MDL	method detection limit
mg/kg	milligrams per kilogram
Monsanto	Monsanto Chemical Company
MS	mass spectrometry

Acronyms and Abbreviations, Continued

MTCA	Model Toxics Control Act
NDL	non-dioxin-like
$ng/m^2/day$	nanograms per square meter per day
NOAA	National Oceanic and Atmospheric Administration
NPDES	National Pollutant Discharge Elimination System
NS&T	National Status and Trends
NWRO	Northwest Regional Office
PBT	persistent, bioaccumulative, and toxic
PCB	polychlorinated biphenyl
PCDF	polychlorinated dibenzofurans
pg/L	picograms per liter
ppb	parts per billion
ppm	parts per million
ppq	parts per quadrillion
ppt	parts per trillion
PQL	practical quantitation limit
PSAMP	Puget Sound Assessment and Monitoring Program
PSEMP	Puget Sound Environmental Monitoring Program
QC	quality control
RCW	Revised Code of Washington
RI	remedial investigation
RM	River Mile
RPD	relative percent difference
SCO	Sediment Cleanup Objective
SMS	Sediment Management Standards
SQS	Sediment Quality Standard
TCDD	2,3,7,8 tetrachlorodibenzo-p-dioxin
TCPS	trichlorophenylsilane
TEF	toxic equivalency factor
TEQ	toxic equivalency quotient
TH	thyroid hormone
TSCA	Toxic Substances Control Act
U.S.	United States
USFWS	U.S. Fish and Wildlife Service
USGS	U.S. Geological Survey
WAC	Washington Administrative Code
WDFW	Washington Department of Fish and Wildlife
WHO	World Health Organization
WQC	Water Quality Criteria
WRIA	Water Resource Inventory Area

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1.0 Introduction

Thousands of environmental samples have been collected in the Green-Duwamish River watershed, and particularly in the Lower Duwamish Waterway (LDW), over the past 30 years. Sampled media include surface and subsurface river sediments, soil, groundwater, surface water, stormwater, storm drain solids, building materials, fish and shellfish tissue, and air. Many of these samples have been analyzed for polychlorinated biphenyls (PCBs), primarily as Aroclors. In recent years, some environmental samples have been analyzed for PCB congeners; congener analysis provides lower detection limits and is potentially a more useful tool to identify sources of PCB contamination.

The purpose of this report is to provide the Washington State Department of Ecology (Ecology) with a concise and readable summary of available information on PCB congeners and Aroclors, which identifies important issues to consider when evaluating historical PCB congener and/or Aroclor data or when collecting new data. In addition, this report compiles available PCB congener data in the Green-Duwamish watershed, including any available information on data quality.

Specific items that are addressed in this report include:

- Sources of PCBs to the environment, including historical and inadvertent new sources, and the relationship between specific Aroclors and congeners to types of source material.
- Fate and transport of PCBs, including volatilization and dechlorination.
- Toxicity of PCBs, including dioxin-like PCBs.
- Relationship between Aroclor and congener data.
- Measurement of Aroclors and PCB congeners, including laboratory methods.
- Laboratory analysis and data comparability issues.
- Numerical standards/criteria under various state and federal regulations.
- PCB congener data collection and management considerations.
- A summary of available congener data, including sediment, surface water, stormwater, storm drain solids, air, tissue, soil, and groundwater.
- Data quality and comparability, available metadata, and data gaps.

Section 2.0 of this report provides background information on the Green-Duwamish watershed and the LDW. Section 3.0 defines and describes PCBs, including Aroclors, congeners, and homologs. Section 4.0 summarizes laws and regulations that are applicable to PCBs. Section 5.0 describes sources and chemistry of PCBs, while Section 6.0 summarizes environmental and human health effects. Section 7.0 provides information on the chemical analysis of PCBs. Section 8.0 summarizes the available chemical data for PCB congeners in the Green-Duwamish watershed, and Section 9.0 lists references cited in this report.

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2.0 Background

The Green-Duwamish River watershed is located in Water Resource Inventory Area (WRIA) 9, which is located within King County, Washington. The watershed comprises a drainage area of approximately 470 square miles of varied terrain and land use, from forested headwater areas at the crest of the Cascade Mountains to industrial and port facilities of the Duwamish estuary (King County 2002). The Green River flows more than 90 miles from its headwaters near Stampede Pass on the Cascade Range crest to Elliott Bay in the City of Seattle (Figure 2-1).

The Upper Green River is located above the Howard Hanson Dam at River Mile (RM) 63; this area is protected forestland and provides drinking water to the city of Tacoma. The Middle Green River extends from the dam to the Auburn Narrows (RM 32), with a tributary area of nearly 180 square miles of residential, forest, and agricultural land uses. The Lower Green River, from the Auburn Narrows to Tukwila (RM 11), has a tributary area of approximately 64 square miles of residential, industrial, and commercial land uses.

The Duwamish River begins at the confluence of the Green and Black Rivers, at approximately RM 11. The LDW Superfund Site consists of the lower 5 miles of the Duwamish River, as measured from the southern tip of Harbor Island to just south of the Norfolk Combined Sewer Overflow (CSO)/Storm Drain (Figure 2-2). The LDW flows into the East/West Waterways, on either side of Harbor Island, and then into Elliott Bay in Seattle, Washington. Releases of contaminants from various human activities within the combined Green and Duwamish watersheds have resulted in contaminated sediments in the LDW.

The LDW site was added to the U.S. Environmental Protection Agency (EPA) National Priorities List in September 2001 due to the presence of chemical contaminants in sediment. The key parties involved in the LDW site are EPA, Ecology, and the Lower Duwamish Waterway Group (LDWG), which is composed of the City of Seattle, King County, the Port of Seattle, and The Boeing Company (Boeing).

In December 2000, EPA and Ecology signed an agreement with the LDWG to conduct a remedial investigation/feasibility study (RI/FS) for the LDW site. The RI was completed in July 2010 (Windward 2010b). The FS was completed in October 2012 (AECOM 2012).

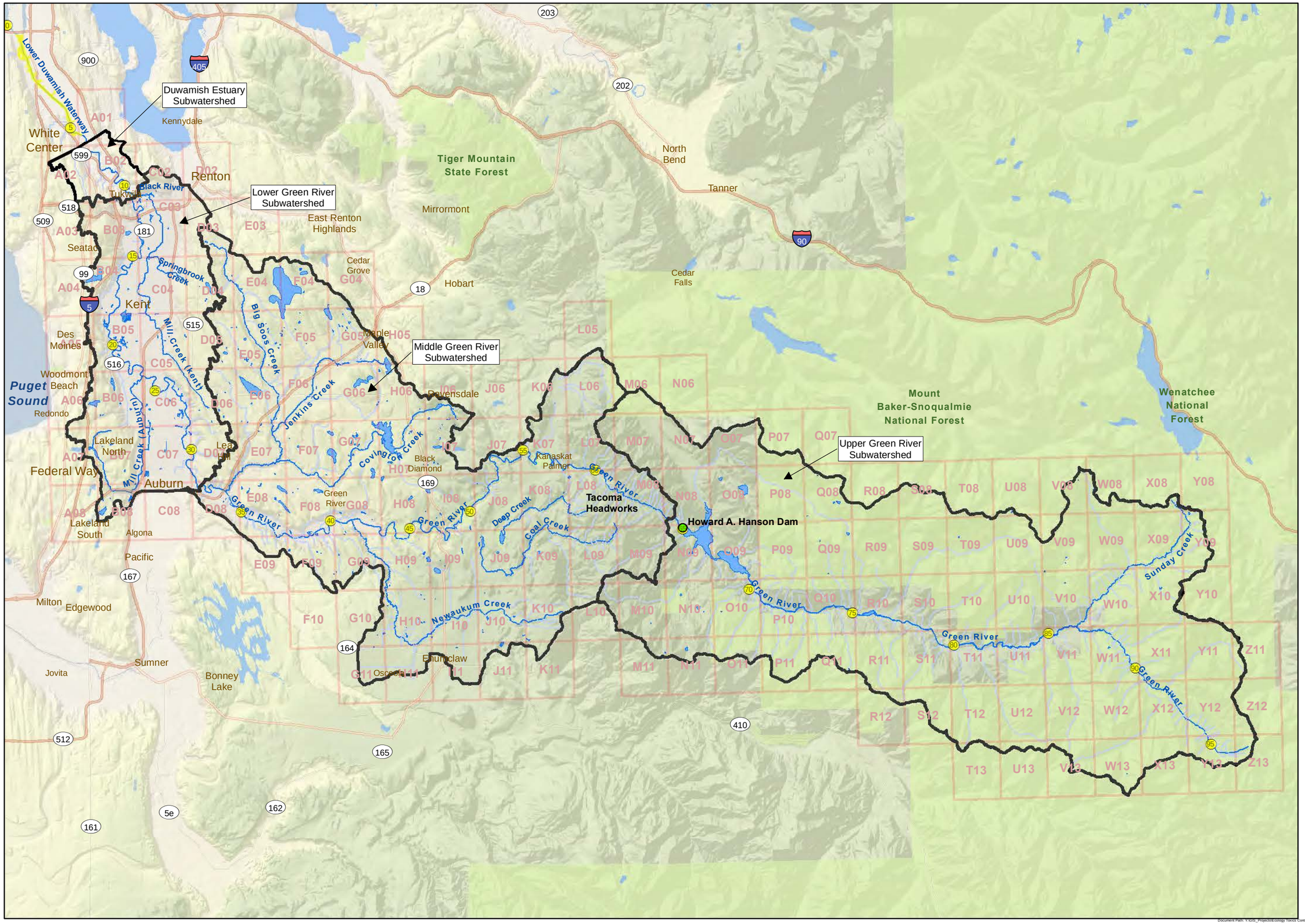
EPA selected a preferred cleanup alternative and published a Proposed Plan in 2013 and published a final cleanup plan in a Record of Decision dated November 2014 (EPA 2014a). Ecology is leading the effort to investigate adjacent and upland sources of contamination and to develop plans to reduce contaminant migration to waterway sediments.

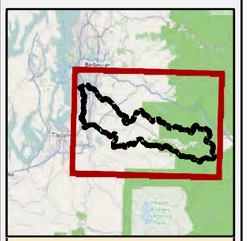
Studies of sediments in the LDW began in the 1980s and intensified in the 1990s (Ecology 2008b). Since 2001, studies of the LDW have measured contaminant levels, mapped distribution of sediment contaminants, estimated risks associated with exposure to contaminated sediments, modeled movement and fate of sediments, and evaluated options for cleanup. PCBs, in addition to other contaminants such as arsenic, dioxins/furans, and carcinogenic polycyclic aromatic hydrocarbons, have been identified as chemicals of concern (COCs) for human health in LDW sediments. River otters were also found to be at risk from exposure to PCBs. In addition, 41 COCs have been found to pose risks to bottom-dwelling organisms in the LDW

(EPA 2013a); these were present in one or more LDW sediment samples at a concentration above the Washington Sediment Quality Standard (SQS; Washington Administrative Code [WAC] Chapter 173-204).

Modeling conducted as part of the LDW RI (Windward 2010b) concluded that approximately 99 percent of the sediment load to the LDW comes from the upstream Green-Duwamish River. An analysis of suspended solids collected upstream of the LDW site indicated that this sediment loading is a possible source of recontamination to the LDW sediments (Ecology 2009b).

Figure 2-1. Green River and Major Tributaries





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River Mile

LDW Superfund Site

Subwatersheds

0 1 2 Miles

1 inch = 18,679 feet

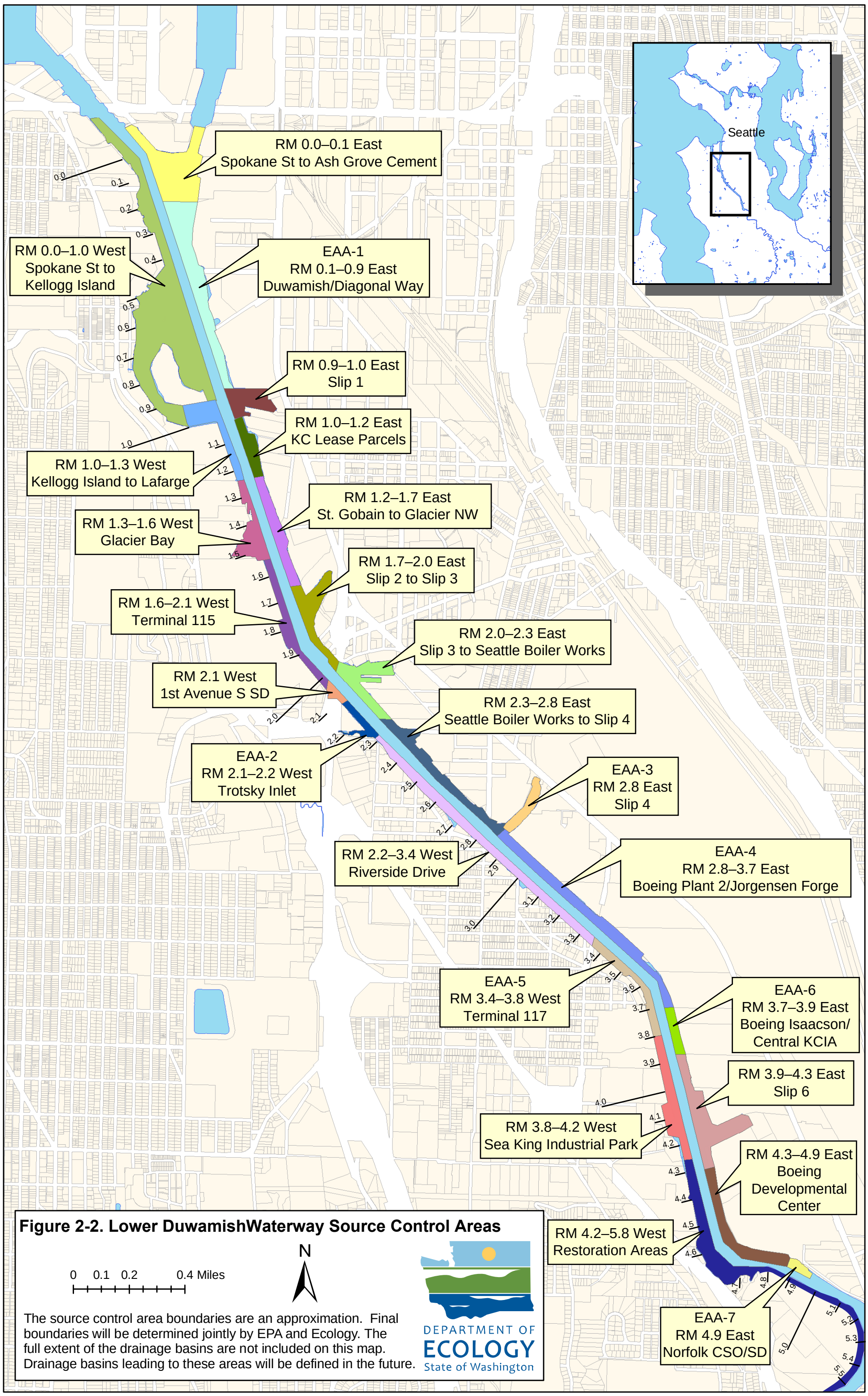


Figure 2-2. Lower DuwamishWaterway Source Control Areas

The source control area boundaries are an approximation. Final boundaries will be determined jointly by EPA and Ecology. The full extent of the drainage basins are not included on this map. Drainage basins leading to these areas will be defined in the future.

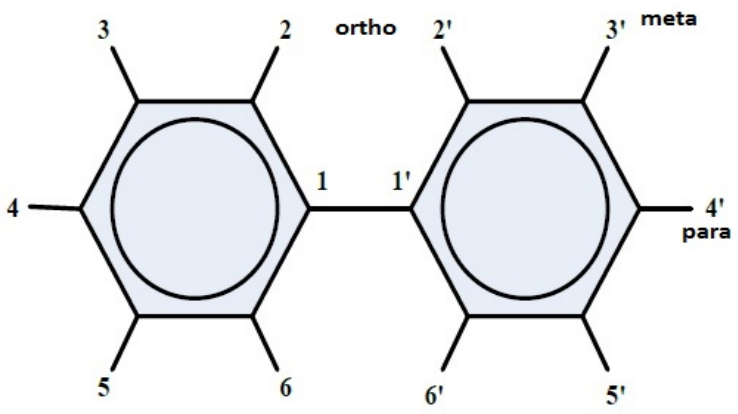
3.0 Description of Polychlorinated Biphenyls

PCBs are a family of man-made chemicals consisting of two joined benzene rings, with 1 to 10 chlorine atoms attached to the benzene rings. PCBs appear as oily liquids to white crystalline solids and hard non-crystalline resins. PCBs are odorless and range from colorless to light yellow or amber. In general, PCBs are hydrophobic (do not mix with or dissolve in water), non-flammable (not easily set on fire), chemically stable (do not break down or react readily with other chemicals), have high boiling points (do not readily volatilize at ambient temperatures), and have electrical insulating properties. PCBs also easily adsorb onto organic particles in soils, sediments, biological systems, and water.

3.1 Congeners

There are 209 possible PCB compounds, called congeners, which differ in the number of chlorine atoms present and where those chlorine atoms are located on the two benzene rings. The naming convention assumes that the two benzene molecules are joined together at the 1 and 1' position. The position of the chlorine atoms is differentiated by using 2 through 6 for one benzene ring and 2' (two prime) through 6' (six prime) for chlorine atoms on the second benzene ring (Figure 3-1).

Figure 3-1. PCB Structure Showing Possible Chlorine Positions



Hydrogen atoms are bound to the benzene rings at any location that does not contain a chlorine atom. A further naming convention using the terms ortho, meta and para is also used to identify the position of the chlorine atoms in a PCB molecule. If a chlorine atom is attached to one of the carbons adjacent to the 1 to 1' bond between the two benzene molecules (positions 2, 2', 6, or 6'), the chlorine atom is said to be in the ortho position. If a chlorine atom is attached to positions 3, 3', 5, or 5', it is in the meta position. If it is attached to the 4 or 4' position, it is in the para position.

Table 3-1 lists the 209 PCB congeners and provides information regarding their presence in Aroclor mixtures. Aroclors are discussed in Section 3.3.

Table 3-1. List of PCB Congeners

Congener Number	Chlorine Positions	Present in <0.1 Percent of Aroclor Mixtures[1]	Constitutes ≥1 Percent of these Aroclors[2]
Monochlorobiphenyls			
1	2		1221, 1232
2	3		1221, 1232
3	4		1221, 1232
Dichlorobiphenyls			
4	2,2'		1016, 1221, 1232, 1242
5	2,3		
6	2,3'		1016, 1221, 1232, 1242
7	2,4		1221, 1232
8	2,4'		1016, 1221, 1232, 1242
9	2,5		1221, 1232
10	2,6		
11	3,3'		
12	3,4		
13	3,4'		1221
14	3,5	●	
15	4,4'		1016, 1221, 1232, 1242
Trichlorobiphenyls			
16	2,2',3		1016, 1232, 1242
17	2,2',4		1016, 1232, 1242, 1248
18	2,2',5		1016, 1232, 1242, 1248
19	2,2',6		
20	2,3,3'		
21	2,3,4		
22	2,3,4'		1016, 1232, 1242, 1248
23	2,3,5		
24	2,3,6		
25	2,3',4		
26	2,3',5		1016, 1242
27	2,3',6		
28	2,4,4'		1016, 1242, 1248
29	2,4,5		
30	2,4,6	●	

Table 3-1. List of PCB Congeners

Congener Number	Chlorine Positions	Present in <0.1 Percent of Aroclor Mixtures[1]	Constitutes ≥1 Percent of these Aroclors[2]
31	2,4',5		1016, 1242, 1248
32	2,4',6		
33	2,3',4'		1016, 1242, 1248
34	2,3',5'		
35	3,3',4		
36	3,3',5	●	
37	3,4,4'		1016, 1242
38	3,4,5	●	
39	3,4',5	●	
Tetrachlorobiphenyls			
40	2,2',3,3'		
41	2,2',3,4		
42	2,2',3,4'		1016, 1242, 1248
43	2,2',3,5		
44	2,2',3,5'		1016, 1242, 1248
45	2,2',3,6		
46	2,2',3,6'		
47	2,2',4,4'		1016, 1248
48	2,2',4,5		1016, 1242, 1248
49	2,2',4,5'		1016, 1242, 1248
50	2,2',4,6	●	
51	2,2',4,6'		
52	2,2',5,5'		1016, 1242, 1248, 1254-L
53	2,2',5,6'		1248
54	2,2',6,6'		
55	2,3,3',4		
56	2,3,3',4'		1242, 1248
57	2,3,3',5		
58	2,3,3',5'	●	
59	2,3,3',6		
60	2,3,4,4'		1242, 1248
61	2,3,4,5	●	
62	2,3,4,6	●	
63	2,3,4',5		

Table 3-1. List of PCB Congeners

Congener Number	Chlorine Positions	Present in <0.1 Percent of Aroclor Mixtures[1]	Constitutes ≥1 Percent of these Aroclors[2]
64	2,3,4',6		1016, 1242, 1248
65	2,3,5,6		
66	2,3',4,4'		1242, 1248, 1254-E, 1254-L
67	2,3',4,5		
68	2,3',4,5'	●	
69	2,3',4,6	●	
70	2,3',4',5		1242, 1248, 1254-E, 1254-L
71	2,3',4',6		1016, 1242, 1248
72	2,3',5,5'	●	
73	2,3',5',6	●	
74	2,4,4',5		1242, 1248, 1254-L
75	2,4,4',6		
76	2,3',4',5'		
77	3,3',4,4'		
78	3,3',4,5	●	
79	3,3',4,5'	●	
80	3,3',5,5'	●	
81	3,4,4',5		
Pentachlorobiphenyls			
82	2,2',3,3',4		1254-E, 1254-L
83	2,2',3,3',5		
84	2,2',3,3',6		1248, 1254-E, 1254-L
85	2,2',3,4,4'		1248, 1254-E, 1254-L
86	2,2',3,4,5		
87	2,2',3,4,5'		1248, 1254-E, 1254-L
88	2,2',3,4,6		
89	2,2',3,4,6'		
90	2,2',3,4',5		
91	2,2',3,4',6		
92	2,2',3,5,5'		1254-E
93	2,2',3,5,6		
94	2,2',3,5,6'		
95	2,2',3,5',6		1248, 1254-E, 1254-L,

Table 3-1. List of PCB Congeners

Congener Number	Chlorine Positions	Present in <0.1 Percent of Aroclor Mixtures[1]	Constitutes ≥1 Percent of these Aroclors[2]
			1260
96	2,2',3,6,6'		
97	2,2',3,4',5'		1248, 1254-E, 1254-L
98	2,2',3,4',6'	●	
99	2,2',4,4',5		1248, 1254-E, 1254-L
100	2,2',4,4',6	●	
101	2,2',4,5,5'		1248, 1254-E, 1254-L, 1260
102	2,2',4,5,6'		
103	2,2',4,5',6		
104	2,2',4,6,6'	●	
105	2,3,3',4,4'		1248, 1254-E, 1254-L
106	2,3,3',4,5	●	
107	2,3,3',4',5	●	
108	2,3,3',4,5'	●	
109	2,3,3',4,6		
110	2,3,3',4',6		1248, 1254-E, 1254-L, 1260
111	2,3,3',5,5'	●	
112	2,3,3',5,6	●	
113	2,3,3',5',6	●	
114	2,3,4,4',5		
115	2,3,4,4',6		
116	2,3,4,5,6	●	
117	2,3,4',5,6		
118	2,3',4,4',5		1248, 1254-E, 1254-L
119	2,3',4,4',6		
120	2,3',4,5,5'	●	
121	2,3',4,5',6	●	
122	2,3,3',4',5'		
123	2,3',4,4',5'		
124	2,3',4',5,5'		
125	2,3',4',5',6		
126	3,3',4,4',5		
127	3,3',4,5,5'	●	

Table 3-1. List of PCB Congeners

Congener Number	Chlorine Positions	Present in <0.1 Percent of Aroclor Mixtures[1]	Constitutes ≥1 Percent of these Aroclors[2]
Hexachlorobiphenyls			
128	2,2',3,3',4,4'		1254-E, 1254-L, 1260
129	2,2',3,3',4,5		
130	2,2',3,3',4,5'		
131	2,2',3,3',4,6		
132	2,2',3,3',4,6'		1254-E, 1254-L, 1260
133	2,2',3,3',5,5'		
134	2,2',3,3',5,6		
135	2,2',3,3',5,6'		1260
136	2,2',3,3',6,6'		
137	2,2',3,4,4',5		
138	2,2',3,4,4',5'		1254-E, 1254-L, 1260
139	2,2',3,4,4',6		
140	2,2',3,4,4',6'	●	
141	2,2',3,4,5,5'		
142	2,2',3,4,5,6	●	
143	2,2',3,4,5,6'	●	
144	2,2',3,4,5',6		
145	2,2',3,4,6,6'	●	
146	2,2',3,4',5,5'		
147	2,2',3,4',5,6		
148	2,2',3,4',5,6'	●	
149	2,2',3,4',5',6		1254-E, 1254-L, 1260
150	2,2',3,4',6,6'	●	
151	2,2',3,5,5',6		
152	2,2',3,5,6,6'	●	
153	2,2',4,4',5,5'		1254-E, 1254-L, 1260
154	2,2',4,4',5,6'		
155	2,2',4,4',6,6'	●	
156	2,3,3',4,4',5		1260
157	2,3,3',4,4',5'		
158	2,3,3',4,4',6		
159	2,3,3',4,5,5'	●	
160	2,3,3',4,5,6	●	

Table 3-1. List of PCB Congeners

Congener Number	Chlorine Positions	Present in <0.1 Percent of Aroclor Mixtures[1]	Constitutes ≥1 Percent of these Aroclors[2]
161	2,3,3',4,5',6	●	
162	2,3,3',4',5,5'	●	
163	2,3,3',4',5,6		1254-E, 1260
164	2,3,3',4',5',6		
165	2,3,3',5,5',6	●	
166	2,3,4,4',5,6		
167	2,3',4,4',5,5'		
168	2,3',4,4',5',6	●	
169	3,3',4,4',5,5'	●	
Heptachlorobiphenyls			
170	2,2',3,3',4,4',5		1260
171	2,2',3,3',4,4',6		1260
172	2,2',3,3',4,5,5'		
173	2,2',3,3',4,5,6		
174	2,2',3,3',4,5,6'		1260
175	2,2',3,3',4,5',6		
176	2,2',3,3',4,6,6'		
177	2,2',3,3',4,5',6'		1260
178	2,2',3,3',5,5',6		
179	2,2',3,3',5,6,6'		
180	2,2',3,4,4',5,5'		1260
181	2,2',3,4,4',5,6	●	
182	2,2',3,4,4',5,6'	●	
183	2,2',3,4,4',5',6		1260
184	2,2',3,4,4',6,6'	●	
185	2,2',3,4,5,5',6		
186	2,2',3,4,5,6,6'	●	
187	2,2',3,4',5,5',6		1260
188	2,2',3,4',5,6,6'	●	
189	2,3,3',4,4',5,5'		
190	2,3,3',4,4',5,6		
191	2,3,3',4,4',5',6		
192	2,3,3',4,5,5',6	●	
193	2,3,3',4',5,5',6		

Table 3-1. List of PCB Congeners

Congener Number	Chlorine Positions	Present in <0.1 Percent of Aroclor Mixtures[1]	Constitutes ≥1 Percent of these Aroclors[2]
Octachlorobiphenyls			
194	2,2',3,3',4,4',5,5'		1260
195	2,2',3,3',4,4',5,6		
196	2,2',3,3',4,4',5,6'		1260, 1268
197	2,2',3,3',4,4',6,6'		
198	2,2',3,3',4,5,5',6		
199	2,2',3,3',4,5,5',6'		1260, 1268
200	2,2',3,3',4,5,6,6'		
201	2,2',3,3',4,5',6,6'		
202	2,2',3,3',5,5',6,6'		1268
203	2,2',3,4,4',5,5',6		1260
204	2,2',3,4,4',5,6,6'	●	
205	2,3,3',4,4',5,5',6		
Nonachlorobiphenyls			
206	2,2',3,3',4,4',5,5',6		1268
207	2,2',3,3',4,4',5,6,6'		
208	2,2',3,3',4,5,5',6,6'		1268
Decachlorobiphenyl			
209	2,2',3,3',4,4',5,5',6,6'		1268

[1] Frame et al. 1996.
[2] Definitive source for Aroclor 1268 congeners not found; congeners included for this Aroclor are based on the work by Kannan et al. 1998.
< Less than.
≥ Greater than or equal to.
PCB = Polychlorinated biphenyl.
Note: 1254-E – early formulation of Aroclor 1254; 1254-L – later (1970s) formulation of Aroclor 1254. See Section 3.3.1 for additional information. Congener number from the International Union of Pure and Applied Chemistry.

3.2 Homologs

PCBs are often grouped by the total number of chlorine atoms in the molecule; these groups are called homologs. Monochlorobiphenyl compounds have one chlorine atom attached to one of the biphenyl molecules. Dichlorobiphenyl compounds have two chlorine atoms; trichlorobiphenyl compounds have three chlorine atoms, and so forth to decachlorobiphenyl, which has all possible open benzene ring positions occupied by a chlorine atom. PCB congeners within the same homolog group have the same molecular weight and tend to have similar chemical properties, such as vapor pressure and water solubility. Chemical properties are discussed in Section 5.2. The number of congeners in each homolog group is listed in Table 3-2.

Table 3-2. PCB Homolog Groups

Homolog Group	No. of Congeners in Group
Monochlorobiphenyl	3
Dichlorobiphenyl	12
Trichlorobiphenyl	24
Tetrachlorobiphenyl	42
Pentachlorobiphenyl	46
Hexacholorbiphenyl	42
Heptachlorobiphenyl	24
Octachlorobiphenyl	12
Nonachlorobiphenyl	3
Decachlorobiphenyl	1

No. = Number.
PCB = Polychlorinated biphenyl.
Note: Individual congeners within each homolog group are identified in Table 3-1.

3.3 Aroclors

From 1929 to 1979, 700,000 tons (or 1.4 billion pounds) of PCBs were commercially manufactured in the United States (U.S.) (Commission for Environmental Cooperation 1996). Most of the PCBs used in the U.S. were manufactured by the Monsanto Chemical Company (Monsanto), which manufactured PCBs in the U.S. until 1977 (Erickson and Kaley 2011). PCBs were not typically manufactured as individual congeners, but as mixtures called Aroclors (a Monsanto trademark name for their mixtures). Aroclor mixtures were manufactured using specific chemical processes that resulted in varying chemical characteristics, such as vapor pressure, solubility, viscosity, or degree of chlorination, which produced a unique distribution of congeners in each mixture.

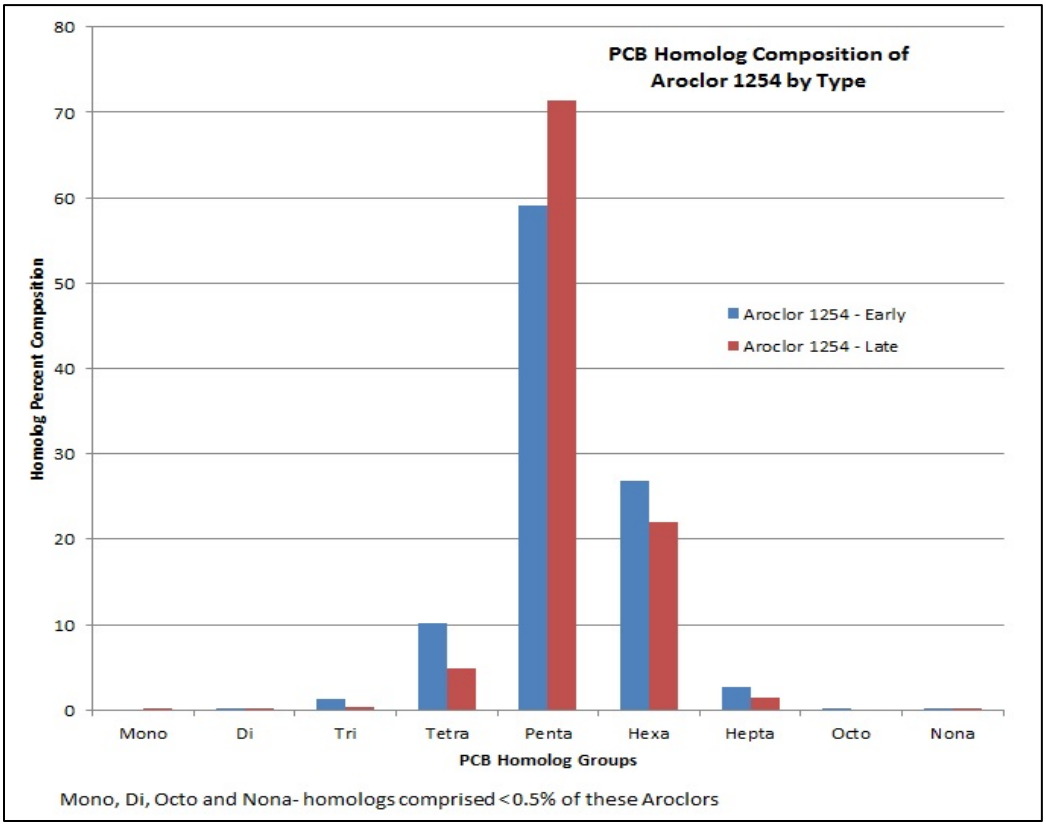
Prior to 1971, Monsanto produced Aroclors 1016, 1221, 1232, 1242, 1248, 1254, 1260, 1262, and 1268. Leidos was unable to find information confirming which Aroclor mixtures were manufactured between 1971 and 1977. Most Aroclor mixtures were named utilizing a code. The first two digits indicate the type of mixture; most begin with a 12, which indicates a refined Aroclor mixture. The last two digits indicate the percentage of chlorine in the mixture. Therefore, Aroclor 1254 contains 54 percent chlorine by weight, Aroclor 1232 contains 32 percent chlorine, etc. The only major Aroclor mixture that deviates from this system is 1016, which contains an average chlorine content of 41 percent (Ecology 2015a). Monsanto also manufactured Aroclors containing mixtures of PCBs with polychlorinated terphenyls, which have three benzene rings. These were sold as Aroclors 2565 and 4465.

Frame et al. (1996) used three different analytical systems to measure complete congener compositions for eight Aroclor types (all of the Aroclor types listed above except for

Aroclors 1268, 2565, and 4465). Two or three different lots[1] were analyzed for each Aroclor type, with the exception of Aroclor 1221, for which only one lot was analyzed. In total, 17 different Aroclor mixtures were analyzed. Frame et al. (1996) found relatively low differences (ranging from 6 to 12 percent) between lots of Aroclors 1242, 1260, and 1262, and higher differences (20 to 53 percent) for different lots of Aroclors 1248 and 1254.

Frame et al. (1996) showed that lots of Aroclor mixtures of the same chlorination level were not the same, which has had consequences for the use of Aroclor standards when calibrating analytical equipment during PCB analyses. The composition of Aroclor 1254 changed at some time in the 1970s to a congener mixture that contained higher concentrations of some of the more toxic congeners (e.g., PCB-126) (Frame 1999; Okun 2011) (Figure 3-2).

Figure 3-2. Homolog Composition of "Early" and "Late" Aroclor 1254



Adapted from ATSDR 2000 and Battelle Memorial Institute et al. 2012.

Called Type 2 Aroclor 1254 or "late" 1254, this Aroclor mixture also contained higher concentrations of toxic polychlorinated dibenzofurans (PCDFs), which were by-products of the manufacturing process and were considered to be impurities in the Aroclor product (Mayes et al. 1998; Okun 2011).

Erickson and Kaley (2011) note, however, that the Aroclor manufacturing processes were very similar among different manufacturers and among batches. If "the processes are well

[1] Different lots of the same Aroclor formulation came from the manufacturer (Monsanto) or from reference standard suppliers AccuStandard and Supelco.

controlled… there will be minor variations, but the major components will always be major components, and the trace components will always be trace components." Also, during Aroclor manufacture, they found that the chlorines tend to be distributed somewhat equally between the two rings; therefore, congeners with three or more chlorines on one ring and none on the other are not present in actual product mixtures, even though such congeners frequently serve as "research curiosities."

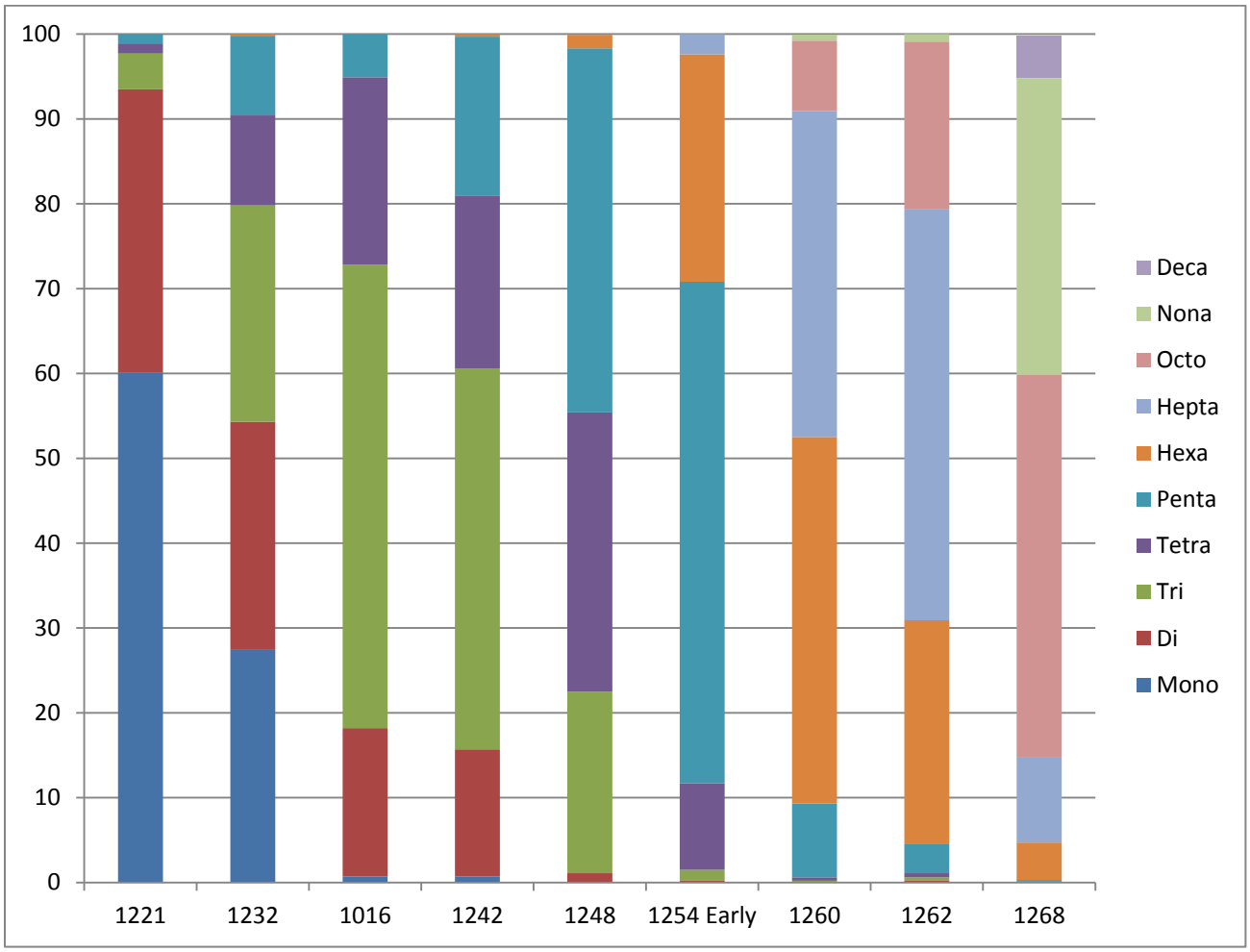
The weight percent of PCB homologs for nine of the primary PCB Aroclors plus the "late" Aroclor 1254 are provided in Table 3-3 and Figure 3-3 (adapted from ATSDR 2000 and Battelle Memorial Institute et al. 2012).

Table 3-3. Homolog Group Composition of PCB Aroclors by Weight Percent

Aroclor	Homolog (Weight Percent)									
	Mono	**Di**	**Tri**	**Tetra**	**Penta**	**Hexa**	**Hepta**	**Octo**	**Nona**	**Deca**
1221	60.1	33.4	4.2	1.2	1.1	0	0	0	0	0
1232	27.5	26.8	25.5	10.6	9.4	0.2	< 0.1	0	0	0
1016	0.7	17.5	54.6	22.1	5.1	0	0	0	0	0
1242	0.7	15	44.9	20.3	18.8	0.3	0	0	0	0
1248	< 0.1	1.1	21.4	32.9	42.9	1.6	< 0.1	0	0	0
1254-E	< 0.1	0.2	1.3	10.2	59.1	26.8	2.7	< 0.1	< 0.1	0
1254-L	0.2	0.1	0.4	4.9	71.4	22	1.4	0	< 0.1	No data
1260	< 0.1	< 0.1	0.2	0.4	8.7	43.2	38.4	8.3	0.7	0
1262	< 0.1	0.2	0.4	0.5	3.4	26.4	48.5	19.7	1.6	0
1268	0	0	< 0.1	0.1	0.2	4.4	10.1	45	35	4.8

PCB = Polychlorinated biphenyl.
Adapted from ATSDR 2000 and Battelle Memorial Institute et al. 2012.
Note that, of the major Aroclors produced, decachlorobiphenyl (PCB-209) was detected only in Aroclor 1268.

Figure 3-3. Graphical Illustration of Aroclor Composition by Homolog Groups



Adapted from ATSDR 2000 and Battelle Memorial Institute et al. 2012.

4.0 Regulation of Polychlorinated Biphenyls

4.1 Relevant Laws and Regulations

4.1.1 Toxic Substances Control Act

The 1976 Toxics Substances Control Act (TSCA) prohibited manufacture, processing, and distribution of PCBs. While TSCA is often referred to as a "ban" on PCBs, the law allowed some historical uses to continue and set allowable levels of inadvertent production of PCBs in other products. Some of the important requirements of TSCA that pertain to PCBs include (Ecology 2015a):

- Prohibits manufacture, sale, and distribution of PCBs, with exceptions.
- Mandates proper disposal for any PCBs unauthorized for use.
- Does not require testing to find PCB sources, but does require proper use and disposal of identified PCB-contaminated items. Ecology notes that many unauthorized uses are not discovered until a release to the environment has occurred.
- Limits use of PCBs to certain "totally enclosed" uses, such as transformers and capacitors, or concentrations below 50 parts per million (ppm) in bulk product. Various other levels exist for remediation waste and other limited uses, typically with EPA approval.
- Required that, by December 1998, all known transformers containing PCBs at concentrations greater than 500 ppm be registered with EPA. Ecology notes that there is no requirement to determine whether a transformer contains over 500 ppm PCBs, only to register it if it is known to be a PCB transformer with greater than 500 ppm PCBs.
- Allows many forms of PCB waste to be disposed of as municipal solid waste, which does not require PCBs to be listed on a manifest. Examples include:
    - Small, non-leaking PCB capacitors.
    - Plastics (e.g., plastic insulation from wire or cable; radio, television, and computer casings; vehicle parts; or furniture laminates); preformed or molded rubber parts and components; applied dried paints, varnishes, waxes, or other similar coatings or sealants; caulking; Galbestos; non-liquid building demolition debris; or non-liquid PCB bulk product waste from the shredding of automobiles or household appliances from which PCB small capacitors have been removed (shredder fluff). Any of these may also be disposed as landfill daily cover or as roadbed under asphalt.
    - Other PCB bulk product waste that leaches PCBs at less than 10 micrograms per liter (µg/L) of water measured using a procedure used to simulate leachate generation.
    - Other PCB bulk product waste other than those materials listed above if the PCB bulk product waste is segregated from organic liquids disposed of in the landfill unit and leachate is collected from the landfill unit and monitored for PCBs.
- Requires labels identifying electrical equipment containing over 500 ppm PCBs.

- Requires quarterly inspections of PCB transformers containing more than 60,000 ppm PCBs. Transformers with less than 60,000 ppm PCBs and those with appropriate secondary containment must be inspected for leaks at least annually.
- Requires EPA authorization for commercial storage of PCBs. Non-commercial storage does not always require EPA oversight.

4.1.2 State Laws and Regulations

PCBs are also regulated under additional state and federal laws. The Washington State laws and regulations listed below are directly related to the management of processes that produce PCBs; the production, use, and disposal of products that contain PCBs; and exposure limits and cleanup levels for PCBs.

Water Laws and Regulations

- Chapter 90.48 Revised Code of Washington (RCW), Water Pollution Control.
- Chapter 173-200 WAC, Water Quality Standards for Groundwaters of the State of Washington.
- Chapter 173-201A WAC, Water Quality Standards for Surface Waters of the State of Washington.
- Chapter 70.142 RCW, Chemical Contaminants and Water Quality.
- Chapter 246-290-72012 WAC, Group A Public Water Supplies – Regulated Contaminants.

Sediment Laws and Regulations

- Multiple Statutes – Chapters 90.48, 70.105D, 90.70, 90.52, 90.54 and 43.21 RCW.
- Chapter 173-204 WAC, Sediment Management Standards.

Air Laws and Regulations

- Chapter 70.94 RCW, Washington Clean Air Act.
- Chapter 173-460 WAC, Controls for New Sources of Toxic Air Pollutants.

Waste, Hazardous Substance, and Cleanup Laws and Regulations

- Chapter 70.105 RCW, Hazardous Waste Management.
- Chapter 173-303 WAC, Dangerous Waste Regulations.
- Chapter 70.95I RCW, Used Oil Recycling.
- Chapter 70.105D RCW, Hazardous Waste Cleanup – Model Toxics Control Act (MTCA).
- Chapter 173-340 WAC, MTCA – Cleanup.
- Chapter 173-360 WAC, Underground Storage Tank Regulations.

Applicable state and federal cleanup standards and criteria by media (sediments, groundwater, surface water, and air) are listed in Table 4-1. Some of the criteria and standards specify how the totals should be reported (e.g., total of Aroclors, total of congeners); most do not.

Table 4-1. Applicable Federal and Washington State Criteria for PCBs in Environmental Media

Name of Standard/Criterion	Statute/Regulation	Parameter	Value	Note
Soil				
MTCA Method A Cleanup Level, Unrestricted Land Use	WAC 173-340-740, Table 740-1	PCB mixtures	1.0 mg/kg	
MTCA Method A Cleanup Level, Industrial Properties	WAC 173-340-745, Table 745-1	PCB mixtures	10 mg/kg	
MTCA Method B Cleanup Level, Cancer	WAC 173-340-740	PCB mixtures and selected Aroclors	PCB mixtures: 0.50 mg/kg Aroclor 1016: 14.3 mg/kg Aroclors 1254, 1260: 0.50 mg/kg	1
MTCA Method B Cleanup Level, Non-cancer	WAC 173-340-740	Selected Aroclors	Aroclor 1016: 5.6 mg/kg Aroclor 1254: 1.6 mg/kg	1
MTCA Method C Cleanup Level, Cancer	WAC 173-340-740	PCB mixtures and selected Aroclors	PCB mixtures: 65.6 mg/kg Aroclor 1016: 1,880 mg/kg Aroclors 1254, 1260: 65.6 mg/kg	1
MTCA Method C Cleanup Level, Non-cancer	WAC 173-340-740	Selected Aroclors	Aroclor 1016: 245 mg/kg Aroclor 1254: 70 mg/kg	1
MTCA Simplified Terrestrial Ecological Evaluation	WAC 173-340-7492, Table 749-2	PCB mixtures (total)	2.0 mg/kg	
MTCA Ecological Indicator Concentration, Terrestrial Plants	WAC173-340-7493, Table 749-3	PCB mixtures (total)	40 mg/kg	
MTCA Ecological Indicator Concentration, Terrestrial Animals	WAC173-340-7493, Table 749-3	PCB mixtures (total)	0.65 mg/kg	
Groundwater				
WQC – Groundwater	WAC 173-200-040	PCBs	0.01 µg/L	

Table 4-1. Applicable Federal and Washington State Criteria for PCBs in Environmental Media

Name of Standard/Criterion	Statute/Regulation	Parameter	Value	Note
MTCA Method A Cleanup Level	WAC 173-340-720, Table 720-1	PCB mixtures	0.10 µg/L	
MTCA Method B Cleanup Level, Cancer	WAC 173-340-720	PCB mixtures and selected Aroclors	PCB mixtures: 0.0438 µg/L Aroclor 1016: 1.25 µg/L Aroclors 1254, 1260: 0.0438 µg/L	1
MTCA Method B Cleanup Level, Non-cancer	WAC 173-340-720	Selected Aroclors	Aroclor 1016: 1.12 µg/L Aroclor 1254: 0.32 µg/L	1
MTCA Method C Cleanup Level, Cancer	WAC 173-340-720	PCB mixtures and selected Aroclors	PCB mixtures: 0.438 µg/L Aroclor 1016: 12.5 µg/L Aroclors 1254, 1260: 0.438 µg/L	1
MTCA Method C Cleanup Level, Non-cancer	WAC 173-340-720	Selected Aroclors	Aroclor 1016: 2.45 µg/L Aroclor 1254: 0.70 µg/L	1
EPA MCL	SDWA, 40CFR141	PCB mixtures	0.50 µg/L	
EPA MCLG	SDWA, 40CFR141	PCB mixtures	0 µg/L	
WA State MCL	WAC 246-290-310	PCB mixtures	0.50 µg/L	
Surface Water				
MTCA Method B Cleanup Level, Cancer	WAC 173-340-730	PCB mixtures and selected Aroclors	PCB mixtures: 0.000105 µg/L Aroclor 1016: 0.00299 µg/L Aroclor 1254: 0.000105 µg/L	1
MTCA Method B Cleanup Level, Non-cancer	WAC 173-340-730	Selected Aroclors	Aroclor 1016: 0.00585 µg/L Aroclor 1254: 0.00167 µg/L	1
MTCA Method C Cleanup Level, Cancer	WAC 173-340-730	PCB mixtures and selected Aroclors	PCB mixtures: 0.00261 µg/L Aroclor 1016: 0.0747 µg/L Aroclor 1254: 0.00261 µg/L	1
MTCA Method C Cleanup Level, Non-cancer	WAC 173-340-730	Selected Aroclors	Aroclor 1016: 0.0146 µg/L Aroclor 1254: 0.00418 µg/L	1

Table 4-1. Applicable Federal and Washington State Criteria for PCBs in Environmental Media

Name of Standard/Criterion	Statute/Regulation	Parameter	Value	Note
WA WQC, Aquatic Life, Fresh/Acute	WAC 173-201A	PCBs	2.0 µg/L	
WA WQC, Aquatic Life, Fresh/Chronic	WAC 173-201A	PCBs	0.014 µg/L	
NR WQC, Aquatic Life, Fresh/Chronic	CWA §304(a)	PCBs	0.014 µg/L	4
NTR WQC, Aquatic Life, Fresh/Chronic	NTR, 40CFR131.36	Aroclors: 1016, 1221, 1232, 1242, 1248, 1254, and 1260	0.014 µg/L	
WA WQC, Aquatic Life, Marine/Acute	WAC 173-201A	PCBs	10 µg/L	
WA WQC, Aquatic Life, Marine/Chronic	WAC 173-201A	PCBs	0.030 µg/L	
NR WQC, Aquatic Life, Marine/Chronic	CWA §304(a)	PCBs	0.030 µg/L	4
NTR WQC, Aquatic Life, Marine/Chronic	NTR, 40CFR131.36	Aroclors: 1016, 1221, 1232, 1242, 1248, 1254, and 1260	0.030 µg/L	
NR WQC, Human Health, Consumption of Organisms	CWA §304(a)	PCBs	0.000064 µg/L	4
NTR WQC, Human Health, Consumption of Organisms	NTR, 40CFR131.36	PCBs	0.00017 µg/L	4
Air				
MTCA Method B Cleanup Level, Cancer	WAC 173-340-750	PCBs and selected Aroclors	PCBs: 0.00439 µg/m³ Aroclor 1016: 0.125 µg/m³ Aroclors 1254, 1260: 0.00439 µg/m³	

Table 4-1. Applicable Federal and Washington State Criteria for PCBs in Environmental Media

Name of Standard/Criterion	Statute/Regulation	Parameter	Value	Note
MTCA Method C Cleanup Level, Non-cancer	WAC 173-340-750	PCBs and selected Aroclors	PCBs: 0.0439 µg/m³ Aroclor 1016: 1.25 µg/m³ Aroclors 1254, 1260: 0.0439 µg/m³	
Sediment				
Marine SQS	WAC 173-204-320	Total PCBs	12 mg/kg OC	2
Marine SCO	WAC 173-204-562	Total PCBs	12 mg/kg OC	2
Marine Sediment CSL	WAC 173-204-562	Total PCBs	65 mg/kg OC	2
Freshwater SCO	WAC 173-204-563	Total PCB Aroclors	110 µg/kg DW	2, 3
Freshwater Sediment CSL	WAC 173-204-563	Total PCB Aroclors	2,500 µg/kg DW	2, 3

[1] MTCA Method B and Method C values are the standard formula values listed on Washington State Department of Ecology's Cleanup Levels and Risk Calculation (CLARC) website. These values are not cleanup levels, as the pre-calculated formula values listed on CLARC do not include site-specific values required to calculate a cleanup level.

[2] Where chemical criteria in this table represent the sum of individual compounds or isomers, the following methods are applied: (i) where chemical analyses identify an undetected value for every individual compound/isomer, then the single highest detection limit shall represent the sum of the respective compounds/isomers; and (ii) where chemical analyses detect one or more individual compound/isomers, only the detected concentrations will be added to represent the group sum.

[3] The total PCB Aroclors criterion represents the sum of the following Aroclors: 1016, 1221, 1242, 1248, 1254, 1260, and 1268.

[4] This criterion applies to total PCBs (e.g., the sum of all congener or isomer or homolog or Aroclor analyses).

CFR = Code of Federal Regulations.
CSL = Cleanup screening level.
CWA = Clean Water Act.
DW = Dry weight.
EPA = U.S. Environmental Protection Agency.
µg/L = Micrograms per liter.
µg/m^3 = Micrograms per cubic meter.
MCL = Maximum contaminant level.
MCLG = Maximum contaminant level goal.
mg/kg = Milligrams per kilogram.
MTCA = Model Toxics Control Act.
NR = National Recommended.
NTR = National Toxics Rule.
OC = Organic carbon normalized.
PCB = Polychlorinated biphenyl.
SCO = Sediment cleanup objective.
SDWA = Safe Drinking Water Act.
SQS = Sediment Quality Standard.
WA = Washington.
WAC = Washington Administrative Code.
WQC = Water Quality Criteria.

Water Quality Criteria (WQC): Washington State's WQC for groundwater (WAC 173-200-040) and surface water for the protection of aquatic life (WAC 173-201A-240[2]) specify "PCBs" in the criteria listings. The federal National Recommended WQC for aquatic life and for human health, published by EPA (2015), have footnotes indicating that the criteria apply to "total PCBs (e.g., the sum of all congener, or all isomer, or homolog, or Aroclor analyses)."

Sediment Management Standards (SMS): The Washington State SMS (WAC Chapter 173-204) include numeric chemical and biological benthic criteria for marine sediments and narrative standards for the freshwater benthic community and protection of human health (Table 4-1). The marine SQS criteria (WAC 173-204-320) correspond to a sediment quality that will result in no adverse effects on biological resources and no significant health risk to humans; numeric criteria apply to marine sediments located within Puget Sound. The SMS also provide numeric criteria for protection of benthic organisms associated with sediment cleanup (WAC 173-204-500), including sediment cleanup objectives (SCOs) and cleanup screening levels (CSLs) for marine and freshwater sediments. The SMS criteria for PCBs in marine sediments are for "total PCBs"; the *Sediment Cleanup User's Manual II* (Ecology 2015b) states that total PCBs in marine sediments are to be calculated as the sum of Aroclors 1016, 1221, 1242, 1248, 1254, 1260, and 1268 for compliance with the numeric benthic criteria. The SCO and CSL chemical criteria of PCBs in freshwater sediments for benthic toxicity specify "total PCB Aroclors," with the criterion representing the sum of Aroclors 1016, 1221, 1242, 1248, 1254, 1260, and 1268. However, Section 6.3.2.3 of the *Sediment Cleanup User's Manual II* specifies the evaluation of total PCB congeners (all 209) along with the dioxin-like PCB congeners using toxicity equivalency factors (TEFs) when bioaccumulative concerns are investigated.

MTCA: MTCA establishes administrative processes and standards to identify, investigate, and clean up facilities where hazardous substances have come to be located. It includes cleanup levels for PCBs in soil, groundwater, and surface water (Table 4-1). MTCA defines PCBs or PCB mixtures as aromatic compounds containing two benzene nuclei with two or more substituted chlorine atoms. It specifies that this includes "those congeners which are identified using the appropriate analytical methods as specified in WAC 173-340-830." Method A cleanup level tables for soil and groundwater note that the cleanup levels listed in Tables 720-1, 740-1, and 745-1 are based on "a total value for all PCBs." MTCA's Analytical Procedures section (WAC 173-340-830) has not been updated since 2003 and cites old methods (e.g., Standard Methods for the Examination of Water and Wastewater; American Public Health Association; American Water Works Association; and Water Pollution Control Federation, 20th edition, 1998). It does not specifically mention EPA Method 1668, the most common analytical method currently used for PCB congener analysis. However, an Ecology Toxics Cleanup Program policy implementation memorandum (#12)[3] specifies when to use Method 1668 in contaminated site cleanup.

Dangerous Waste Regulations: The Dangerous Waste Regulations (WAC 173-303) apply to specific wastes if they contain PCBs at a concentration of 2 ppm or greater (see also Ecology [2010] *Polychlorinated Biphenyl Dangerous Waste Discussion Paper*). Examples of wastes covered by the Dangerous Waste Regulations include residues, contaminated soils, absorbents,

[2] <https://fortress.wa.gov/ecy/publications/publications/0610091.pdf>.
[3] <https://fortress.wa.gov/ecy/publications/SummaryPages/1509052.html>.

and personal protective equipment contaminated as a result of salvaging, rebuilding, or discarding transformers, capacitors, or bushings; cores from transformers that are being salvaged, rebuilt, or discarded; and transformers, capacitors, or bushings that will no longer be used for their intended use and are being salvaged, rebuilt, or discarded, unless they have been drained. PCB wastes whose disposal is regulated by EPA under 40 Code of Federal Regulations (CFR) 761.60 (excluded under WAC 173-303-071[3][k]) are not covered by the Dangerous Waste Regulations; these include wastes with greater than 50 ppm PCB and small capacitors.

5.0 Sources and Chemistry of Polychlorinated Biphenyls

5.1 Sources of Polychlorinated Biphenyls

Transformers and capacitors represent the largest historical use of PCBs and were targeted for PCB removal by utilities and owners of electrical equipment that worked to identify and replace PCB-containing equipment. Recent estimates of PCBs still in use cite data from older publications. There are no current estimates for the quantities of PCBs that may remain in use in closed or open applications (see Section 5.1.1) or that have been landfilled, destroyed by incineration, or released to the environment.

5.1.1 Historical Uses and Sources of Polychlorinated Biphenyls

The largest historical use for PCBs produced in the U.S. before manufacturing was halted in 1977 was in electrical equipment (such as capacitors and transformers). These two uses alone accounted for an estimated 77 percent of the PCBs produced in the U.S. between 1929 and 1975 (Commission for Environmental Cooperation 1996). Other uses included (in decreasing order of quantity) plasticizers, hydraulics and lubricants, carbonless copy paper, heat transfer fluids, and petroleum additives. These historical PCB sources are often referred to as “legacy uses” because continued manufacturing of these items containing PCBs was banned, although the items may have remained in service for many years and may still be in use today.

PCB products can be defined as being either “closed” or “open” sources. A closed PCB source is contained inside a structure (e.g., an electrical transformer). In closed systems, PCBs are only exposed to the environment in the event of a leak or spill. While many PCB-containing closed sources have been retired, some remain in use, including electrical transformers and fluorescent light ballasts.

Open sources of PCBs are exposed to the environment and include such uses as additives in paints and caulks, wire insulators, inks, and dyes; these account for an estimated 15 percent of legacy uses in the U.S. (Ecology 2015a). Natural gas pipelines, inadvertently contaminated with PCBs when PCBs were used in compressor systems, are considered “partially closed” PCB sources. Other partially closed PCB sources include hydraulic fluids and heat exchange fluids. Partially closed PCB sources account for an estimated 10 percent of legacy uses in the U.S.

A list of historical PCB Aroclor end uses is provided in Table 5-1. For example, capacitors used Aroclors 1016, 1221, and 1254, while transformers used Aroclors 1242, 1254, and 1260.

A little more than 100 of the possible 209 PCB congeners were included in the various Aroclor formulations at easily detectable levels (Battelle Memorial Institute et al. 2012). Frame et al. (1996) used 3 different analytical techniques to completely characterize the congener makeup of 17 different Aroclors (see Section 3.3). Figures 5-1 through 5-7 show the congener profiles of five Aroclor mixtures graphically, including two different lots for Aroclors 1248 and 1254 (1248a/1248g, and 1254a/1254g). Some of the congener peaks indicated in the figures are comprised of more than one congener (called co-elutes). Issues with co-eluting congeners (e.g., overestimation of particular congeners) are discussed in Section 7.0.

Table 5-1. Historical Uses of PCB Aroclors

End Use	Aroclor								
	1016	1221	1232	1242	1248	1254	1260	1262	1268
Capacitors	●	●				●			
Transformers				●		●	●		
Heat transfer				●					
Hydraulics/lubricants									
Hydraulic fluids			●	●	●	●	●		
Vacuum pumps					●	●			
Gas-transmission turbines		●		●					
Plasticizers									
Rubbers		●	●	●	●	●			●
Synthetic resins						●	●	●	●
Carbonless paper			●	●					
Miscellaneous									
Adhesives		●	●	●	●	●			
Wax extenders				●		●			●
De-dusting agents						●	●		
Inks						●			
Cutting oils						●			
Pesticide extenders						●			
Sealants/caulks						●			

PCB = Polychlorinated biphenyl.
Adapted from ATSDR 2000.

Figure 5-1. Congener Profile of Aroclor 1016

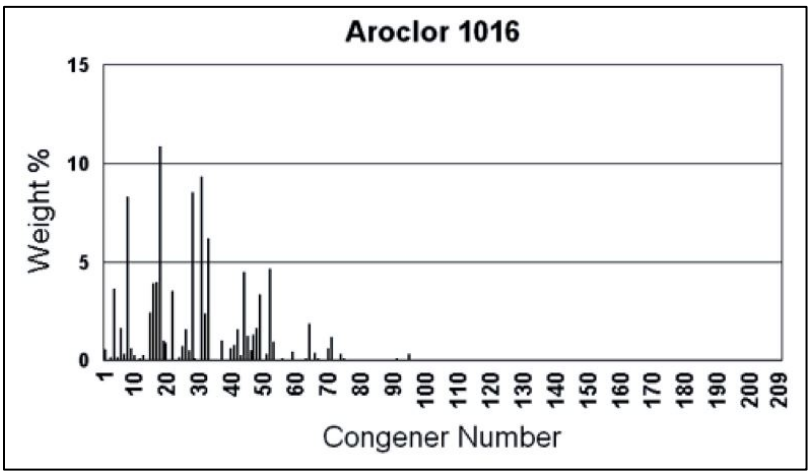


Figure 5-2. Congener Profile of Aroclor 1242

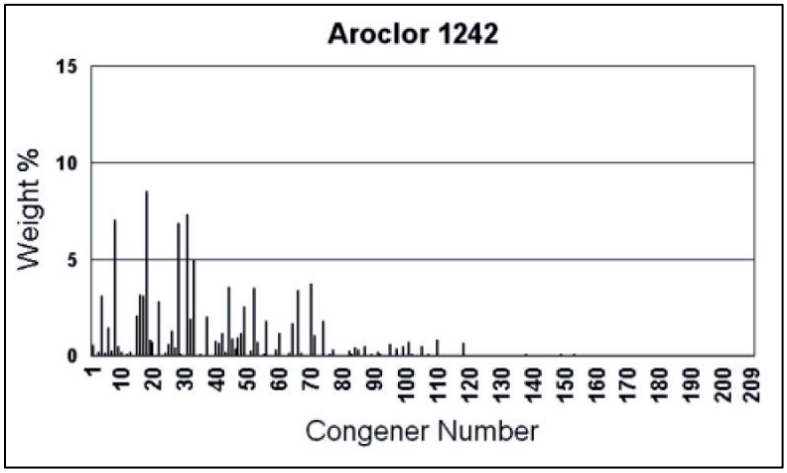


Figure 5-3. Congener Profile of Aroclor 1248a

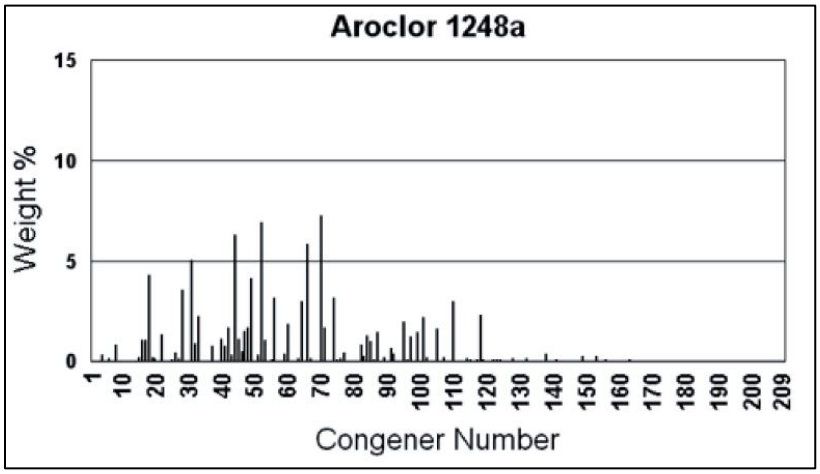


Figure 5-4. Congener Profile of Aroclor 1248g

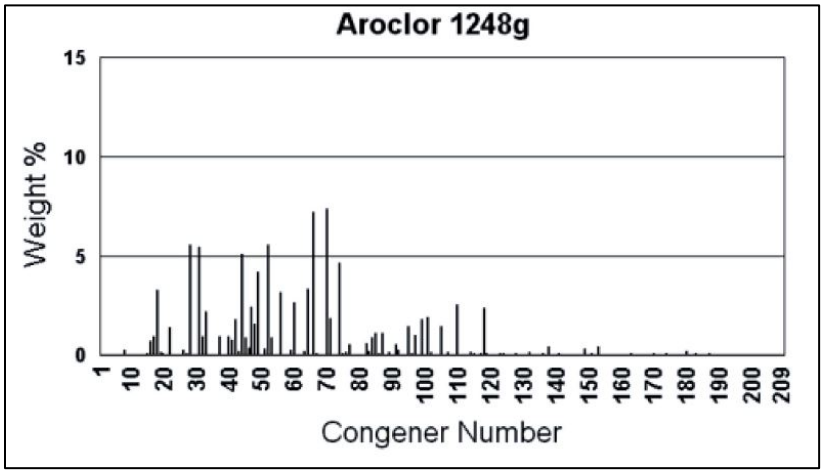


Figure 5-5. Congener Profile of Aroclor 1254a

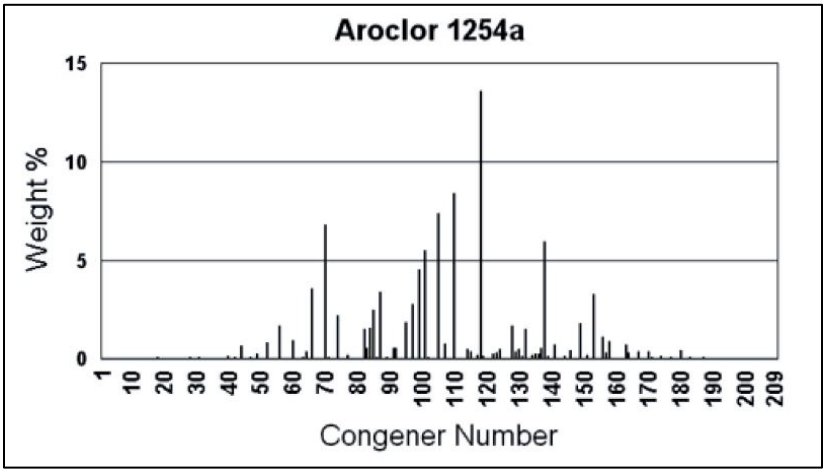


Figure 5-6. Congener Profile of Aroclor 1254g

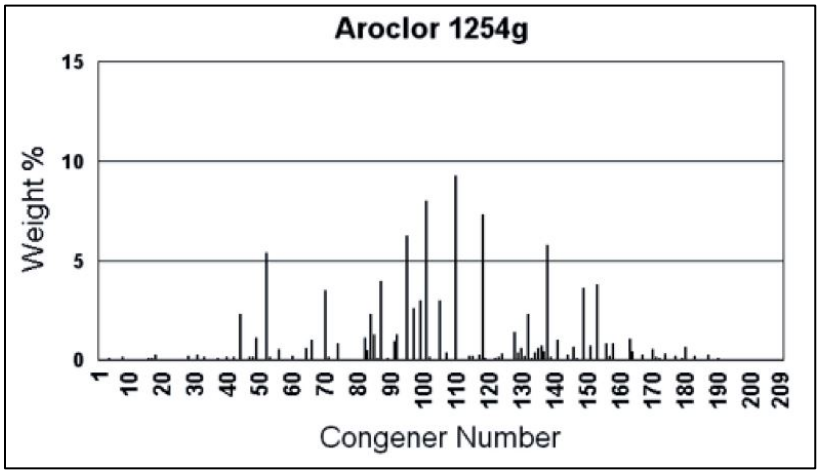


Figure 5-7. Congener Profile of Aroclor 1260

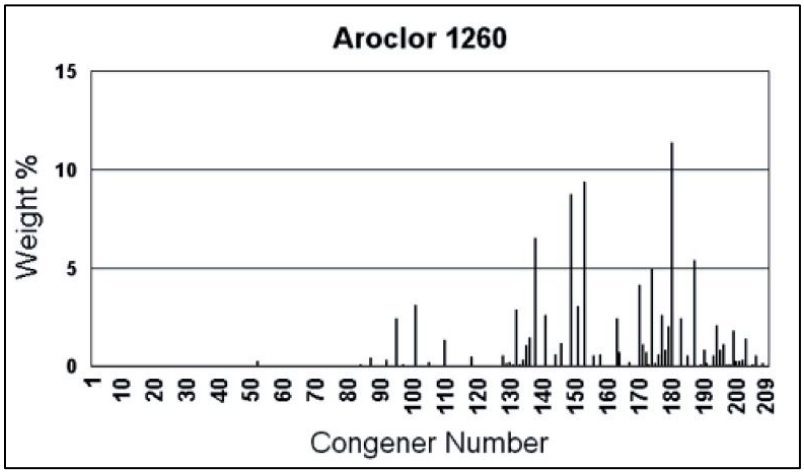
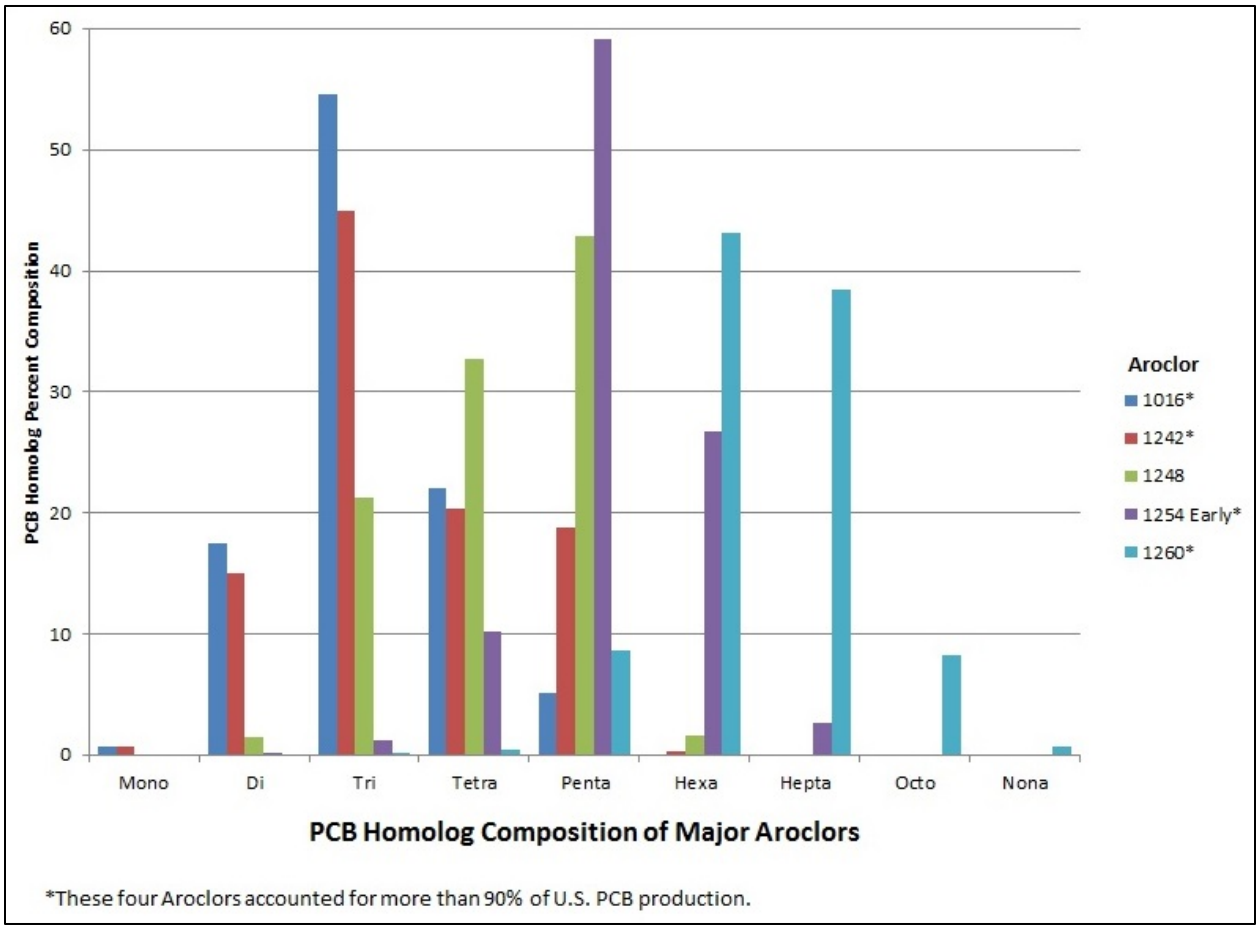


Figure 5-8 shows the differences between four major Aroclor mixtures (1016, 1242, 1254-E, and 1260) plus Aroclor 1248 (commonly tested for in the LDW) by homolog group (adapted from ATSDR 2000 and Battelle Memorial Institute et al. 2012).[4] The four major Aroclors accounted for more than 90 percent of PCB production (Battelle Memorial Institute et al. 2012).

Figure 5-8. Homolog Composition of PCB Aroclors



A total of 52 PCB congeners did not appear at concentrations above 0.01 percent by weight in any of the Aroclors tested by Frame et al. (1996) (shown in Table 3-1). Analyses of environmental samples as Aroclors may have missed some of the congeners that did not typically occur in Aroclors. For additional discussion of this issue, see Section 7.1.1.

PCBs added to paints and caulks have been implicated as contributing to soil, sediment, and air contamination in studies across the globe (see, for example, studies by Priha et al. 2005; Herrick et al. 2007; Jartun et al. 2009; Robson et al. 2010; Klausterhaus et al. 2014). A recent study conducted in the LDW drainage basin (SAIC 2011) found PCBs in 15 of 38 (39 percent) of building paint composite samples, with detected concentrations from 0.85 to 61 milligrams per kilogram (mg/kg). PCBs were detected in 8 of 17 (47 percent) of building caulk samples, with detected concentrations from 3.0 to 920 mg/kg.

[4] Note that these are the same data presented in Figure 3-2, but in a different format.

Studies of PCB sources at North Boeing Field have revealed that expansion joint materials used in flight lines were formulated with PCBs. Boeing and others have collected numerous samples of the joint material in conjunction with investigations of PCB sources to Slip 4 in the LDW. Two concrete joint material samples were analyzed for PCB congeners (Exponent 2009). Penta-, hexa, and heptachlorinated congeners were dominant in both samples, but the total PCB concentrations were markedly different, with the total of PCB congeners approximately 59 mg/kg in one sample and 18,710 mg/kg in the other. The dominance of highly chlorinated congeners in the concrete joint material is consistent with the results of other researchers examining PCBs in caulk materials. Robson et al. (2010), investigating the significance of building sealants as a source of PCBs to the environment, found that joint sealants lost lower chlorinated congeners over time to volatilization.

Table 5-2 presents concentrations for some of the congeners present in the highest concentrations (Exponent 2009).

Table 5-2. PCB Congeners in Concrete Joint Material Samples

Congener(s)	Sample Concentration in µg/kg	
	Sample 1	Sample 2
44/47/65	82,800	330
52	178,000	681
61/70/74/76	277,000	1,250
95	736,000	2,020
99	402,000	1,520
108/119/86/97/125/87	1,050,000	3,080
110	1,890,000	5,220
113/90/101	1,280,000	3,870
118	1,360,000	4,450
132	662,000	1,350
147/149	943,000	2,440
153/168	1,090,000	3,080
163/138/129	2,380,000	4,870
180/193	400,000	1,990
187	122,000	1,390
Total of PCB congeners:	18,709,317	58,745

µg/kg = Micrograms per kilogram.
PCB = Polychlorinated biphenyl.

5.1.2 Current Sources (Non-Legacy)

PCBs are no longer intentionally manufactured in the U.S., and the manufacture, processing, and distribution in commerce of PCBs at concentrations of 50 ppm or greater is not allowed. EPA promulgated a rule under TSCA in 1984 for inadvertent generation of PCBs that are not in closed or controlled manufacturing processes (49 Federal Register [FR] 28172). Inadvertently generated PCBs in products must have an annual average concentration of less than 25 ppm, with a

maximum of 50 ppm. For comparison to water quality considerations, 50 ppm is equivalent to 50,000,000,000 parts per quadrillion (ppq) (City of Spokane 2015). The current Washington State human health surface water quality standard for PCBs is 0.00017 µg/L or 170 ppq (derived from the National Toxics Rule, 40 CFR 131.36). TSCA has a discounting factor for mono- and di-chlorobiphenyls when they are produced inadvertently: "inadvertently generated non-Aroclor PCBs are defined as the total PCBs calculated following division of the quantity of monochlorinated biphenyls by 50 and dichlorinated biphenyls by 5" (40 CFR Part 761.3). EPA required manufacturers with processes inadvertently generating PCBs and importers of products containing inadvertently generated PCBs to report to EPA any process or import for which the PCB concentration is greater than 2 mg/kg for any resolvable PCB gas chromatographic peak (Ecology 2015a). More details on TSCA are presented in Section 4.0.

As part of the rulemaking on inadvertently generated PCBs, EPA generated a list of 200 chemical processes with a potential for generating PCBs and narrowed it to 70 with a high potential. The list does not include every process that inadvertently generates PCBs, and not all of the chemical processes on this list will inadvertently generate PCBs, but the potential for inadvertent production exists (Ecology 2015a; the list is presented in Appendix D of the PCB Chemical Action Plan). In general, processes that involve chlorine, carbon, and high temperatures have the potential to produce PCBs (Muñoz 2007). Sources of inadvertently generated PCBs are discussed below.

Pigments and Dyes

PCBs are known to be inadvertently generated in certain pigments and dyes, including diarylides (yellow and orange), naphtharylamides (oranges and reds), phthalocyanines (blue), and basic dye complex pigments (reds, violets, blues, and greens) (Ecology 2014a). Hu and Hornbuckle (2010) found PCBs in azo (yellows, reds, and oranges) and phthalocyanine (blues and greens) pigments, including PCB-11 and higher-chlorinated PCB-206 through PCB-209. Previously, PCB-209 was only thought to be found in ferric oxide as a by-product of titanium dioxide production (Panero et al. 2005). PCB-11 and PCB-209 have been found in Washington's environment and animals (Ecology Environmental Information Management [EIM] database). PCB-11 is thought to be produced primarily from pigment production and not from legacy uses of Aroclors (Hu and Hornbuckle 2010; Guo et al. 2014), and it was not listed among the 52 congeners comprising less than 0.01 percent (by weight) of Aroclors as analyzed by Frame et al. (1996). Studies conducted of azo and phthalocyanine pigments in Japan and China have also shown PCB-11 to be a major constituent of the PCBs found in pigment (Anezaki and Nakano 2014; Shang et al. 2014).

Higher-chlorinated PCBs are inadvertently generated during the production of the inorganic pigment titanium dioxide using the chlorine process (UNEP 2007). Ecology was unable to locate estimates on the amount of PCB inadvertently generated in this process.

The City of Spokane (2015) analyzed a variety of traffic paints (such as traffic line paint and thermoplastic road striping) for PCB congeners. Total PCBs in yellow traffic marking paints ranged from 0.565 to 64.88 micrograms per kilogram (µg/kg) and was measured at 10.78 µg/kg in yellow thermoplastic striping. PCB-11 dominated the yellow paint sample with the greatest total PCB concentration, making up 98 percent of the total PCBs in the sample, and comprised 79 percent of the total PCBs in the thermoplastic sample. This congener accounted for 17 percent or less of the total PCB content of the less-contaminated paint samples, which were comprised of

more heavily chlorinated congeners (such as PCB-77 and PCB-209). PCB-35 and PCB-209 were also present in significant amounts in yellow paint. Analysis of a dried yellow paint sample showed that lower-chlorinated congeners volatilized during drying.

White traffic paint samples analyzed by the City of Spokane contained total PCB concentrations ranging between 0.28 and 0.41 µg/kg, the white thermoplastic striping sample contained 3.33 µg/kg total PCBs. In contrast to results for the yellow paint samples, there were no discernible differences in congener patterns for the white wet and dried samples. PCB-11 was present at greater concentrations in the white paints than the yellow paints, and the PCB-209 concentration was similar in both types. Titanium dioxide, a known source of heavily chlorinated congeners PCB-206, PCB-208, and PCB-209, constituted 3 to 7 percent and 7 to 13 percent of the yellow and white paint samples, respectively. Both the yellow and white thermoplastic striping samples contained primarily lower-chlorinated PCBs; the yellow type contained more dichlorinated PCBs, and the white type contained a greater variety of congeners and more of the tri- and tetrachlorinated congeners. Both thermoplastic paints exhibited homolog and congener patterns similar to Aroclor 1016 (City of Spokane 2015).

The City of Spokane also sampled and analyzed two other types of paint frequently used near roadways – fire hydrant paint and utility locate paint. Total PCBs detected in the fire hydrant paint (Rustoleum Professional High Performance Enamel Fast-Dry spray paint in Silver Aluminum) were 0.0032 µg/kg and consisted only of congener PCB-19 (a trichlorobiphenyl). Total PCBs in a green-colored utility locate paint (Rustoleum Industrial Choice Solvent-Based Precision Line inverted marking paint in safety green) were 21.527 µg/kg, with PCB-209, PCB-6, PCB-5/8, and PCB-11 being the greatest contributors to the total.[5] The most likely source of PCBs in the spray paint is the pigment and is most likely a phthalocyanine green based on the presence of PCB-11, PCB-206, PCB-207, PCB-208, and PCB-209. Congeners PCB-206, PCB-208, and PCB-209 may have been contributed by titanium dioxide in the marking paint (City of Spokane 2015). A summary of results from the City of Spokane study is provided in Table 5-3.

Ecology conducted a study of PCBs in general consumer products in 2014 (Ecology 2014c). Products tested included packaging, paper products, paints and paint colorant, caulks, and a miscellaneous category consisting of two printer inks and two food samples. Although the samples were analyzed for all PCB congeners, the initial report published in June 2014 discussed only four congeners: PCB-11, PCB-206, PCB-208, and PCB-209. In this study, 68 products were tested for PCBs. Several products were separated into more than 1 sample, and 74 samples were submitted for analysis. Almost all paint and colorant samples tested contained one or more PCB congeners at detectable levels. PCB-11 was found in 66 percent of the samples tested. PCB concentrations in an additional 2 percent of the samples were estimated to be near the reporting limit and likely to contain PCB-11. PCB-206 and PCB-208 were not present in most of the products tested. One sample contained both PCB-206 and PCB-208, while seven contained PCB-209. One product, a phthalocyanine green-based colorant used to color white paint, contained detectable levels of all four PCB congeners and is the only sample to contain PCB-206 and PCB-208 at detectable levels.

[5] Congeners are listed in order from highest to lowest contribution to the total PCB concentration.

Table 5-3. PCB Congener Concentrations in Traffic Paint

Product Type	Total PCBs (µg/kg)	Percent of Total from PCB-11	Percent of Total from PCB-77	Percent of Total from PCB-209	Other Significant Congeners[1]	Primary Constituent Homolog Groups[2]
Yellow Traffic Marking						
Ennis paint - wet[3]	0.73 – 2.69	7 – 17	35 – 58	8 – 36	35[4]	variable[5]
Ennis paint - dried	0.565	9	39	35	none	tetra-, deca-, di-
Promar	64.88	98	1	0	35	di-, tri-, tetra-
Thermoplastic	10.78	79	1	0	18, 5/8, 35	di-, tri-
White Traffic Marking						
Ennis paint - wet[3]	0.4 – 0.41	18 – 23	0	57 – 61	none	deca-, di-
Ennis paint - dried	0.38	17	0	69	none	deca-, di-
Promar	0.28	41	1	0	none	di-
Thermoplastic[6]	3.33	22	0	0	52/69, 22, 5/8	tetra-, di-/tri-
Other Traffic Paint						
Hydrant spray paint, silver	0.0032	0	0	0	19 (100 percent)	tri-
Utility marking paint, green	21.527	3[7]	0	79	6, 5/8, 208	deca-, di-

[1] Congener numbers separated by a slash indicate co-eluting congeners; congener constituents listed in order of decreasing abundance.
[2] Primary homolog constituents listed in order of decreasing abundance; groups listed with a slash were similar.
[3] The range represents the results of a sample and its replicate.
[4] Congener was detected in the replicate sample.
[5] Homolog patterns were varied between the sample and replicate.
[6] White thermoplastic tape was the only paint tested that resembled an Aroclor; its congener and homolog pattern resembled Aroclor 1016.
[7] Estimated from figure.
µg/kg = Micrograms per kilogram.
PCB = Polychlorinated biphenyl.
Source: City of Spokane 2015.

Silicone Rubber Tubing and Testing Equipment

Perdih and Jan (1994) discuss the formation of PCBs in silicone rubber. A total of 29 congeners were detected, with lower-chlorinated congeners dominating. Anezaki and Nakano (2015) cite this article, stating that "Perdih and Jan investigated the PCB contamination of silicone rubber and reported that the contamination occurs when m-dichlorobenzene undergoes dimerization and PCB-47, 68, and 51 are produced." A summary of the results from the Perdih and Jan study is provided in Table 5-4.

Table 5-4. PCB Congener Concentrations in Silicone Rubber

PCB Congener	Silicone Rubber (mg/kg)	PCB Congener	Silicone Rubber (mg/kg)
4/10	1.495	42/37	0.057
6	0.162	44	0.142
7/9	0.052	45	0.043
8	1.735	46	0.121
16/32	0.63	47/48/49	0.482
17	0.487	51/22	0.169
18	0.66	52	0.197
19	0.231	56/60	0.05
24	0.07	66	0.159
25	0.077	68	0.427
26	0.099	70	0.151
28	0.341	74	0.283
33	0.82	99	0.13
40	0.152	101	0.066
Total PCBs	9.76 mg/kg		

mg/kg = Milligrams per kilogram.
PCB = Polychlorinated biphenyl.
Source: Perdih and Jan 1994.

Anezaki and Nakano (2015) examined inadvertent PCBs in silicone-based adhesives and chlorophenylsilanes as a source of contamination in environmental samples. These researchers had previously conducted atmospheric testing for PCBs and discovered that adhesives used in their high-volume air sampler were contaminated with lower-chlorinated PCB congeners, which created irregular results in their air samples (Anezaki and Nakano 2013, 2015). Total PCBs in glue samples they analyzed ranged from non-detect to 40 mg/kg and were comprised of mono- and dichlorobiphenyl congeners.

Silicone is manufactured from organochlorosilanes. Chlorophenylsilanes are intermediate substances in the manufacturing of phenyl silicone. Anezaki and Nakano (2015) analyzed several varieties of chlorophenylsilanes for PCBs: trichlorophenylsilane (TCPS), dichlorodiphenylsilane (DCDPS), chlorotriphenylsilane (CTPS), and diphenylsilanediol (DPSDO). Total PCBs in the various compounds tested varied widely within and among types.

For TCPS, all congeners detected were monochlorobiphenyls; the concentrations ranged from 0.0081 to 2.7 mg/kg. Total PCBs in DCDPS were comprised primarily of mono- and dichlorobiphenyl congeners, although some contained small amounts of trichlorobiphenyls, and one sample had relatively small amounts of tetrachlorobiphenyls. Total PCBs in DCDPS ranged from 6.5 to 1,500 mg/kg. Samples of CTPS contained total PCBs ranging from 0.019 to 1.1 mg/kg. The homolog compositions varied widely in this group, with one sample containing only monochlorinated congeners, two containing mono- and dichlorinated congeners, two samples containing mono- through trichlorinated congeners, and one containing mono- through

tetrachlorinated congeners. Total PCBs in three DPSDO samples analyzed ranged from 0.12 to 120 mg/kg. The least-contaminated sample contained only three congeners, PCB-1, PCB-2, and PCB-3. The two more heavily contaminated samples contained mono- and dichlorinated congeners, and one contained one trichlorinated congener. PCB-11 was identified in both of those samples.

Anezaki and Nakano (2015) concluded "the congener patterns of the silicone-based adhesives contaminated with PCBs were similar to those of DCDPS and DPSDO, indicating that the adhesive was produced from phenyl silicone. The PCBs included in these chlorophenylsilanes had low chlorine content at high vapor pressures. It can be inferred that the processes of manufacturing, commercializing phenyl silicone, and using the products at high temperatures most probably led to their release into the atmospheric environment."

Motor Vehicle Fluids

Motor vehicles dripping fluids in the roadway is one of the most common illicit discharge complaints received by the City of Spokane (City of Spokane 2015). As part of PCB source tracing efforts, the City of Spokane tested a wide variety of materials used in motor vehicles with the potential to contain PCBs, which can become sources of contamination to the Spokane River via runoff. In 2011, the City of Spokane tested off-the-shelf motor oils and transmission fluid to assess the potential for PCBs to commingle with stormwater through this pathway (City of Spokane 2013). PCBs were detected in each of the samples tested. Total PCBs in motor oil ranged from 14 to 116 µg/kg, and the single transmission fluid sample contained 8.8 µg/kg. Tests of new and used motor oil sampled in 2014 found total PCBs ranging from 0.5 to 2.375 µg/kg. Motor lubricant sampled in 2014 contained 0.623 µg/kg total PCBs. In the 2014 samples, there was a wide range of PCB congener distribution for the various oil and lubricant samples. Most of the congeners were in the low- to mid-chlorinated range. Synthetic motor oil contained more than 60 percent dichlorobiphenyls.

The City of Spokane tested regular unleaded gasoline and #2 dyed diesel from fuel tanks at the City's Riverside Park Water Reclamation Facility. Co-extracting interferences during laboratory analysis of the diesel caused high detection limits (2 µg/kg) for the mono-, di-, and trichlorinated PCBs in that sample. PCBs were not detected, but could have been present at concentrations less than the detection limit of 2 µg/kg.

One antifreeze product containing a yellow colorant (Kool Green Extended Life) was analyzed by the City of Spokane. Total PCB concentration in the sample was 0.018 µg/kg. Penta-, hexa-, and heptachlorobiphenyl congeners were detected. PCB-11, often found in yellow pigments, was not detected in this product. A summary of the results from the City of Spokane study is provided in Table 5-5.

Table 5-5. PCB Congener Concentrations in Motor Vehicle Fluids

Product Type	Range of Total PCBs in Products Tested (µg/kg)	Primary Constituent Congeners[1,2]	Primary Constituent Homolog Groups[3]
Gasoline, regular unleaded	0.935	2 (>99 percent)	mono-
Diesel, #2 dyed	ND[4]	ND	ND
New motor oil 2011 study	14 – 116	not provided	di-, mono-, tri-
New motor oil 2014 study - conventional	0.856	--	di-, hexa-, penta-
New motor oil 2014 study - synthetic	0.969	--	di-, tri-, tetra-, penta-
Used motor oil	0.502 – 2.375	variable[5]	variable[6]
Lubricant	0.623	--	di-, tri-
Transmission fluid	8.8	not provided	tri-, di-, mono- tetra-, penta-
Antifreeze	0.018	132/161, 180, 95, 89, 136/148	hexa-, penta-, hepta-

[1] Congener numbers separated by a slash indicate co-eluting congeners; congener constituents listed in order of decreasing abundance.
[2] Dash indicates congener not specified in text and could not be determined from figure in the report (i.e., peak not labeled).
[3] Primary homolog constituents listed in order of decreasing abundance; groups listed with a slash were similar.
[4] Not detected; co-extracting interferences during laboratory analysis raised the detection limits to 2 µg/kg for each of the mono-, di-, and trichlorinated congeners.
[5] Two samples of used motor oil were taken; congener composition varied widely between the two samples; figure did not label all major constituents.
[6] Two samples of used motor oil were taken; one contained congeners from three PCB homolog groups and the replicate contained congeners from seven homolog groups.
µg/kg = Micrograms per kilogram.
ND = Not detected.
PCB = Polychlorinated biphenyl.
Source: City of Spokane 2015.

Asphalt and Related Products

The City of Spokane (2015) analyzed samples of three asphalt products for PCBs: asphalt tack, crack sealer, and an asphalt release agent. Total PCBs ranged from 0.085 µg/kg in the asphalt tack to about 8 µg/kg in the crack sealer. The congener and homolog patterns of the crack sealer resembled Aroclor 1242 and were dominated by tri-, tetra-, di-, and pentachlorinated congeners, in that order. PCB-11 was present in all three products, but was the dominant congener only in the asphalt release agent. A summary of the results from the City of Spokane study is provided in Table 5-6.

Table 5-6. PCB Congener Concentrations in Asphalt-Related Products

Product Type	Total PCBs (µg/kg)	Primary Constituent Congeners[1]	Primary Constituent Homolog Groups[2]	Aroclor Similarity?[3]
Asphalt tack	0.085	5/8	di-, tri-, tetra-, mono-, hexa-	
Crack sealer	7.975	31, 28, 18, 5/8, 52/69, 44, 11	tri-, tetra-, di-, penta-	1242
Asphalt release agent	0.558	11	di-, tri-/tetra-, mono-	

[1] Congener numbers separated by a slash indicate co-eluting congeners; congener constituents listed in order of decreasing abundance.
[2] Primary homolog constituents listed in order of decreasing abundance; groups listed with a slash were similar.
[3] If the source report indicated similarity of the sample congener or homolog profiles to an Aroclor congener profile, the Aroclor is noted.
µg/kg = Micrograms per kilogram.
PCB = Polychlorinated biphenyl.
Source: City of Spokane 2015.

Other Materials

Three roadway deicer samples were analyzed by the City of Spokane (2015), with total PCBs ranging from 0.038 to 1.95 µg/kg. Magnesium chloride deicer (used in most western Washington municipalities) samples were dominated by tetrachlorinated biphenyls. The deicer used by Washington State Department of Transportation Eastern Region (an enhanced salt brine with sugar beet boost) had the lowest total PCB concentration and was dominated by hexachlorinated biphenyls, with some contribution from tri-, penta-, and heptachlorinated biphenyls, in that order. A yellow-tinted antifreeze tested by the City of Spokane contained 0.018 µg/kg total PCBs. PCB-11 was not detected in the sample, despite the yellow color.

Three herbicides were tested by the City of Spokane. No PCBs were detected in Weedar 64 (2,4-dichlorophenoxy acetic acid formula). Portfolio 4F (a broadleaf and sedge herbicide) contained 6.89 µg/kg total PCBs, primarily as congeners PCB-64 and PCB-72, but with some other congeners (PCB-47/48, PCB-90, PCB-91, PCB-99, and some lesser contributors). Total PCBs detected in a sample of Roundup Pro Max were 0.012 µg/kg. The only congener discernible for the Roundup sample in the report figure showing pesticide congener profiles was PCB-4. A deposition and drift management agent used with pesticides and herbicides, called Crosshair, contained 0.316 µg/kg total PCBs, with the primary congener constituents PCB-18, PCB-99, and PCB-181.

Three dust suppressants approved for use in the City of Spokane to control dust on unimproved roads were analyzed: a petroleum-based product (emulsified asphalt dust abatement [EADA]), a magnesium chloride-based product (Dust Guard), and a lignosulfonate-based product (Ligno Road Binder). EADA and Ligno Road Binder contained similar concentrations of total PCBs: 0.091 and 0.086 µg/kg, respectively. EADA was comprised of the dichlorobiphenyl congeners PCB-11 and PCB-19, while the Ligno Road Binder was comprised of heavier penta-, hexa-, and heptachlorobiphenyls, including the co-eluting congeners PCB-132/161 and PCB-138/160. The magnesium chloride Dust Guard contained 3.574 µg/kg total PCBs, primarily as congeners PCB-50 and PCB-53.

Samples of five types of personal care products were also analyzed in the City of Spokane study. These included hand soap, laundry detergent, dish soap, shampoo, and toothpaste. All contained measurable amounts of PCBs. PCB-11 was the dominant congener in the laundry soap and shampoo samples, but was not detected in the hand soap, dish soap, or toothpaste samples. Predominant congeners in the hand soap were PCB-44, PCB-18, and PCB-22, and in the toothpaste were PCB-22, PCb-95, and PCB-194. A summary of the results from the City of Spokane study is provided in Table 5-7.

Table 5-7. PCB Congener Concentrations in Other Materials

Product Type	Total PCBs (µg/kg)	Primary Constituent Congeners[1,2]	Primary Constituent Homolog Groups[3]	Aroclor Similarity[4]
Roadway Deicer				
Magnesium chloride	1.332 – 1.952	78, 53, 59, 74, 50	tetra-	
SB Boost	0.038	--	hexa-, tri-, penta-, hepta-	
Herbicides/Adjuvants				
Portfolio 4F	6.89	64/72, 90, 47/48	tetra-, penta-	
Roundup Pro Max	0.012	4	mono-	
Crosshair adjuvant	0.316	18, 99, 181	tri-, penta-, hepta-	
Dust Suppressants				
Emulsified asphalt dust abatement (EADA)	0.091	11, 19, 70	di-, tri-, tetra-	
Lignosulfonate	0.086	138/160, 132/161	hexa-, penta-, hepta-, tri-	1016 H
Magnesium chloride	3.574	50, 53, 11	tetra-, di-, tri-	
Hydroseed				
Hydroseed mix (green colored)	2,509	44, 70, 31, 52/69	tetra-, tri-, penta-, hexa-, hepta-	1248 H
Pipe and Pipe Repair Materials				
PVC (ASTM 3034) pipe, new	1.999	209, 56, 77, 6	tetra-, di-, deca-, tri-	
Cured-in-place pipe liner	1.110	--	tri-, tetra-, penta-, di-, hexa-	
Shortliner	17.780	118, 70, 52/69, 110	penta-, tetra-, hexa-, tri-, di-	1242/1254 C
Firefighting Materials				
Firefighting foam	0.029	153, 20/33, 61, 30, 95	tri-, penta-/hexa-	
Cleaners/Degreasers				
Hotsy Super XL detergent	0.003	36 (only)	tri- only	
Simple Green degreaser	0.068	11 (>50 percent)	di-, tetra-/hexa-, penta-/hepta-, tri-	

Table 5-7. PCB Congener Concentrations in Other Materials

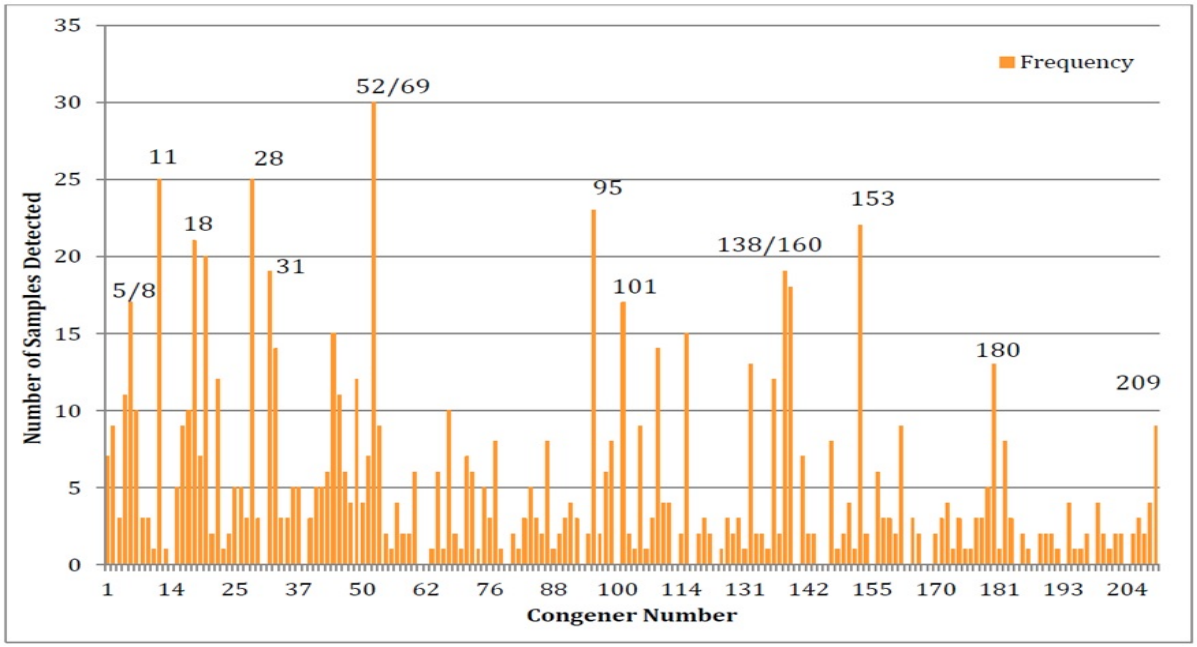
Product Type	Total PCBs (µg/kg)	Primary Constituent Congeners[1,2]	Primary Constituent Homolog Groups[3]	Aroclor Similarity[4]
Personal Care Products				
Hand soap (pomegranate and tangerine)	0.037	44, 18, 22	tri-, tetra-	
Tide laundry detergent	0.174	11, 31, 18, 60	di-, tri-, tetra-	
Dawn Ultra antibacterial dish soap	0.083	43/49, 18, 5/8	di-, tetra-, tri-, mono-	
Suave Naturals shampoo	0.058	11	di-, tri-, tetra-	
Aquafresh extreme clean whitening toothpaste	0.032	22, 95, 194	penta-, hexa-, tri-/octa-, nona-	

[1] Congener numbers separated by a slash indicate co-eluting congeners; congener constituents listed in order of decreasing abundance.
[2] Dash indicates congener not specified in text and could not be determined from figure in the report (i.e., peak not labeled).
[3] Primary homolog constituents listed in order of decreasing abundance; groups listed with a slash were similar.
[4] If the source report indicated similarity of the sample congener (C) or homolog (H) profiles to an Aroclor congener profile, the Aroclor is noted.
ASTM = American Society for Testing and Materials.
µg/kg = Micrograms per kilogram.
PCB = Polychlorinated biphenyl.
PVC = Polyvinyl chloride.
Source: City of Spokane 2015.

Figure 5-9 from the City of Spokane (2015) illustrates the frequency of detection of PCB congeners in the samples analyzed for the study. The figure includes all of the material types discussed in the previous sections.

Diesel engine exhaust is another source of PCBs to the environment. Diesel engines power trucks and other machines, such as generators. Chang et al. (2014) analyzed emissions from a heavy-duty diesel engine burning diesel or diesel fuel blended with cooking-oil based biodiesel. In this study, the chlorine content of the biodiesel was 3.4 and 4.6 times higher than conventional gasoline and diesel fuel, respectively, and the aromatics (benzene family hydrocarbons) content was lower than commercial gasoline and diesel fuel. The mean mass of PCBs in diesel engine exhaust declined with increasing percentage of biodiesel. Mean mass of PCBs in picograms/N cubic meter (pg/N m^3) (N means at normal temperature and pressure, 25 degrees Celsius at sea level) was 267 in diesel fuel, 210 in fuel blended with 10 percent biodiesel, and 103 in fuel blended with 20 percent biodiesel. The samples were analyzed for the 12 World Health Organization (WHO) toxic congeners (for more information on these congeners, see Section 6.1). Dominant congeners were PCB-118 (40 percent), PCB-105 (22 percent), and PCB-77 (13 percent).

Figure 5-9. Frequency of Detection of PCB Congeners in Commercial Products



Source: City of Spokane 2015.

The EPA National Vehicle and Fuel Emissions Laboratory in Ann Arbor, Michigan, tested a 2008 model year, 280-horsepower, turbocharged diesel engine for PCB formation with and without after-treatment (Laroo et al. 2011). After-treatments tested included diesel oxidation catalyst, catalyzed diesel particulate filter, copper zeolite urea selective catalytic reduction, iron zeolite, and ammonia slip catalyst. The research paper focused on the WHO toxic congeners; although, homologs were measured and reported in the supporting information. These researchers stated that "for a modern diesel engine, both with and without after-treatment, PCB emissions are near zero levels." It is unclear, however, whether this claim was based on the WHO toxic congeners, rather than all congeners, considering the considerably higher concentrations of non-WHO PCBs detected. Table 5-8 shows the PCBs emitted per liter of fuel burned with and without the various treatments.

Table 5-8. Average PCB Emissions for Steady State Diesel Engine Tests (in pg/L fuel consumed)

	Engine Out		CuZ SCR HT		CuZ SCR LT		FeZ SCR		DOC+CDPF	
Compound	AVG	SD	AVG	SD	AVG	SD	AVG	SD	AVG	SD
PCB-77	23.3	29.4	66.3	88	273.3	93.6	61.4	20.9	2.9	2.8
PCB-81	ND	--	ND	--	85.3	45.6	ND	--	ND	--
PCB-105	430.2	452.9	971.2	540.3	1130	827.7	334.3	157.5	13.7	4.9
PCB-114	ND	--	ND	--	147.4	100.7	20.1	7.3	0.4	--
PCB-118	1255	502.1	1998	1,227	3408	3,146	858.8	324.2	36.1	10.3
PCB-123	ND	--	ND	--	146.8	78.1	8.5	11.7	13.5	5

Table 5-8. Average PCB Emissions for Steady State Diesel Engine Tests (in pg/L fuel consumed)

Compound	Engine Out		CuZ SCR HT		CuZ SCR LT		FeZ SCR		DOC+CDPF	
	AVG	SD	AVG	SD	AVG	SD	AVG	SD	AVG	SD
PCB-126	ND	--	ND	--	98.8	83.1	ND	--	ND	--
PCB-156/157	ND	--	95.1	207	349.8	145.3	41.1	14.9	5	2.6
PCB-167	ND	--	ND	--	161.9	84.5	15.4	10.2	3.1	2
PCB-169	ND	--	ND	--	170.5	57.8	ND	--	ND	--
PCB-189	ND	--	ND	--	140.2	39.9	ND	--	ND	--
Total Mono-CBs	1,664	2,509	61,803	17,596	36,825	7,306	1645	642	142.8	113
Total Di-CBs	31,646	90,527	12,061	4,440	16,314	10,069	7,626	3,290	2,151	705.2
Total Tri-CBs	10,218	6,994	18,017	7,088	23,859	17,082	11,122	3,385	6,248	1,497
Total Tetra-CBs	96,796	135,509	164,598	193,800	122,638	83,942	36,256	22,078	290,355	73,062
Total Penta-CBs	20,121	11,417	28,126	15,218	57,384	61,160	14,959	3,169	1,451	447.7
Total Hexa-CBs	10,173	7,516	11,205	7,378	26,396	25,116	5,655	1,226	511.3	124.9
Total Hepta-CBs	2,796	3,401	2,645	1,371	7,590	6,518	1,859	531.2	299.2	153.3
Total Octa-CBs	221.2	880.3	125.6	191.3	1,397	951.9	324.1	127.9	27.8	15.2
Total Nona-CBs	18.8	77.5	ND	--	338.3	153.8	34.2	35.8	ND	--
PCB-209	ND	--	ND	--	192.8	51.4	1.6	3.6	ND	--
WHO 2005 TEQ (ND=0)	0.05	--	0.1	0.1	15.2	10	0.04	--	0.002	0.001
WHO 2005 TEQ (ND=DL/2)	24.5	5.5	8.8	3.6	17.9	8.4	2.5	0.6	0.06	0.03
WHO 2005 TEQ (ND=DL)	48.9	11	17.46	7.2	20.7	7	4.86	1.1	0.12	0.05

AVG = Average.
CB = Chlorinated biphenyl.
CDPF = Catalyzed diesel particulate filter.
CuZ = Copper zeolite.
DL = Detection limit.
DOC = Diesel oxidation catalyst.
FeZ = Iron zeolite.
HT = High temperature.
LT = Low temperature.
ND = Not detected.
PCB = Polychlorinated biphenyl.
pg/L = Picograms per liter.
SCR = Selective catalytic reduction.
SD = Standard deviation.
TEQ = Toxic equivalency quotient.
WHO (World Health Organization) 2005: As published in Van den Berg et al. 2006.

5.2 Fate and Transport of Polychlorinated Biphenyls

In Washington and elsewhere, PCBs with three to six chlorines (tri- through hexachlorobiphenyls) are the dominant compounds found in environmental samples.

5.2.1 Transport and Partitioning

Once in the environment, PCBs do not readily break down and may remain for very long periods of time. They can easily cycle between air, water, and soil. PCB transport refers to the movement of PCBs from one environmental compartment to another (such as from soil to air). PCB fate refers to where PCBs end up after they have been released to the environment (e.g., bound to soils, dissolved in water or air, etc.).

The fate of a chemical in the environment is a function of its chemical and physical properties. The chemistry terms in the text box below are important in understanding the fate and transport of PCBs in the environment.

Specific gravity, boiling point, and viscosity increase as the chlorine content of the PCB congener increases, while the water solubility and vapor pressure decrease. The boiling point, log K_{ow}, and bioconcentration factors (BCFs) generally increase with increasing chlorine content across the homolog groups. Vapor pressure, solubility in water, and volatilization show a corresponding decrease by 6, 4, and 7 orders of magnitude. The chemical properties of the 10 PCB homolog groups are summarized in Table 5-9.

Figure 5-10 from the International Agency for Research on Cancer (IARC 2015) shows how K_{ow} generally increases with increasing chlorine content of the PCB congener.

Brief Explanation of Some Chemistry Terms

Vapor pressure, or equilibrium vapor pressure, is defined as the pressure exerted by a vapor in thermodynamic equilibrium with its condensed phases (solid or liquid) at a given temperature in a closed system. The equilibrium vapor pressure is an indication of a liquid's evaporation rate. It relates to the tendency of particles to escape from the liquid (or a solid). A substance with a high vapor pressure at normal temperatures is often referred to as volatile.

Water solubility is the extent to which a compound will dissolve in water. The log of solubility is generally inversely related to molecular weight (i.e., the heavier a molecule is, the less soluble it will be in water).

K_{ow} is an indirect measure of the polarity of a substance. Octanol is a substance that is very similar in polarity to the fats and other materials found in living tissue. It is only slightly soluble in water. If a substance is added to a mixture of octanol and water and mixed thoroughly, the substance will distribute itself into both of these solvents to some extent. The octanol-water partition coefficient is then defined as the ratio of the concentration of that substance in the octanol to its concentration in the water. In general, more polar materials will have relatively high water solubility and will, therefore, have a relatively low value for K_{ow}. Relatively less-polar materials are characterized by a high value of K_{ow}. This parameter models how substances will distribute themselves between the fatty tissues of living organisms and water (or aqueous solutions, such as blood or urine). Log K_{ow} is the natural log of the octanol-water partition coefficient.

Table 5-9. Chemical Properties of PCB Homolog Groups

Congener Group	Molecular Weight	Vapor Pressure (Pa)	Water Solubility (g/m^3)[1]	Log K_{ow}
Monochlorobiphenyl	188.7	0.9 – 2.5	1.21 – 5.5	4.3 – 4.6
Dichlorobiphenyl	223.1	0.008 – 0.60	0.06 – 2.0	4.9 – 5.3
Trichlorobiphenyl	257.5	0.003 – 0.22	0.015 – 0.4	5.5 – 5.9
Tetrachlorobiphenyl	292.0	0.002	0.0043 – 0.001	5.6 – 6.5
Pentachlorobiphenyl	326.4	0.0023 – 0.051	0.004 – 0.02	6.2 – 6.5
Hexachlorobiphenyl	360.9	0.0007 – 0.012	0.0004 – 0.0007	6.7 – 7.3
Heptachlorobiphenyl	395.3	0.00025	0.000045 – 0.000	6.7 – 7
Octachlorobiphenyl	429.8	0.0006	0.0002 – 0.0003	7.1
Nonachlorobiphenyl	464.2	--	0.00018 – 0.0012	7.2 – 8.16
Decachlorobiphenyl	498.7	0.00003	0.000001 – 0.000	8.26

[1] Values for heptachlorobiphenyl and decachlorobiphenyl are likely not zero, but are listed as presented in Ritter et al. 1995.

g/m^3 = Grams per cubic meter. PCB = Polychlorinated biphenyl.
Pa = Pascals. Source: Ritter et al. 1995.

Figure 5-10. Relationship Between K_{ow} and Chlorine Content

Source: IARC 2015.
Note: BZ numbers[6] are equivalent to the polychlorinated biphenyl congener numbers used in this report.

[6] BZ numbers are the Ballschmiter and Zell PCB numbering scheme that the International Union of Pure and Applied Chemistry (IUPAC) adopted (with minor adjustments). Ballschmiter and Zell published the first papers that discussed the analysis of PCB congeners.

The discussions below are based on the EPA Advance Notice of the PCB Reassessment (EPA 2010a, 75 FR 17645) summary of information from the Agency for Toxic Substances and Disease Registry (2000) PCB toxicological profile and are supplemented with Washington State information from the 2015 PCB Chemical Action Plan (Ecology 2015a) and other summaries (e.g., Eisler 2000; EPA Region 4 2013).

Air

PCB levels in the atmosphere have been decreasing slowly since the late 1970s when EPA began restricting their use. However, in some regions of the U.S., recent studies have shown that PCB levels in air are no longer declining (e.g., Chicago [Venier et al. 2012] and New Jersey [Rodenburg 2015a]). PCBs can be transported long distances in air and have been found in snow and seawater in areas far away from where they were released into the environment (e.g., in the Arctic). As a consequence, PCBs are found all over the world. Most of the PCBs in air come from volatilization of PCB-contaminated soil and surface water. In general, the less chlorinated the type of PCB, the further it may be transported from the source of contamination. Therefore, highly chlorinated PCBs tend to stay closer to the source of contamination. PCBs in the atmosphere are primarily associated with the gaseous phase, with only about 10 percent adsorbed to particulates, especially the higher-chlorinated forms.

PCBs are removed from the air by settling as dust or in rain or snow. This is referred to as atmospheric deposition. According to Ecology (2015a), "Atmospheric deposition is the dominant source of background PCBs to most soil and water *surfaces*[7]." King County recently completed an air deposition study at seven stations in the Green-Duwamish River watershed (King County 2013b, 2015a). Station locations included four in the LDW area, two in downtown Kent, and one in Enumclaw. Median fluxes of total PCBs ranged from 0.35 nanograms per square meter per day (ng/m^2/day) in Enumclaw to 205 ng/m^2/day in the Georgetown neighborhood of the LDW. PCB-129 was a dominant congener at Beacon Hill, Duwamish, Georgetown, South Park, and Kent Senior Center (SC), with PCB-20 and PCB-61 co-dominating at the Georgetown and South Park stations. PCB-110 was the dominant congener in samples at Kent and Enumclaw. Other significantly contributing congeners[8] include PCB-52 (Enumclaw), PCB-147 (Beacon Hill, Duwamish, and Kent), PCB-153 (Beacon Hill, Duwamish, Kent SC, and Enumclaw), and PCB-180 (Beacon Hill, Duwamish, Kent, and Kent SC). PCB-11, although not discussed in the report or labeled on the report figures, averaged approximately 2 percent or greater contribution to total flux at all stations except Beacon Hill. Figures showing the congener profiles for each of the seven stations are presented in Appendix A.

Water/Sediments

In water, PCBs may be transported by currents, attach to bottom sediment or particles in the water, and evaporate into air. Sediments that contain PCBs can also release the PCBs into the surrounding water. In general, PCBs are relatively insoluble in water, and the solubility decreases with increased chlorination. PCBs in the water column may be freely dissolved or bound to dissolved or particulate organic carbon, dissolved organic matter, other particles, or

[7] Emphasis added.
[8] Congener labeled on congener profile figure and among the highest contributing congeners identified for the station.

colloids (review in Howell 2012). PCBs leave the water column by partitioning onto sediments and suspended particulates and by volatilization at the air/water interface. The results of field studies suggest that the partitioning behavior of PCBs in the water column is location specific. For example, the presence of some types of dissolved organic carbon can affect the solubility of PCBs and, therefore, PCB concentrations in water (Chiou et al. 1987; Howell 2012).

Organic sediments adsorb PCBs onto fine surface particles. Currents move those particles to slower-moving sections of water, where the particles fall out of suspension and deposit on the bottom. Flood events can move sediment-associated PCBs onto land.

Assessments of both Puget Sound (Ecology 2011b) and freshwater systems (Ecology 2011a, 2015a) have found that, statewide, the largest pathway for PCBs to reach the aquatic environment is stormwater. Direct air deposition was estimated to be the second largest pathway in the Puget Sound and Lake Washington study (Ecology 2011b; King County 2013a)[9]. There is a large and variable amount of PCB loading to freshwater systems from unknown pathways. PCB levels in Washington's marine and fresh waters have decreased substantially since peaking in the 1970s.

It was estimated that approximately 97 percent of the total mass of PCBs currently in the aquatic ecosystem of Puget Sound is contained in the active sediment layer (top 10 centimeters [cm]), and less than 1 percent is stored in the water column (Ecology 2015a).

Soil

Atmospheric deposition is the dominant source of PCBs to most soil. PCBs adhere strongly to soil and will not usually be carried deep into the soil with rainwater. An exception to this is when PCB contamination is associated with chlorinated solvent contamination. Because PCBs are freely soluble in non-polar organic solvents (such as benzene and toluene), PCBs can be transported through soils contaminated with these solvents to deeper soils or to groundwater. PCBs do not readily break down in soil and may stay in the soil for months or years[10]; generally, the more chlorine atoms that the PCBs contain, the more slowly they break down. Evaporation appears to be an important way by which the lighter PCBs leave soil.

With the exception of site-specific determinations for contaminated sites, the PCB background concentration in Washington soils has not been well characterized. PCB levels in U.S. background soils generally average from several hundred to several thousand parts per trillion (ppt) dry weight (DW) (Hornbuckle and Robertson 2010).

Biota

PCBs are taken up into the bodies of small organisms and fish in water. They are also taken up by other animals that eat these aquatic animals as food. PCBs accumulate in fish and marine

[9] This result is similar to what has been observed in New York Harbor, where stormwater contributions of PCBs dominate in urban areas and atmospheric deposition dominates in the New York/New Jersey Bight where there is a large surface area (Rodenburg 2015b).

[10] An evaluation of movement of PCBs through the Delaware River watershed (Rowe et al. 2007) found that only about 3 percent of the PCBs atmospherically deposited to soil exited the watershed via the river's tributaries. Volatilization of PCBs from the soil back to the atmosphere, particularly of low molecular weight congeners, was extensive in the watershed.

mammals (such as seals and whales), reaching levels that may be many thousands of times higher than in water. PCB levels are highest in animals high up in the food chain. Less-chlorinated PCBs are generally more water soluble, more volatile, and more readily metabolized (biodegraded) and eliminated from organisms. Highly chlorinated PCB congers are often more resistant to degradation and volatilization and tend to sorb more strongly to particulate matter. Some highly chlorinated PCBs tend to bioaccumulate to greater concentrations in tissues of animals than less-chlorinated congeners. These more heavily chlorinated congeners can also biomagnify in food webs. However, there are some highly chlorinated congeners that have specific structures that make them susceptible to metabolism by such species as fish, crustacea, birds, and mammals (Beyer and Biziuk 2009).

Compared to historical levels, PCBs have declined in Puget Sound harbor seals (1972 to 1997) and killer whales (1993 to 1995 versus 2004 to 2006) that inhabit or transit Puget Sound (Calambokidis et al. 1999; Hickie et al. 2007; Krahn et al. 2007). While PCB levels in Puget Sound fish are probably 10 times lower than they were in the 1970s, levels in most species tested have not declined in the past 20 years[11] (Puget Sound Partnership 2013). It was estimated that less than 3 percent of the total mass of PCBs currently in the aquatic ecosystem of Puget Sound is stored in the biota (Ecology 2015a).

Table 5-10 provides estimated half-lives for a series of congeners representing each of the 10 PCB homolog groups in water, soil, sediments, and air. An estimated BCF is also provided. These values are predicted, modeled values from EPA's Persistent, Bioaccumulative, and Toxic (PBT) Profiler (EPA 2012). EPA's PBT Profiler defines the BCF as "… a measure of the ability for a water-borne chemical substance to concentrate in fatty tissue of fish and aquatic organisms relative to its surroundings. EPA defines bioconcentration as the net accumulation of a substance by an aquatic organism as a result of uptake directly from the ambient water through gill membranes or other external body surfaces (60 FR 15366)." With respect to aquatic organisms, bioavailable PCBs are generally considered to be those that are freely dissolved in the water column and what is present in the prey for higher-trophic organisms (Howell 2012). PCBs bound to particles and colloids are less bioavailable. The BCF can be expressed as the ratio of the concentration of a chemical in an organism to the concentration of the chemical in the surrounding environment, usually the water. A BCF greater than 1 is indicative of a hydrophobic or lipophilic chemical that does not dissolve or poorly dissolves in water. It is an indicator of the probability that a chemical will bioaccumulate in organisms. The greater the BCF, the more likely a chemical is to bioaccumulate.

Table 5-10. Estimated Half-Lives of Representative PCB Congeners in Environmental Media

PCB	Congener Number	CAS Number	Half-Life (days)				BCF
			Water	Soil	Sediment	Air	
4-Chlorobiphenyl	PCB-3	2051-62-9	38	75	340	4.2	510
3,3'-Dichlorobiphenyl	PCB-11	2050-67-1	38	75	340	3.9	5,400
2,3,4'-Trichlorobiphenyl	PCB-22	38444-85-8	60	120	540	15	6,700

[11] In the Hudson River, PCB declines leveled off after around 1990 (Rodenburg 2015b).

Table 5-10. Estimated Half-Lives of Representative PCB Congeners in Environmental Media

PCB	Congener Number	CAS Number	Half-Life (days)				BCF
			Water	Soil	Sediment	Air	
2,3',5,5'-Tetrachlorobiphenyl	PCB-72	41464-42-0	180	360	1,600	13	27,000
2,2',4,4'5-Pentachlorobiphenyl	PCB-99	38380-01-7	180	360	1,600	13	40,000
2,3,3',4,4',5-Hexachlorobiphenyl	PCB-156	38380-08-4	180	360	1,600	75	26,000
2,3,3',4,4',5,6'-Heptachlorobiphenyl	PCB-190	41411-64-7	180	360	1,600	130	12,000
2,2',3,3',4',5,5',6'-Octachlorobiphenyl	PCB-199	52663-75-9	180	360	1,600	290	5,900
2,2',3,3',4,4',5,6,6'-Nonachlorobiphenyl	PCB-207	52663-79-3	180	360	1600	370	2,900
Decachlorobiphenyl	PCB-209	2051-24-3	180	360	1,600	880	12,000
Washington PBT Characteristics			> 60	> 60	> 60		> 1,000

> Greater than.
BCF = Bioconcentration factor.
CAS = Chemical Abstracts Service.
half-life = The amount of time it takes for the concentration of a chemical to diminish to half its original value.
PBT = Persistent, bioaccumulative, and toxic.
PCB = Polychlorinated biphenyl.
Source: Ecology 2015a.

Food is the main source of exposure to PCBs for humans not occupationally exposed. Humans accumulate PCB compounds primarily through the ingestion of high-fat foods (e.g., dairy products, eggs, animal fats, and some fish and wildlife). In some populations, such as Asian Pacific Islanders and Native Americans, higher concentrations of PCBs in the blood have been linked to higher rates of fish consumption (Xue et al. 2014).

5.2.2 Transformation/Degradation

The ability of PCBs to be degraded or transformed in the environment depends on the degree of chlorination of the biphenyl molecule as well as on the isomeric substitution pattern (i.e., where the chlorines are on the benzene rings). The most heavily chlorinated PCB congeners (e.g., hepta-, octa-, nona-, and decachlorobiphenyls) tend to be less susceptible to dechlorination than the less-chlorinated congeners (e.g., tri-, tetra-, penta-, and hexachlorobiphenyls) (Battelle Memorial Institute et al. 2012).

Although very stable in the environment, the major pathways for degradation (ATSDR 2000) are:

- Vapor-phase degradation with hydroxyl radicals.
- Photolysis in water.

- Aerobic biodegradation (preferentially less-chlorinated congeners).
- Anaerobic microbial degradation (more highly chlorinated congeners favored).

These are discussed by environmental medium below.

Air

PCBs in the atmosphere undergo complicated reactions primarily with hydroxyl radicals. The hydroxyl radical can be created by multiple pathways in the atmosphere, such as when water absorbs sunlight and separates into hydroxyl (●OH) and hydrogen (●H) radicals, or when sunlight splits ozone (O_3) molecules and water reacts with the oxygen atom that is released. Reactions with hydroxyl radicals are most prevalent. A radical is an atom or chemical that has a net charge of zero (neither negative nor positive), but has less than the preferred number of electrons in its outer shell. This instability causes a radical to be very reactive (ATSDR 2000).

Water

In water, photolysis is the primary pathway for degradation, as other more common reaction mechanisms (such as hydrolysis and oxidation) do not appear to contribute substantially. In these reactions, a carbon-to-chlorine bond absorbs energy from sunlight and separates into PCB and chlorine radicals. The PCB radical reacts with water, forming a stable PCB compound, but with one less chlorine atom. This reaction is particularly important for the highly chlorinated PCBs, as the more chlorines are present, the easier it is to cleave a carbon-to-chlorine bond. In highly chlorinated PCB molecules, cleavage occurs preferentially on the ring with the most chlorine atoms (ATSDR 2000).

Sediment and Soil

PCBs in sediment and soil degrade under both aerobic and anaerobic conditions, but aerobic degradation is faster. No abiotic process is known that significantly degrades PCBs. Both bacterial and fungal species have been shown to biodegrade PCBs using aerobic processes. PCBs with one to four chlorine atoms are most likely to be degraded under aerobic conditions because they are more likely to have two adjacent open (not chlorinated) positions. Degradation occurs via a two-step process. First, one of the two benzene rings is oxygenated and separated from the other ring. The remaining benzene ring is left as a chlorobenzoic acid. This combined process is called co-metabolism. After co-metabolism has occurred, the remaining chlorobenzoic acid is further broken down into water and carbon dioxide (mineralization) in a series of reactions that continually add oxygen to the compound. Because PCBs with fewer chlorine atoms degrade faster, a fractionating effect is created where less-chlorinated species biodegrade first, while those with higher levels of chlorine atoms are left behind for long-term buildup in the environment (ATSDR 2000). Different pathways may favor chlorine in specific positions on the PCB molecule. Bacteria generally remove chlorines at the meta and para positions, but not the ortho positions. This results in a less-toxic mixture. Removal of ortho chlorines could convert ‘benign’ congeners into dioxin-like congeners (Rodenburg 2015b).

Demirtepe et al. (2015) identified 93 anaerobic dechlorination pathways in a long-term (500-day) sediment microcosm study. The study examined sediments spiked with Aroclor 1260 as the initial source of PCBs. The highest rates of dechlorination during the first 185 days were seen for the pentachlorobiphenyls. In general, congeners having fewer than five chlorine atoms

(PCB-16/32, PCB-22/51, PCB-49, PCB-64/68/71/72, PCB-52/73, and PCB-67/100) had accumulated in the sediment at the 500th day. Major congeners that were common intermediates of dechlorination pathways were PCB-49, PCB-92, PCB-101, PCB-137, PCB-146, and PCB-180. The dominant terminal products of anaerobic dechlorination of the parent Aroclor 1260 were PCB-32, PCB-49, PCB-51, PCB-52, PCB-72, PCB-73, and PCB-100. These congeners did not appear as the parent congener in any anaerobic dechlorination pathway. Demirtepe et al. (2015) note that the potential toxicity of the sediment, as defined by the presence of dioxin-like congeners (see discussion of dioxin-like PCBs and toxicity in Section 6.1), was reduced by the degradation.

Biota

Mammals: In humans, the less-chlorinated PCB congeners generally tend to have shorter residence times because they are biotransformed and eliminated faster than the more-chlorinated congeners. However, a 2013 analysis of the structural properties that influence the persistence of PCBs in humans found that less-chlorinated congeners are not always biotransformed faster than higher-chlorinated compounds (Megson et al. 2013). PCB-28 (a trichlorobiphenyl) appears to be more resistant to biotransformation than PCB-101 and PCB-110, both pentachlorinated biphenyls. The study determined that congeners with chlorine atoms in the 2,5- and 2,3,6-positions appear to be more susceptible to biotransformation; whereas, congeners with chlorine bonds in the 2,3,4-; 2,4,5-; 3,4,5-; and 2,3,4,5- positions appear to be more persistent. For a detailed discussion of PCB metabolism in humans and other mammals, including the enzyme systems involved in biotransformation, please refer to the recently released IARC monograph on PCBs and polybrominated biphenyls (IARC 2015) and the review paper by Grimm et al. (2015).

Fish: Rodenburg et al. (2015) note that absorption, distribution, metabolism, and excretion processes can alter PCB congener patterns in fish. Sturgeon and whitefish from the Columbia River near the Hanford Superfund Site exhibit congener patterns consistent with Aroclor weathering, thus suggesting potential PCB metabolism in these species. Notably, depleted congeners (PCB-52, PCB-44/47, PCB-70/74, PCB-87, PCB-101, PCB-110, and PCB-118) relative to Aroclor 1254[12] in these fish were not substituted at adjacent positions, but often the meta/para or ortho/meta positions, where cytochrome P450[13] (CYP) oxidation may occur. PCBs that increased in concentration compared to the original Aroclor (referred to as "enhanced" PCBs; these include PCB-153, PCB-180, and PCB-187) have substituted adjacent sites where oxidation does not occur. Fish whose congener patterns indicated little or no metabolism of PCBs were bass, carp, sucker, and walleye.

A laboratory study of PCB metabolism in rainbow trout demonstrated that these fish can biotransform a number of PCB congeners to a hydroxylated form (OH-PCBs) (Buckman et al. 2006). Most of the congeners that were biotransformed in the fish are congeners that have vicinal (side-by-side on the same ring) hydrogen atoms in the meta and para positions with two or less ortho chlorines and/or congeners that have vicinal hydrogen atoms in the meta and para positions with three or more ortho chlorine atoms.

Birds: In general, highly chlorinated PCB congeners are metabolized to a lesser extent than less-chlorinated congeners. However, PCB congeners lacking vicinal chlorine substituents at

[12] Aroclor 1254 is the mixture responsible for most PCB contamination at the Hanford site.
[13] A family of enzymes.

meta and para sites (m,p-unsubstituted) on at least one phenyl ring are readily cleared and have lower biomagnification factors than congeners with vicinal chlorine atoms at these sites (meta-, para-substituted), independent of their degree of chlorination or log K_{ow} (Manning 2012).

Invertebrates: As reviewed by Koenig et al. 2012, a number of studies have reported different contaminant accumulation patterns between fish and crustacean species. In these studies, crustaceans exhibited a higher capacity to metabolize PCBs than fish. Koenig et al. (2012) examined variations in CYP enzyme[14] distribution and function in three species of deep-sea fish and a decapod crustacean and found a direct relationship between metabolic activities and PCB accumulation profiles. These researchers analyzed 41 PCB congeners in the muscle tissue of these organisms to investigate the potential relationship between biotransformation capacities and PCB bioaccumulation profiles. Their results indicated a marked difference in the presence and activities of CYP isoforms between fish and the crustacean. The crustacean accumulated significantly higher proportions of PCB-28, PCB-52, PCB-118, PCB-138, PCB-158, and PCB-169 than fish, indicating that this species lacks some of the biotransformation capacities that were seen in the fish. The crustacean also exhibited lower levels of PCB-87, PCB-149, PCB-153, PCB-170, PCB-180, PCB-183, PCB-194, and PCB-206, indicating that this crustacean is able to metabolize congeners that are also metabolized by mammals.

[14] CYP enzymes have many functions in humans and other animals, including the decontamination of potentially toxic compounds.

6.0 Environmental Effects of Polychlorinated Biphenyls

PCBs are persistent in the environment, build up in the food chain, and can cause adverse health effects in humans and wildlife, including cancer and harm to immune, nervous, and reproductive systems. PCBs disrupt thyroid hormone levels in animals and humans, hindering growth and development. PCB-induced toxicity is highly variable, with variability being attributed to:

- Differences between species in ability to metabolize PCBs and in primary sites of action (e.g., the thyroid or liver).
- Differences in the age, growth rate, biomass, and lipid content of the species.
- Differences in dose rate, duration of exposure, route of administration, and tested congeners.
- Differences in physico-chemical characteristics of the habitat during exposure.
- Differences in PCB interactions with other PCBs, other organochlorine compounds, and heavy metals (Eisler 2000).

6.1 Dioxin-Like Versus Non-Dioxin-Like Polychlorinated Biphenyls

The issue of rotation can have an impact on the relative toxicity of the PCB congeners. PCB congeners can either exist as planar, where the two benzene rings are in the same plane, or non-planar, where the benzene rings are at a 90-degree angle from each other (ATSDR 2000). Planar and non-planar PCBs can have very different toxicity, as described below (Ecology 2015a).

6.1.1 Dioxin-Like Polychlorinated Biphenyls

Some PCB congeners are called “dioxin-like” because their chemical structure is similar to 2,3,7,8 tetrachlorodibenzo-p-dioxin (TCDD; or often referred to as just “dioxin”). PCB congeners with at least four chlorines and with no chlorine atoms in the ortho positions can assume the planar (flat) conformation necessary for binding to the aryl hydrocarbon (Ah) receptor. These congeners do not have any chlorine atoms in the positions closest to the double bond between the benzene rings. The Ah receptor is a cellular receptor protein that binds planar organic compounds (e.g., polychlorinated dibenzo-p-dioxins and PCDFs) with high affinity, leading to various toxic effects. The most potent ligand (a signal-triggering molecule that binds to a site on a target protein) for the Ah receptor is TCDD. Toxicity is mediated through changes in gene transcription initiated by binding to the Ah receptor.

Subsequent to binding of the Ah receptor, there are changes in gene expression (e.g., induction of cytochrome p450 CYP1A1/1A2) leading to toxic responses. Induction varies by degree and pattern of chlorines and is the basis for the WHO TCDD TEFs for dioxins and dioxin-like PCB congeners. The TCDD TEFs reflect the potential relative potencies associated with binding to the Ah receptor, compared to that of TCDD. These have been reviewed and modified several times. Consensus TEFs for wildlife were developed in 1998 (Van den Berg et al. 1998). In 2005, WHO updated the TEFs for humans and mammals to replace the 1998 values (Van den Berg et al. 2006). The WHO Working Group harmonized the TEFs across different taxa as much as

possible, *but there are large differences in responses among different taxa*.[15] The dioxin-like PCB congeners include co-planar congeners (PCB-77, PCB-81, PCB-126, and PCB-169), which do not have any chlorine atoms in the ortho positions, and mono-ortho congeners (PCB-105, PCB-114, PCB-118, PCB-123, PCB-156, PCB-157, PCB-167, and PCB-189), which have a single chlorine atom in an ortho position.

The TEF reevaluation conducted in 2005, which led to the 2006 TEFs, concluded that more research on human systems was needed. Since the 2006 TEFs were published, newer research called into question the validity of directly extrapolating rodent-based TEFs in human risk assessment. In 2015, Larsson et al. published new consensus TEFs for rats and humans based on screening individual PCB congeners, using 17 human and rodent bioassays, to assess their induction of Ah-related responses relative to TCDD. The studied human cell types included primary keratinocytes[16], lymphocytes, and liver cells. The rodent cell types studied included rat cells (hepatocytes and hepatoma, liver epithelial, and lung epithelial cells), mouse/murine cells (hepatoma and primary splenic cells), and guinea pig intestinal adenocarcinoma cells. PCBs induced weak or negligible responses in all of the tested human systems; PCB-126 was the only congener that frequently evoked a response strong enough to calculate relative potency. The analysis of the new data confirmed the results of earlier studies showing that human cells are less sensitive to PCBs than rodent cells. The data also show the overall lower activity of PCBs in human cells.

TEFs for dioxin-like PCB congeners in fish, birds, and mammals are summarized in Table 6-1.

Table 6-1. TEFs of Dioxin-Like PCBs in Fish, Birds, and Mammals

PCB Congener	TEFs				
	Fish[1]	Birds[1]	Mammals/ Humans[2]	Consensus TEF[3] Rat	Consensus TEF[3] Human
77	0.0001	0.05	0.0001	0.0004	--[4]
81	0.005	0.1	0.0003	0.0002	--[4]
126	0.005	0.1	0.1	0.09	0.003
169	0.00005	0.001	0.03	0.002	--[4]
105	<0.000005	0.0001	0.00003	0.00001	--[4]
114	<0.000005	0.0001	0.00003	0.00006	--[4]
118	<0.000005	0.00001	0.00003	0.000009	--[4]
123	<0.000005	0.00001	0.00003	0.000009	--[4]
156	<0.000005	0.001	0.00003	0.00008	--[4]
157	<0.000005	0.0001	0.00003	0.00003	--[4]
167	<0.000005	0.00001	0.00003	0.000007	--[4]
189	<0.000005	0.00001	0.0003	0.000007	--[4]

[1] Van den Berg et al. 1998.
[2] Van den Berg et al. 2006.
[3] Larsson et al. 2015.
[4] No reported value due to inactivity of PCBs in the human bioassays. Larsson et al. also tested congener PCB-74, which has previously been found to be active toward the aryl hydrocarbon receptor and has been discussed for inclusion in the TEF scheme and found this congener was also inactive in humans.
PCB = Polychlorinated biphenyl. TEF = Toxic equivalency factor.

[15] Emphasis added.
[16] Keratinocytes are the primary cells of the epidermis, the outermost layer of the skin; these cells are involved in inflammatory immune responses.

6.1.2 Non-Dioxin-Like Polychlorinated Biphenyls

Non-dioxin-like (NDL) congeners are not good ligands for the Ah receptor; they contain chlorine atoms in the ortho positions on the benzene rings. These are also called "non-planar congeners." Because they have one or more chlorine atom(s) in the ortho positions (next to the carbon-carbon bond), the molecule is not "flat."

6.2 Modes of Polychlorinated Biphenyl Toxicity

The field of PCB toxicity research is vast. A search of Google Scholar[17] on the terms "polychlorinated biphenyl" plus "toxicity" and "restricted" to just the years 2014 and 2015 yielded nearly 2,800 results. Because this field is ever changing and expanding, it is not possible to cover every aspect of PCB toxicity in this report. Therefore, this section highlights some recent research and reviews on the topic of PCB toxicity. Of particular importance is the growing realization that some PCB metabolites, which have not been as well studied as the parent compounds, are biologically active, and that non-Aroclor PCBs (e.g., PCB-11) need additional research (e.g., as highlighted by Grimm et al. 2015). Research is showing that low molecular weight PCBs (e.g., PCB-11) are readily hydroxylated. These mono- and dichloro- PCBs often cannot be measured in biota because they would have already been hydroxylated, and measuring these metabolites is both difficult and not required by TSCA (Rodenburg 2015b).

The primary toxicity concern for dioxin-like PCBs in humans and laboratory animals (such as rats and mice) is carcinogenicity. The International Agency for Research on Cancer (IARC; 2015) recently completed an extensive review on the carcinogenicity of PCBs and determined that "there is sufficient evidence in humans for the carcinogenicity of PCBs. PCBs cause malignant melanoma. Positive associations have been observed for non-Hodgkin lymphoma and cancer of the breast." In laboratory animals, particularly rats, there is sufficient evidence that PCBs are carcinogenic.

Adverse effects reported in laboratory animals following exposure to individual NDL congeners (PCB-18, PCB-28, PCB-47, PCB-52, PCB-95, PCB-101, PCB-110, PCB-128, PCB-132, PCB-149, PCB-153, PCB-170, PCB-180, PCB-206, and PCB-209) were effects on the thyroid, liver, brain biochemistry, immunotoxicity, oestrogenicity, reproductive system, and neurodevelopment, in particular in the offspring of rodents following in utero exposure. NDL congener effects in fish include altered muscle coordination, depressed immune system with increased susceptibility to infections, and loss of fins and tails in flatfish.

The Scientific Panel on Contaminants in the Food Chain (2005) noted: "It is important to consider that very few of the commercial PCB mixtures were analyzed with respect to their precise composition or even their content of PCDF and dioxin-like-PCBs. Consequently, the information derived from studies with commercial products does not have full relevance for the toxicological evaluation of NDL-PCB." Note that the same can be said for dioxin-like PCBs—studies that used technical mixtures (i.e., Aroclors) dosed the organisms with dioxin-like and NDL congeners, plus whatever contaminants were in the mixture (e.g., PCDFs) that may preclude toxicological evaluation of the components. Similarly, studies that look for effects in

[17] <https://scholar.google.com/scholar?as_ylo=2014&q=%22polychlorinated+biphenyl%22+%2Btoxicity&hl=en&as_sdt=1,48>.

environmentally exposed subjects (whether human or ecological) are often confounded by the fact that these organisms are concurrently exposed to many other contaminants (e.g., mercury, polycyclic aromatic hydrocarbons, etc.). As an example, one human study (Bouchard et al. 2014) attempts to relate PCB levels in human blood to cognitive function in older individuals. Although mercury is a contaminant with known neurological impacts, it was not measured in the blood samples and, therefore, its possible confounding impact could not be assessed by the researchers.

While the dioxin-like PCBs have been recently shown to have little activity in human cells, some of these congeners are very toxic to fish and to some species of birds and mammals (e.g., rats, mink, and marine mammals). Fish are most susceptible in early life stages. There are other differences among species that affect PCB metabolism. For example, invertebrates lack the enzyme systems that react with dioxin-like PCBs. Adverse effects in fish attributable to dioxin-like PCB congeners include early life stage mortality (e.g., reduced hatchability in eggs) and immune system depression (ATSDR 2000; Duffy et al. 2002). A “no adverse effects” level was not identified. Adverse effects in birds attributable to dioxin-like PCB congeners include mortality; reduced egg hatchability and live births; reduced avoidance response; altered mating, reproductive, parenting, and nesting behavior; and suppression of immune response. Domestic chickens, in particular, appear to be very sensitive to PCB toxicity. Research is discussed below by mode of toxicity.

Cancer

Cancer and dioxin-like PCB exposure was discussed above. Regarding NDL PCBs and cancer, a critical review of this topic published in 2006 (Knerr and Schrenk), found that “technical PCB mixtures and individual [dioxin-like]-and NDL-PCBs act as liver tumor promoters *in rodents*[18]. Based on these data, a weak carcinogenic potency of individual NDL-PCB congeners cannot be excluded. In epidemiological studies, increased mortality from cancers of the liver, gallbladder, biliary tract, gastrointestinal tract, and from brain cancer and malignant melanoma were observed in workers exposed to a series of technical PCB mixtures. A significant association between PCB concentrations in adipose tissue and non-Hodgkin’s lymphoma was found in another study. While in all human studies mixed exposure to [dioxin-like]- and NDL-PCBs occurred, no comprehensive data are available on the relative contribution of NDL-PCBs to the overall external and/or internal PCB exposure in those cohorts.”

Immunotoxicity

In addition to cancer, PCBs have been shown to be immunotoxic. Dioxin-like PCBs can cause immunotoxic responses by binding to the Ah receptor, which is present in several tissues and cells of the immune system. However, individual congeners may antagonize each other’s effects by mechanisms that have not been fully elucidated. It is thought that some immunotoxic PCBs that are not Ah receptor active may exert their effect by their metabolism to intermediates that alkylate critical cellular macromolecules (IARC 2015).

[18] Emphasis added.

The inflammatory response (inflammation)[19] occurs when tissues are injured by bacteria, trauma, toxins, heat, or any other cause. The damaged cells release chemicals, including histamine, bradykinin, and prostaglandins. These chemicals cause blood vessels to leak fluid into the tissues, causing swelling. This helps isolate the foreign substance from further contact with body tissues. Inflammation is associated with the immune response. PCBs have been shown to initiate inflammatory responses in many tissue types. Sipka et al. (2008) performed an oral exposure study with mice, examining the induction of proinflammatory proteins of three different types of PCBs in multiple tissues (brain, liver, and lungs). The PCB congeners studied were PCB-77 (a co-planar dioxin-like PCB), PCB-104 (a non-co-planar NDL PCB with multiple ortho-chlorine constituents), and PCB-153 (a common non-co-planar NDL PCB). All three PCB types induced inflammatory responses within 24 hours after single oral doses, although not all proteins measured were induced. PCB-77 preferentially accumulated in the liver of exposed animals. PCB-153 accumulated in the liver and lungs about equally, but also in the brain to a lesser extent. PCB-104 levels were low in all tissues analyzed but appeared to be one of the most biologically active of the three congeners studied, indicating it may have had low absorption or was highly metabolized in this species.

Thyroid

Regarding the thyroid hormone (TH), Zoeller (2011) states: “Despite the universally held recognition that TH is required for brain development, the specific role of TH in brain development is incompletely understood at best. In part, the difficulty in understanding the role of TH in brain development is due to the complexity of the processes whereby biologically active TH is delivered to target cells and to the complexity of TH action on its receptors.” Further, “Given the complexity of TH action on development, it is not surprising that environmental chemicals that interfere with TH action will likewise have complex effects.” And molecular studies with PCBs and other chemicals “indicate that a number of chemicals to which the human population is routinely exposed during development can interfere both directly and indirectly with TH action, producing consequences that are not identical to thyroid disease itself.”

The following review on thyroid effects of PCBs is primarily from the critical review of PCB metabolism and metabolites by Grimm et al. (2015) and research by Grimm et al. (2013). OH-PCB metabolites have been identified as disruptors of thyroid homeostasis, and a clear relationship between elevated OH-PCB levels and decreased TH levels has been observed in animal and human studies. Grimm et al. state that most studies report negative correlations between PCB and TH levels and fewer studies indicate positive correlations, particularly between levels of serum PCB and concentrations of free TH. In addition, a few studies report no correlation between elevated plasma PCB and TH levels. These findings indicate that thyroid effects may not just be related to the extent of the exposure, but are in fact congener- or metabolite-dependent. Animal models clearly indicate a negative correlation between PCB concentrations and L-thyroxine levels. PCBs have been suggested to affect the thyroid homeostatic system at various stages in a congener-dependent manner, and there is increasing evidence for distinct roles of various classes of PCB metabolites. For example, a study of the effects of sulfated metabolites of five lower-chlorinated PCBs, including PCB-11, found that

[19] Description from the National Library of Medicine article on immune response; available online at: <https://www.nlm.nih.gov/medlineplus/ency/article/000821.htm>.

these compounds bind with high affinity to the TH transport protein transthyretin. Because transthyretin can transport TH across the placenta, the ability of PCB metabolites to bind to this protein suggests a possible mode of transmission of these compounds to the developing fetus.

Neurotoxicity/Blood Brain Barrier Effects

Research by Lee and Yang (2012) indicates that neurotoxicity caused by PCB exposure is independent of the Ah receptor induction pathway. This supports the observations by others that non-co-planar (i.e., NDL) PCBs produce more neuroactive responses than co-planar (i.e., dioxin-like) PCBs. Breakdown of the blood brain barrier is a commonality of several nervous system disorders, including Parkinson's disease, amyotrophic lateral sclerosis (Lou Gehrig's Disease), and non-Alzheimer-related dementia (review in Seelbach et al. 2010). An *in vivo* study with mice found that PCBs can affect the blood brain barrier by altering tight junction proteins (Seelbach et al. 2010). All three PCB types tested (dioxin-like congeners co-planar PCB-126, mono-ortho-substituted PCB-118, and NDL congener non-co-planar PCB-153) caused alterations in the integrity of brain capillary endothelium. Permeability was increased the most by PCB-118, followed by PCB-126 and PCB-153. Treatment with PCBs allowed melanoma cancer cells to migrate to the brain; potency followed the same pattern as the permeability study, with PCB-118 showing greatest tumor growth and PCB-153 showing the least, with PCB-126 in the middle. Their findings suggest that the different PCB congeners may be acting through different mechanisms to elicit the same functional effect on the integrity of the blood brain barrier. Developmental exposure of rats to NDL PCB-52 increased extracellular GABA in the cerebellum (European Commission 2010). GABA is an inhibitory neurotransmitter widely distributed in the neurons of the cortex that contributes to motor control, vision, and other functions. Increased GABA may have contributed to motor coordination impairment observed *in vivo*.

Oxidative Stress/Cell Damage

Zhu et al. (2013) found that a hydroxylated metabolite of PCB-11 induces oxidative damage in a human cell line *in vitro*. In this study, the PCB-11 4-hydroxyl metabolite induced alterations in steady-state levels of reactive oxygen species and cytotoxicity in human prostate epithelial cells. Elevated and/or chronic levels of reactive oxygen species (radicals) can cause oxidative stress and damage cell structures. Dihydroxylation reactions of some hydroxylated PCBs can generate catechols and other hydroquinone species that promote oxidative stress (review in Grimm et al. 2015).

Osteoarthritis

An *in vitro* study by Abella et al. (2015) showed that NDL PCBs (PCB-101, PCB -153, and PCB-180) could contribute to osteoarthritis (a type of joint disease that results from breakdown of joint cartilage and underlying bone). Chondrocytes are the cells that make up articular cartilage. Chondrocyte cell death causes damage to the cartilage. These NDL PCB congeners caused cell death of chondrocytes involving mechanisms of apoptosis, necrosis, and oxidative stress.

Estrogenic/Androgenic Activity

PCB congeners can be estrogenic or anti-estrogenic. *In vivo* research has shown lower-chlorinated congeners to be more estrogenic and higher-chlorinated congeners to be more anti-estrogenic. Effects can be caused by either binding to an estrogen receptor or increasing estrogen production. Some hydroxylated lower-chlorinated PCB metabolites have been found to have higher efficacy than estradiol, the primary female sex hormone. Hydroxylated PCB metabolites can also indirectly induce estrogenicity by inhibiting estrogen sulfotransferase, an enzyme which inactivates estrogens by sulfation (as reviewed in Grimm et al. 2015).

Researchers evaluating *in vitro* activity of NDL PCBs (via bioassays derived from human cell lines) found that all of the congeners tested showed anti-androgenic effects (Hamers et al. 2011). The highest androgen receptor-antagonistic potencies were found for PCB congeners, in order from greatest to least, PCB-168, PCB-125, PCB-19, PCB-122, PCB-104, PCB-128, PCB-51, PCB-126, PCB-47, PCB-136, and PCB-28. In fact, most of the PCBs tested had higher androgen receptor-antagonistic potency than the reference compound flutamide, an anti-androgenic drug used in prostate cancer treatment. IARC (2015) notes that "studies with cultured cells demonstrated that some PCBs are androgen-receptor antagonists, the anti-androgenic effects of dioxin-like PCBs being more pronounced than those of ortho-substituted PCBs."

Researchers evaluating the toxicity of NDL PCBs in rats found decreases in testosterone and increases in luteinizing hormone and follicle stimulating hormone (both female reproductive hormones) levels in PCB-180-treated male offspring (European Commission 2010). Decreased prostate weights and sperm counts were observed at the highest exposure level. The changes observed were suggestive of testicular damage and consistent with anti-androgenic activity seen for all NDL PCBs tested.

Embryotoxicity in Fish

High doses of dioxin-like PCBs, particularly PCB-126, have been shown to be embryotoxic to salmonid and other fish species, causing a syndrome called blue sac disease (Fitzsimons et al. 1999). This disease is characterized by induction of P4501A enzymes, developmental delay, craniofacial malformations, hemorrhages, pericardial and sac edemas, and death. This syndrome was observed in the Great Lakes with rainbow trout (*Oncorhynchus mykiss*) and lake trout (*Salvelinus namaycush*) in the mid-1900s when dioxin and dioxin-like compounds reached lethal concentrations.

In a comparison study, researchers exposed mummichogs (*Fundulus heteroclitus*), an estuarine minnow common on the mid-Atlantic coast, to TCDD; 2,3,4,7,8-pentachlorodibenzofuran; Aroclor 1254; dioxin-like congener PCB-77; and NDL congeners PCB-52 and PCB-100 (Rigaud et al. 2014). The 2,3,4,7,8-pentachlorodibenzofuran and PCB-77 caused embryotoxic responses in the mummichog similar to those observe with TCDD and PCB-126. Exposures to the NDL congeners caused no cranial malformations or edema and induced no significant behavioral alterations (as measured by prey capture ability, reaction to light stimulus, and locomotor activity). Prey capture ability was altered by all the dioxin-like compounds tested, suggesting that this response may be Ah receptor dependent.

6.3 Toxicity Summary

Dioxin-like and NDL PCBs can have different toxic effects due to their different physical structures. The main toxicity concern for dioxin-like PCBs has historically been cancer. However, dioxin-like PCBs are associated with other negative impacts (e.g., hormone disruption, embryotoxicity, and immunotoxicity). NDL PCB exposures in laboratory animals have been associated with effects on the thyroid, liver, brain biochemistry, immunotoxicity, oestrogenicity, reproductive system, and neurodevelopment. PCB metabolites (e.g., hydroxylated PCBs [OH-PCBs] and non-Aroclor PCB congeners) are likely important toxicants but have not been as well studied as the primary PCB constituents of the Aroclors.

7.0 Chemical Analysis of Polychlorinated Biphenyls

Prior to 2007, most environmental monitoring for PCBs was not designed to sample for all 209 PCB congeners but focused only on those that were produced intentionally. This means that environmental monitoring was likely missing detection of PCBs that are unintentional by-products (Grossman 2013; Grace 2015b). PCB detection methods have improved over time. Current methods provide detailed data on specific congeners, while earlier methods provided data on specific PCB mixtures and homologs. This improvement of analytical methods can prove challenging because it is often difficult to compare data over time as the methods do not provide comparable information at similar detection levels.

7.1 Measurement of Polychlorinated Biphenyls

Historically, analytical methods were developed to analyze for Aroclor mixtures or to determine values for total PCB concentrations. Recently, more-sensitive congener-specific analyses have been developed to enable more-detailed study of PCBs in the environment. Methods range from traditional gas chromatography (GC), more-recent high-resolution GC/mass spectrometry (GC/MS), to new immunoassay techniques. The three most commonly used analytical methods are screening, Aroclor, and congener-specific (Ecology 2015a).

The MTCA and SMS rules specify standard analytical methods and under what circumstances Ecology may require or approve alternate methods (Ecology 2015d). The MTCA rule specifies standard analytical methods and testing requirements for contaminated sites in WAC 173-340-830(3):

- For PCB mixtures, the standard analytical method is EPA Method 8082, which is included in EPA SW-846 (WAC 173-340-830[3][a][i]).

The SMS rule specifies standard analytical methods and testing requirements for sediment at contaminated sites in WAC 173-204-600(3). The rule references the method and requirements in the Puget Sound Estuary Protocols (2015), which are defined in WAC 173-204-200(21):

- For PCB mixtures, the standard analytical method is EPA Method 8082, which replaced EPA Method 8081 in EPA SW-846.
- For co-planar PCB congeners, the standard analytical method is EPA Method 1668.

MTCA and the SMS rule also provide for alternate methods (see Ecology 2015d for additional information). The various analytical methods are discussed below.

7.1.1 Screening Methods

Screening methods were developed to test wastes quickly and cheaply. Screening methods, such as Method 9077 and 9078, are effective if PCBs are the sole source of halogens in the material being tested, but provide inaccurate results if other chlorinated compounds are present. For example, Method 9077 only tests for total chlorine and cannot differentiate PCBs from other chlorinated compounds such as chlorinated solvents. In those instances where PCB contamination is known, Method 9078 provides a quick and easy method to determine the extent

of contamination and is often used as a screening tool to limit the number of samples sent to a laboratory for more-detailed analyses. Because screening methods do not provide reliable PCB quantitation, their use is not discussed further in this document.

7.1.2 Analysis of Aroclors

EPA developed specific methods to comply with TSCA and other applicable legislation. To meet the requirements of the 1976 Resource Conservation and Recovery Act, EPA developed *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*, more commonly known as SW-846 (EPA 2014b). Included in SW-846 are two specific methods for analyzing PCBs in a wide range of media:

- Method 8082A: Polychlorinated Biphenyls (PCBs) by Gas Chromatography[20]
- Method 8275A: Semi-volatile Organic Compounds (PAHs and PCBs) in Soils/Sludges and Solid Wastes Using Thermal Extraction/Gas Chromatography/Mass Spectrometry (TE/GC/MS)[21]

Method 8082A is the more traditionally used method; it is the successor to Method 8080, one of the earliest methods developed to meet regulatory requirements, and Method 8082. EPA's Contract Laboratory Procedures method for analyzing Aroclors in water, soil, or sediments (Aroclors Analysis) is based on EPA SW-846 Method 8082. The method includes sample extraction, extract cleanup techniques, and GC/electron capture detector (ECD) analytical methods for Aroclors. Methods 8080, 8082, and 8082A are responsible for much of the legacy data reported as Aroclor mixtures or specific PCB congeners identified in the method. Method 8082A is "… used to determine the concentrations of polychlorinated biphenyls (PCBs) as Aroclors or as [a selected subset of] individual PCB congeners in extracts from solid, tissue, and aqueous matrices, using open-tubular, capillary columns with electron capture detectors (ECD) or electrolytic conductivity detectors (ELCD)" (EPA 2007). The specific Aroclors reported by this method (Aroclor 1016, Aroclor 1221, Aroclor 1232, Aroclor 1242, Aroclor 1248, Aroclor 1254, and Aroclor 1260) and 19 congeners (PCB-1, PCB-5, PCB-18, PCB-31, PCB-44, PCB-52, PCB-66, PCB-87, PCB-101, PCB-110, PCB-138, PCB-141, PCB-151, PCB-153, PCB-170, PCB-180, PCB-183, PCB-187, and PCB-206) are detected in the parts per billion (ppb) to ppm levels depending upon the complexity of sample and matrix involved (Ecology 2015a).

For water samples, EPA Method 625, Methods for Organic Chemical Analysis of Municipal and Industrial Stormwater - Base/Neutrals and Acids, can be used to analyze for a number of organic compounds, including PCBs as Aroclors. EPA Method 608, Methods for Organic Chemical Analysis of Municipal and Industrial Stormwater - Organochlorine Pesticides and PCBs, is also used to analyze water samples for PCBs as Aroclors.

There are several possible problems associated with Aroclor analyses. First, Aroclor identification and quantitation can be highly dependent on subjective decisions made by the analyst reviewing the sample chromatograms and may provide only semi-quantitative total PCB

[20] Method 8082A is the currently used method and replaced Method 8080 (Organochlorine Pesticides and Polychlorinated Biphenyls by Gas Chromatography) and Method 8082.
[21] King County indicates this method is not commonly used and is not used by laboratories analyzing samples from the LDW.

concentrations when multiple Aroclors are present or weathering has occurred. Because the identification of Aroclors is based on 5 to 10 key congeners for each Aroclor, congeners not included in the quality control (QC) used during the analysis might not be identified (Grace 2015b) and lead to PCB totals that are biased low. Second, Aroclor analysis can be biased high because GC-ECD detects halogenated compounds, including non-chlorinated compounds (e.g., polybrominated diphenyl ethers). This could bias PCB estimates high (Grace 2015b). Third, Aroclor patterns may be transformed through selective transport of some congeners. For example, when an Aroclor is released to soil, the more-chlorinated congeners within the Aroclor will sorb to the soil and the more-soluble congeners will move down the soil column with pore water. Aroclor analysis may be imprecise or yield false-negative data for total PCBs when Aroclor patterns are weathered (EPA Region 4 2013). Therefore, Aroclor analyses should be avoided if weathering is expected at a site (Battelle Memorial Institute et al. 2012; EPA Region 4 2013). The quality of Aroclor data is based on extraction method, cleanups, experience of the analyst, and nature of the matrix being analyzed. King County laboratories note, however, that all analytical methods have uncertainty and Aroclors can perform reasonably well for a total PCB approach. The LDW RI and East Waterway Supplemental RI data also support this statement that Aroclors can be appropriate for total PCB quantification (Williston 2015).

7.1.3 Analysis of Congeners

Method 1668 was created to analyze PCBs in water, soil, sediment, biosolids, and tissue. It provides analytical results for "… the 12 polychlorinated biphenyls (PCBs) designated as toxic by the World Health Organization (WHO): congeners 77, 81, 105, 114, 118, 123, 126, 156, 157, 167, 169, and 189 [and] the remaining 197 CBs [chlorinated biphenyls], approximately 125 of which are resolved adequately on an SPB-octyl gas chromatographic column to be determined as individual congeners. During the laboratory analytical process, some congeners cannot be distinguished from one another and are quantified as a complex of more than one congener. These are known as co-eluting congeners. The remaining approximately 70 congeners are determined as mixtures of isomers (coelutions)" (EPA 2010b). Other columns resolve different congeners and have different co-eluting sets of congeners. This is discussed in Section 7.3.1.

Method 1668 requires the use of a high-resolution mass spectrometer (HRMS) for detection and is considerably more expensive than Method 8082. Method 1668, however, is becoming more common as concerns have been raised about PCBs from non-legacy sources and potential degradation products from legacy Aroclor mixtures. Detection limits for Method 1668 can be in the ppq to ppt levels depending upon the complexity of sample and matrix involved.

As noted above, EPA Method 1668 was developed to specifically determine the 12 WHO toxic congeners. It was published in March 1997 (EPA-821-R-97-001). In mid-1997, EPA began expanding the method for analysis of all 209 congeners. Revision A was published in December 1999 (EPA-821-R-00-002). Both the original method and Revision A were based on single laboratory testing and development. An interlaboratory study was conducted in 2003-2004 to validate Method 1668A, revise the method to reflect the study results, and to evaluate and revise the QC acceptance criteria. Of the 14 laboratories that were recruited for the study, 11 submitted data to EPA. Of those 11, only 6 submitted data EPA deemed as usable for wastewater, 6 submitted data usable for tissue, 4 submitted data usable for biosolids, and 4 submitted data usable for all three matrices (EPA 2010c). Version B was published in November 2008. This version replaced single-laboratory QC acceptance criteria with

interlaboratory criteria, and included other changes, but was replaced with Method 1668C in April 2010.

EPA used the results of the interlaboratory validation study of Method 1668A, a peer review of that study, user suggestions, and additional interlaboratory data to write version 1668C (EPA 2010b). The QC acceptance criteria were updated, and estimated method detection limits (EMDLs) and estimated minimum levels of quantitation were replaced with method detection limits (MDLs) and minimum levels. EPA issued a proposed rule in September 2010 to approve several new or revised analytical methods, including Method 1668C. Hundreds of comments were received on the proposal, including 35 comments on Method 1668C. Of these 35 comments, 5 were supportive of approving the method and 30 were critical of the method. Among the criticisms are issues with how the method handles background contamination and that soil and sediment matrices were excluded from the interlaboratory validation study. A summary of the criticisms of the method is included in the FR Final Rule (77 FR 29763). In the Final Rule, EPA acknowledged and stated "EPA is still evaluating the large number of public comments and intends to make a determination on the approval of this method at a later date." To date, however, Method 1668C has not been officially promulgated.

Analysis of Congener Subsets

Much of the sediment congener data collected in the LDW consists only of subsets of congeners (see study summaries in Section 8.2 and Table 8-1). In general, congener subsets can be considered "purpose-specific shortcuts" (Grace 2015b). Battelle Memorial Institute et al. (2012) note that "total PCB concentrations can be estimated by summing the individual PCB congener concentrations, if those congeners are expected to capture a sufficiently large proportion of the total PCB." Congener subset data can be used to analyze trends or to provide indicators of contamination but they are not suitable for modeling or mass balance, which require 'true' totals or all quantifiable congeners. The following are examples of PCB congener analyte lists in use historically or currently by various agencies:

- 12 Most Toxic Congeners (WHO): This subset consists of congeners PCB-77, PCB-81, PCB-105, PCB-114, PCB-118, PCB-123, PCB-126, PCB-156, PCB-157, PCB-167, PCB-169, and PCB-189, which are those that exhibit dioxin-like toxicity.
- 18 National Oceanic and Atmospheric Administration (NOAA) National Status and Trend (NS&T) Congeners: This subset consists of congeners PCB-8/5, PCB-18, PCB-28, PCB-52, PCB-44, PCB-66, PCB-101/90, PCB-118, PCB-105, PCB-128, PCB-138, PCB-153/132/168, PCB-170/190, PCB-180, PCB-187, PCB-195/208, PCB-206, and PCB-209[22]. These congeners were chosen for multiple reasons: (1) congener was already being quantified by other scientific organizations and laboratories, (2) toxicity, (3) ubiquitous in the marine environment, and (4) at the suggestion of leaders in the field of PCB quantitation (Lauenstein and Cantillo 1993). This subset has been used by NOAA to monitor temporal and geographic trends in the level of contaminants in coastal environments and in organisms, such as mussels and oysters. The PCBs analyzed have changed over time, with some congeners being added (e.g., PCB-169) and some not regularly reported (e.g., PCB-77) (Lauenstein and Cantillo 1993, 1998). Currently, 51 congeners are quantified as part of the program (Kimbrough et al. 2008). The program

[22] In the NOAA NS&T subset, co-eluting congeners are counted as a single congener.

has not used EPA methods, but rather, methods developed by laboratories for the program. The most recent method used for PCBs is Quantitative Determination of Chlorinated Hydrocarbons Using Gas Chromatography/Electron Capture Detection 2000-2005 (Kimbrough et al. 2007). Battelle Memorial Institute et al. (2012) note that "It has been shown that the 18 NOAA National Status and Trends Monitoring Project PCB congeners capture about 50 percent of the total PCBs in most U.S. coastal sediment environments, and summing the concentrations of those congeners and then multiplying that by 2 has been widely used to estimate the total PCB concentration in such sediments." For example, Lefkovitz et al. (2001) validated this method in lobster tissues. Also, an analysis of data from Ecology's National Pollutant Discharge Elimination System (NPDES) Inspection Sampling Support project (Leidos 2015a, 2015b, 2015c), which included PCB congener data from two laboratories, showed total PCBs varied from the NOAA 18 congener total by factors of 2.5 and 2.7 times. Both data sets had R-squared values above 0.99 (Alam 2015).

- McFarland and Clarke (1989) 36 priority PCB congeners: This older list of highest-concern congeners was based on potential toxicity, frequency of occurrence (based on the old methods, which did not measure all congeners), and abundance. McFarland and Clarke organized the 36 congeners based on known and potential toxicity. According to McFarland and Clarke, the Group 1 or highest priority congeners are those that are most likely to contribute to adverse biological effects due to their presence in environmental samples. Group 1 congeners fall into two classifications. Group 1A comprises congeners PCB-77, PCB-126, and PCB-169. Although these congeners have been reported rarely in environmental samples, and only in the ppt range, the very high individual toxicities of the three warrant special consideration. Group 1B are mono-ortho substituted congeners and have been reported frequently in environmental matrices, and most are relatively abundant as well (except for congener PCB-105, which was included because it has a high potential for toxicity). As a group, the congeners in Group 2 contributed 26 to 41 percent of total PCBs in the bird and mammal samples and 7 to 25 percent of total PCBs in fish and invertebrates. The congeners in Group 3 are important in terms of environmental prevalence and relative abundance in animal tissues, although they are not as biologically active. Group 3 congeners are most abundant in fish and invertebrate samples, collectively contributing from 20 to 48 percent of the total PCBs. Group 4 congeners may be of lesser significance in the environment but are toxicologically active. Individual congeners in Groups 2, 3, and 4 are listed in Figure 7-1 (from McFarland and Clarke [1989]). Note that the International Union of Pure and Applied Chemistry (IUPAC) numbers correspond to the PCB congener numbers used in this report.
- Seven Indicator Congeners (European): This subset consists of congeners PCB-28, PCB-52, PCB-101, PCB-118, PCB-138, PCB-153, and PCB-180. Boalt et al. (2013) note that the seven PCB congeners have been monitored since the beginning of the HELCOM and OSPARCOM monitoring programs[23]. These congeners were selected due to their relatively uncomplicated identification and quantification in gas chromatograms and because they usually contribute a very high proportion of the total PCB content in

[23] HELCOM is the Baltic Marine Environment Protection Commission; OSPARCOM is the OSPAR Commission, named for the original Oslo and Paris Convention against dumping that works to protect the marine environment.

environmental samples. These congeners indicate the presence/absence of PCBs. Note that the Norwegian Institute of Public Health used six indicator PCBs in its multi-laboratory comparison studies; PCB-118 was not included, likely because it is one of the WHO toxic congeners and was analyzed in a different suite of congeners in the studies.

Figure 7-1. McFarland and Clarke (1989) 36 Priority Congeners

IUPAC no.			
Group 1	Group 2	Group 3	Group 4
A	87[a,b]	18[a]	37[b]
77[a,b]	99[b]	44[a,b,c]	81[c]
126[b]	101[a,b,c]	49[a,b,c]	114[a,b,c]
169[b,c]	153[a,b]	52[a,b,c]	119
	180[a,b]	70[b]	123
B	183[a,b,c]	74[b]	157
105[a,b]	194[a,c]	151[a,c]	158[b]
118[a,b]		177[c]	167
128[a,b,c]		187[a,c]	168
138[a,b,c]		201[a]	189[a,b,c]
156[a,b]			
170[a,b,c]			

[a]Congeners included in Canadian Standard CLB-1. The remaining congeners making up CLB-1 are nos. 15, 31, 40, 54, 60, 86, 103, 121, 129, 137, 141, 143, 154, 159, 171, 173, 182, 185, 191, 195, 196, 200, 202, 203, 205, 206, 207, 208, and 209.

[b]Congeners suggested for inclusion in a selective congener analysis for human foodstuffs and tissues (*83*). Other congeners listed were 8, 28, 60, 66, 82, 166, 179, and 187.

[c]Identified as prevalent congeners that elute (or probably elute) as single-congener peaks from a single SE-54 glass capillary column using GC/ECD (*81*). Others listed were 24, 29, 26, and 84.

7.1.4 Analysis of Homologs

Analysis of PCBs by homolog groups can provide compositional information that can be useful for characterizing PCB contamination at a site (Battelle et al. 2012). This analysis generates PCB concentration data for each of the 10 levels of chlorination (see Figure 3-3). It can be used to reliably obtain total PCB data (Battelle Memorial Institute et al. 2012). Further investigation may then proceed to include high-resolution methods for specific congeners.

The Toxics Studies Unit of the Environmental Assessment Program at Ecology conducted a study of different methods to analyze PCBs in sediments (Ecology 2014b). EPA Method 8270D GC/low-resolution mass spectrometry (LRMS) 8081 modified by EPA 625 was used to analyze homologs. The detection limits using this method are higher than for the congener analysis described above, but lower than for Aroclors, and QC is performed at a higher level. This

method reports a PCB total for each homolog group. In sediment, MDLs are in the fractional ppb (µg/kg DW) range (i.e., between 0.1 and 1 ppb).

EPA Method 680, Determination of Pesticides and PCBs in Water and Soil/Sediment by Gas Chromatography /Mass Spectrometry, detects the presence of PCB homologs. EPA Region 4 initially proposed homolog analyses because the associated costs were typically lower than congener analysis. However, due to complications with Method 680, EPA Region 4 reconsidered recommendations for homolog analysis. In addition, private laboratories were running the congener analysis using EPA Method 1668B and then summing the congeners into the homolog groups and presenting the data as a homolog analysis. While this method is acceptable for reporting homolog data, the cost of the analysis is the same or higher than running the samples for congeners. Therefore, EPA Region 4 now considers only Aroclor and congener analysis (EPA Region 4 2013).

7.2 Data Comparisons and Translation

Ecology's Toxic Studies Unit (in the Environmental Assessment Program) conducted a study that examined high and low resolution methods for analyzing PCBs in sediments (Ecology 2014b). This is the only study identified in the literature that compared results of different analytical methods across the same samples, although there may be others. Ten archived sediment samples were split three ways and analyzed as congeners (EPA 1668, high resolution), homologs (EPA 8270D, low resolution), and Aroclors (SW-846 EPA 8082A, low resolution). Sample mean estimated detection limits for congeners averaged slightly more than 50 times lower than those reported for homolog analyses and about 400 times lower than Aroclors. Homolog sample average detection limits were about seven times lower than Aroclors (Table 7-1).

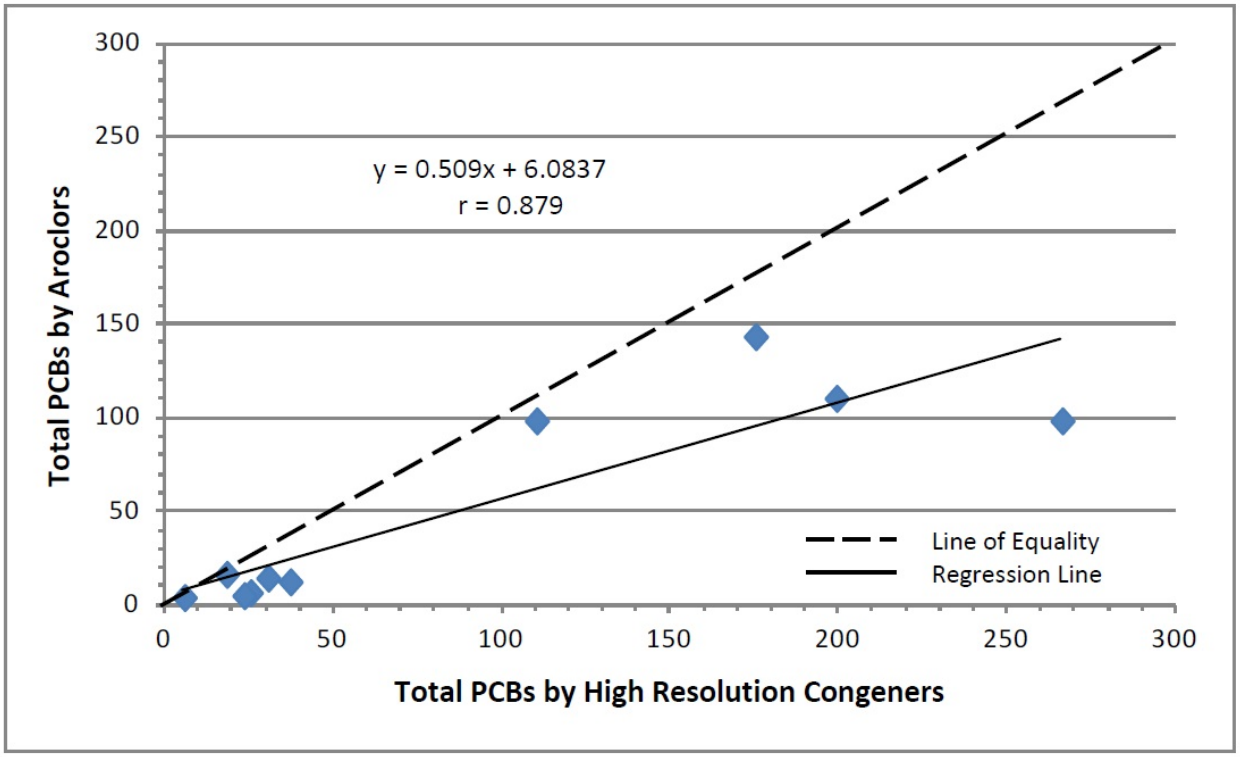
Table 7-1. Comparison of PCB Sediment Analyses by Low- and High-Resolution Methods

Analysis	Method	Estimated Cost per Sample	Estimated Mean Quantitation Limit	Estimated Mean Detection Limit	Percent Non-Detects[8]
Congeners	HRGC/HRMS[1] 1668C	$800 – 1200	0.498 ng/kg[4]	2.19 ng/kg[6]	10 percent (16/159)
Homologs	GC/LRMS[2] EPA Methods 8270D, 625, and 8081 A/B	$400 – 600	205 ng/kg[4]	123 ng/kg[6]	51 percent (82/160)
Aroclors	GC/ECD[3] SW-846, EPA 8082A	$225 – 350	5700 ng/kg[5]	904 ng/kg[7]	NA[9]

[1] High-resolution gas chromatography (HRGC)/high-resolution mass spectrometry (HRMS).
[2] Gas chromatography (GC)/low-resolution mass spectrometry (LRMS).
[3] GC/electron capture detector (ECD).
[4] The mean of sample estimated quantitation limits.
[5] The mean of sample reporting limits.
[6] The mean of sample estimated detection limits.
[7] The mean of sample method detection limits.
[8] Per sample mean percent of non-detected congeners (non-detected/total possible x 100).
[9] "Not Applicable" (NA); individual congeners not resolved by Aroclor analysis.
EPA = U.S. Environmental Protection Agency.
PCB = Polychlorinated biphenyl.
ng/kg = Nanograms per kilogram.
Source: Ecology 2014b.

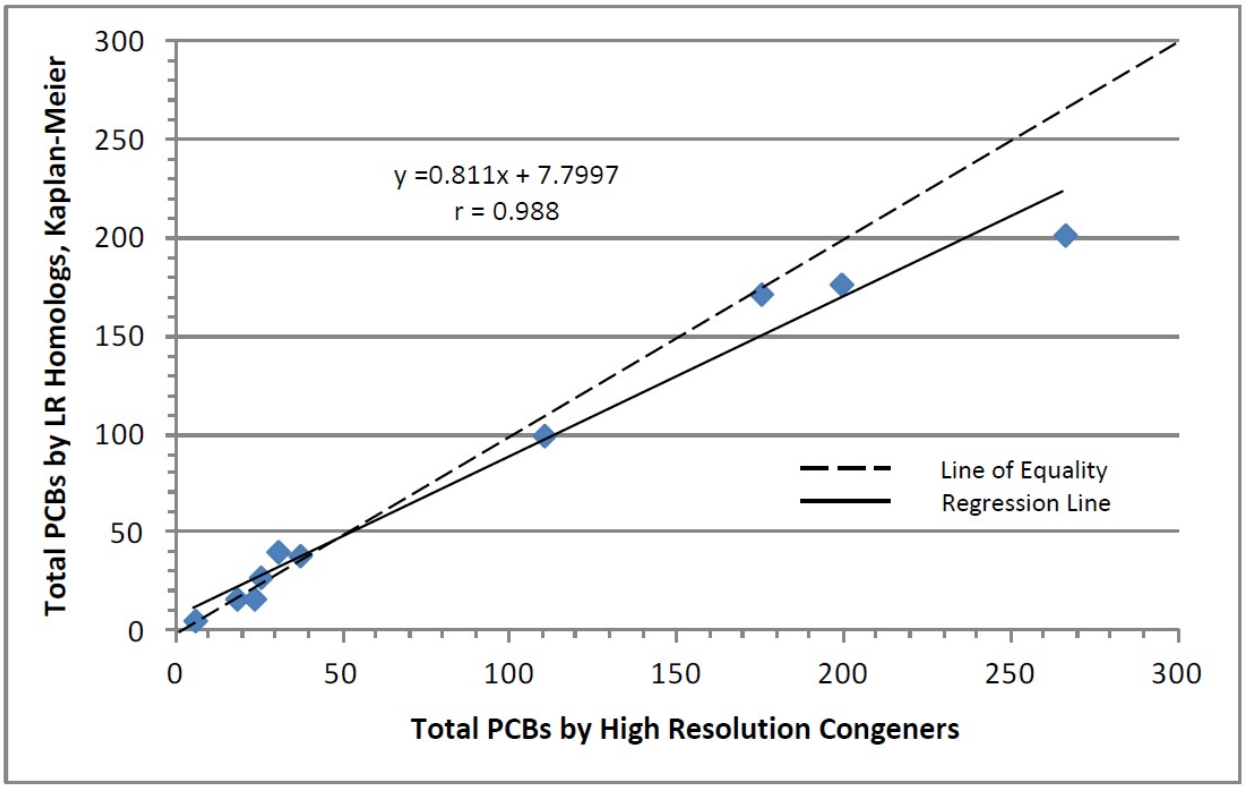
Based on this study, the high-resolution congener analyses reported few non-detected congeners. The low-resolution homolog method appeared to have a slight low bias at concentrations above 50 µg/kg, compared to the high-resolution congener method. Total PCB Aroclors compared to congeners showed a weaker relationship than homologs compared to congeners. Differences in total PCB concentrations measured by Aroclors compared to congeners or homologs reported relative percent differences (RPDs) averaging about 60 percent. In contrast, when total PCBs from congeners were compared to homolog methods, RPDs averaged about 13 percent. Study data suggest Aroclor analysis may be biased low when compared to either congeners or homolog analyses. Note that the Ecology author states that the sample size for the study was small, and "drawing definitive conclusions based on 10 measurements is not advised." Results are shown graphically in Figures 7-2 and 7-3.

Figure 7-2. Total PCBs by High-Resolution Congeners and Aroclor Analyses



Source: Ecology 2014b.

Figure 7-3. Total PCBs by High-Resolution Congeners and Kaplan-Meier-Adjusted Low-Resolution Homolog Analyses

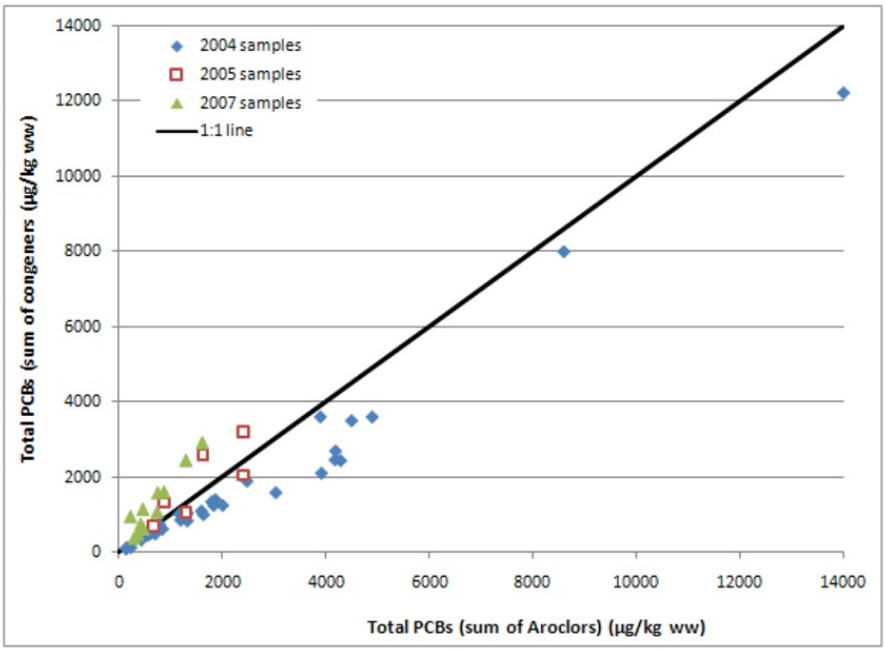


Source: Ecology 2014b.

The LDWI RI Report (Windward 2010b) compared total PCBs in fish and crab tissue samples calculated as sum of Aroclors and as sum of congeners. The sums of Aroclors ranged from 30 to 189 percent of the sums of congeners from the same samples, varying by year (Figure 7-4). For samples collected in 2004, the sums of congeners were less than the sums of the Aroclors. PCB congener sums in samples collected in 2005 were generally similar to Aroclor sums. For samples collected in 2007, PCB congener sums were consistently greater than the Aroclor sums.

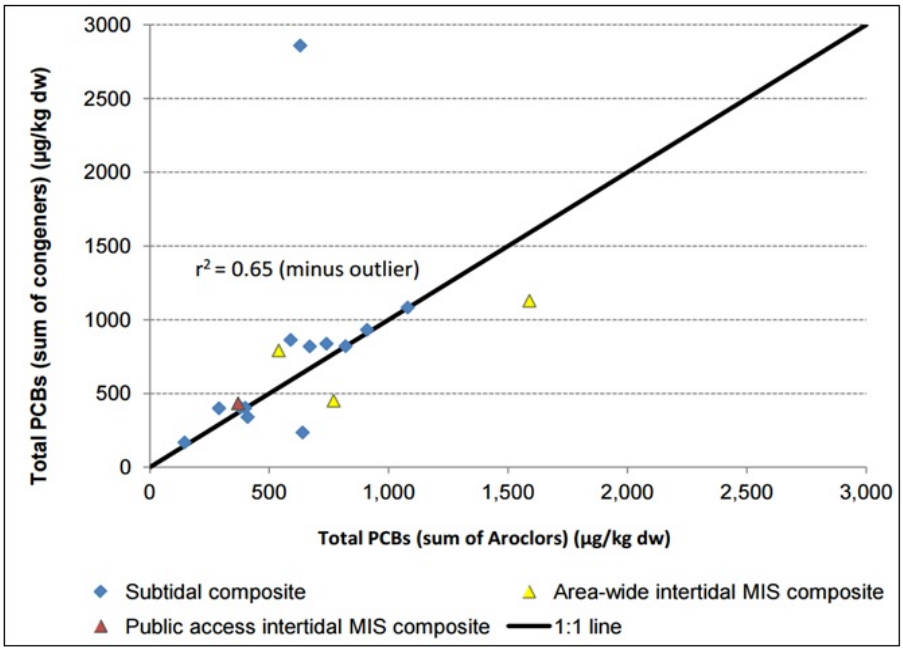
Similar comparisons of sums of congeners and sums of Aroclors were conducted during the East Waterway RI for sediment samples. In this study, total PCBs based on the sum of PCB Aroclors and the sums of PCB congeners were quite variable among the various sediment sample types (Figure 7-5). Comparisons of sums of Aroclors and sums of congeners for tissue samples in the East Waterway study were relatively similar for some species and tissue types (e.g., geoduck samples) but not for others (e.g., edible crab and clam tissue samples) (Figures 7-6 and 7-7), with the exception of some edible crab and clam tissue samples (seen in Figure 7-7).

Figure 7-4. Total PCBs in LDW Fish and Crab Tissue Samples – Comparison of Sums of Congeners and Sums of Aroclors



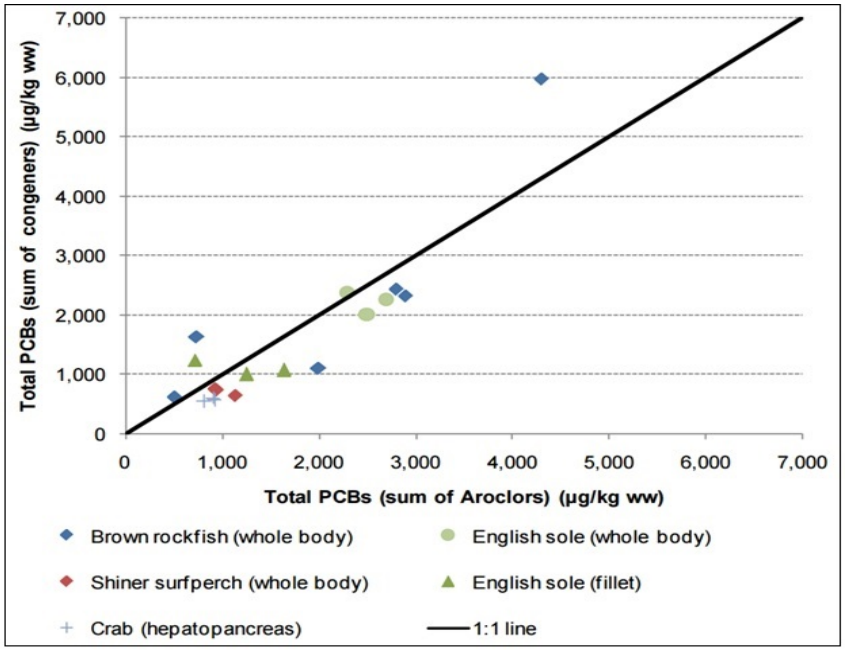
Source: Windward 2010b.

Figure 7-5. Total PCBs in Surface Sediment Samples – Comparison of Sums of Congeners and Sums of Aroclors



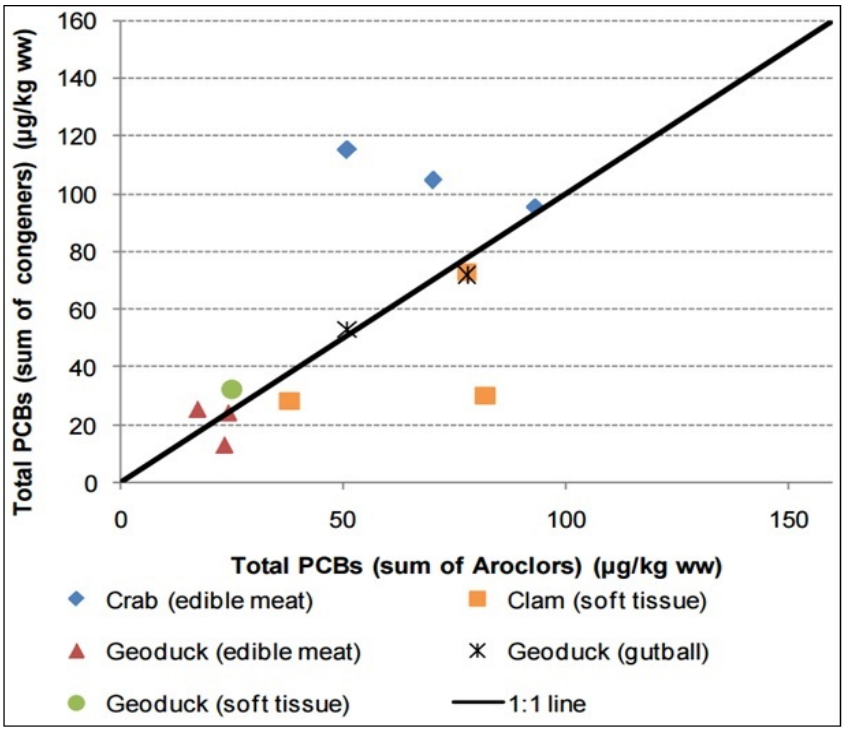
Source: Windward and Anchor QEA 2014.

Figure 7-6. Total PCBs in East Waterway Fish and Crab Samples – Comparison of Sums of Congeners and Sums of Aroclors



Source: Windward and Anchor QEA 2014.

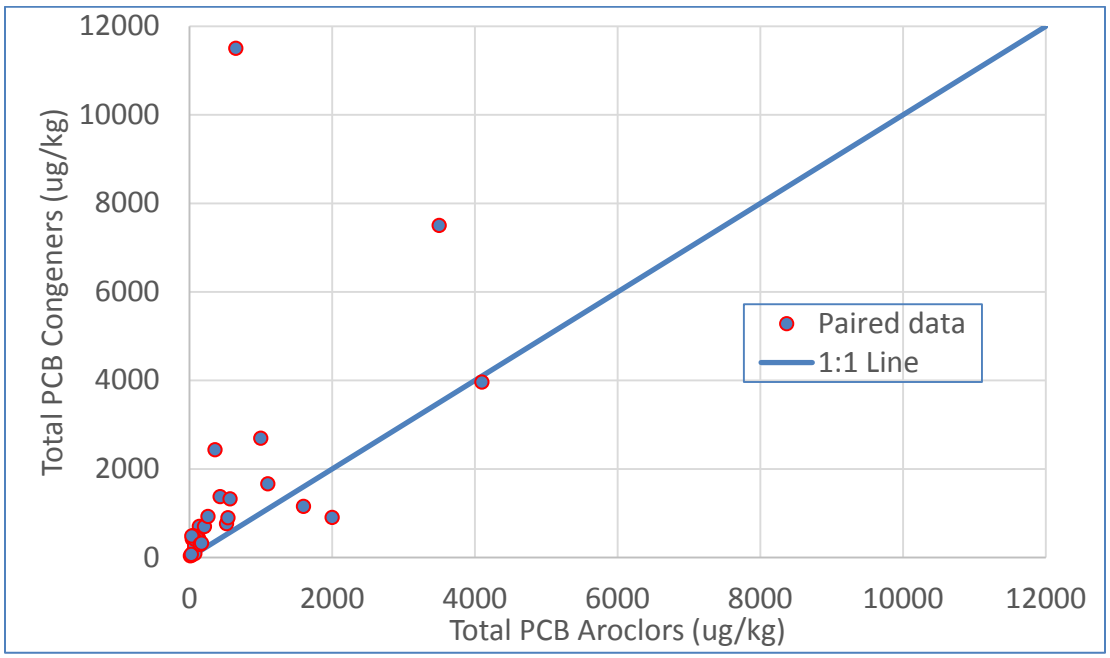
Figure 7-7. Total PCBs in East Waterway Clam and Crab Samples – Comparison of Sums of Congeners and Sums of Aroclors



Source: Windward and Anchor QEA 2014.

During the 2015 NPDES Inspection Sampling Support project (Leidos 2015b), Ecology collected 30 solids samples from different industrial stormwater drainage systems. The samples were analyzed for PCB Aroclors (Method 8082) and 209 PCB congeners by high-resolution gas chromatography (HRGC)/mass spectrometry (MS) (Method 1668C). The sums of Aroclors ranged from 6 to 222 percent of the sums of congeners from the same samples with an average of 49 percent (Figure 7-8). The linear regression between total PCB Aroclors and total PCB congeners showed a poor relationship ($R^2 = 0.269$), and the Pearson correlation coefficient was 0.518 (p-value = 0.003). The results showed that total PCBs measured in these samples by the Aroclor method were biased low when compared to total PCBs by the congener method (Alam 2015).

Figure 7-8. Total PCBs in LDW Storm Drain Samples – Comparison of Sums of Congeners and Sums of Aroclors



Source: Alam 2015.

When PCB Aroclors have been in the environment for a long time, weathering can cause underestimates due to degraded patterns. Overestimates can also occur when Aroclor analysis is used for low concentrations of PCBs or when a mixture of Aroclors containing the same congeners is present because these may be double counted. Low-resolution homolog analysis by MS methods is determined by resolution of congeners, but has higher (less-sensitive) detection limits as compared to HRGC/HRMS methods. Thus, LRMS methods may also be suitable for do rough fingerprinting if critical fingerprinting congener concentrations are above detection limits for the samples (Ecology 2014b).

EPA Region 4 (2013) identified the following considerations for selecting analytical methods:

- When the data user desires to determine which congeners are present and which congeners have been lost due to weathering, GC/LRMS may not be sensitive enough to quantitate congeners PCB-77, PCB-81, PCB-126, and PCB-169, the most toxic of the WHO high-risk congeners, due to their very low concentrations in manufactured Aroclors.

- GC/HRMS is used to determine the concentrations for the 12 WHO dioxin-like congeners.
- Congener analysis is used over homolog analysis because this is a published EPA Method and it is more sensitive, more selective, and more suited for risk assessment purposes. GC-ECD is acceptable to analyze the NOAA 18 congeners; however, it cannot be used to determine all 209 congeners.

As noted above, early analyses of environmental samples for PCBs were based on identifying Aroclor mixtures. Generally, only more recent data identify specific congeners in environmental samples. Given these different data types, the following question arises: is it possible to translate between older Aroclor data and newer congener data? Dr. Lisa Rodenburg of Rutgers University notes that, for environmental samples, generally Aroclor data cannot be translated to congeners (Rodenburg 2015a). She noted that some have multiplied Aroclor sums by 2 to get a rough estimation of a total sum of PCBs. This has been used to check if a model (such as water quality or bioaccumulation models) was "in the ballpark," but it is considered a crude method that likely is not relevant today.

Regarding translating congener data backward to Aroclors, Dr. Richard Grace of Axys Analytical noted that it is possible to go from PCB congeners to Aroclors, but only to a single Aroclor (Grace 2015b). This must be done by looking at the chromatograph patterns. According to Dr. Grace, this can build a bridge between older GC data and congener data. However, GC reported halogenated compounds (not just PCBs), and most PCB patterns do not fit neatly into Aroclors. In the 1990s, some conversion factors were developed to determine if congener analyses were accurate. Examples are provided in Figure 7-9 (Grace 2015a).

Figure 7-9. Conversion of EPA 1668C Congener Values to Aroclors

Conversion of EPA 1668C Congener Values to Aroclors (Basis for NELAP Accreditation as Domestic PE Providers Use Aroclor Standards)

- Aroclor 1016 = the sum* of PCBs 8, 18/30, 31, 28/20 concentrations multiplied by 2.7
- Aroclor 1221 = the sum of PCBs 1, 3, 8 concentrations multiplied by 1.4
- Aroclor 1232 = the sum of PCBs 1, 3, 18/30 concentrations multiplied by 3.4
- Aroclor 1242 = the sum of PCBs 8, 18/30, 31, 28/20 concentrations multiplied by 3.0
- Aroclor 1248 = the sum of PCBs 44/47/65, 49/69, 66 concentrations multiplied by 6.1
- Aroclor 1254 = the sum of PCBs 86/87/97/108/119/125, 99 concentrations multiplied by 8.0
- Aroclor 1260 = the sum of PCBs 183/185, 180/193, 170 concentrations multiplied by 5.0

The Positive Matrix Factorization method used by Dr. Rodenburg can also translate congener data into Aroclors, including more than one Aroclor. This method apportions congeners to Aroclors and shows how much is non-Aroclor residual (i.e., PCB-11 from pigments or congeners resulting from dechlorination of Aroclor-associated congeners) (Rodenburg 2015b).

Narquis et al. (2007) found that analytical data from the Hanford Superfund Site showed all of the samples from the site were identified as single Aroclors; most were Aroclor 1254, while Aroclor 1248 and Aroclor 1260 were present in isolated samples. These authors posit that in non-weathered samples (where the congener makeup of the contaminated media is relatively unchanged from the original Aroclor), it would be possible to estimate specific congener concentrations based on the research of others where the congener composition of Aroclors was determined. Narquis et al. then note that such congener concentrations could be used to calculate dioxin toxic equivalency quotients (TEQs) of the samples. The authors state that for this to work, the source of the PCB contamination would have to be well characterized, must not be a mixture of Aroclors, and the samples cannot have significant matrix interferences that complicate analysis. These are key limitations for data conversion.

Butcher et al. (1997) reported a technique to translate reported Aroclor concentrations. The authors addressed translations between historical packed-column GC quantitations of PCBs relative to Aroclor standards obtained using a variety of methods and observed concentrations of PCB congeners obtained using capillary column GC with ECD. The results were applied to an extensive database of PCB burden in fish from the upper Hudson River. The Aroclor data covered fish sampled from 1977 through 1992. Congeners were analyzed in fish samples collected in 1993. Their work demonstrated that certain apparent changes in the historical record of PCB Aroclor concentrations were partly attributable to changes in quantitation methods. The congener peaks used to calculate the Aroclors of interest (Aroclors 1016 and 1254) changed in 1979 and 1983. Analysis of the historical data showed that the method used in 1977 consistently overestimated wet-weight total PCBs, while the methods used in 1979 and 1983 consistently underestimated total PCBs. The 1977 method used PCB-52 as part of the quantitation peaks for both Aroclors 1016 and 1254, resulting in double-counting. The change in analysis method was shown to affect quantitation of Aroclor 1016 more than quantitation of Aroclor 1254 in the historical data sets.

In summary, there are several key limitations to converting data types. Converting older data from Aroclors to congeners would be impractical if the samples contained weathered Aroclors, mixtures of Aroclors, or matrix interferences. Changes in analytical methods can affect historical data. Also, conversion of congener data sets to Aroclors may be possible only to single Aroclors, rather than the mixtures that are often present at contaminated sites.

7.3 Laboratory and Reporting Issues

The following sections describe some of the potential issues associated with laboratory analyses and data reporting.

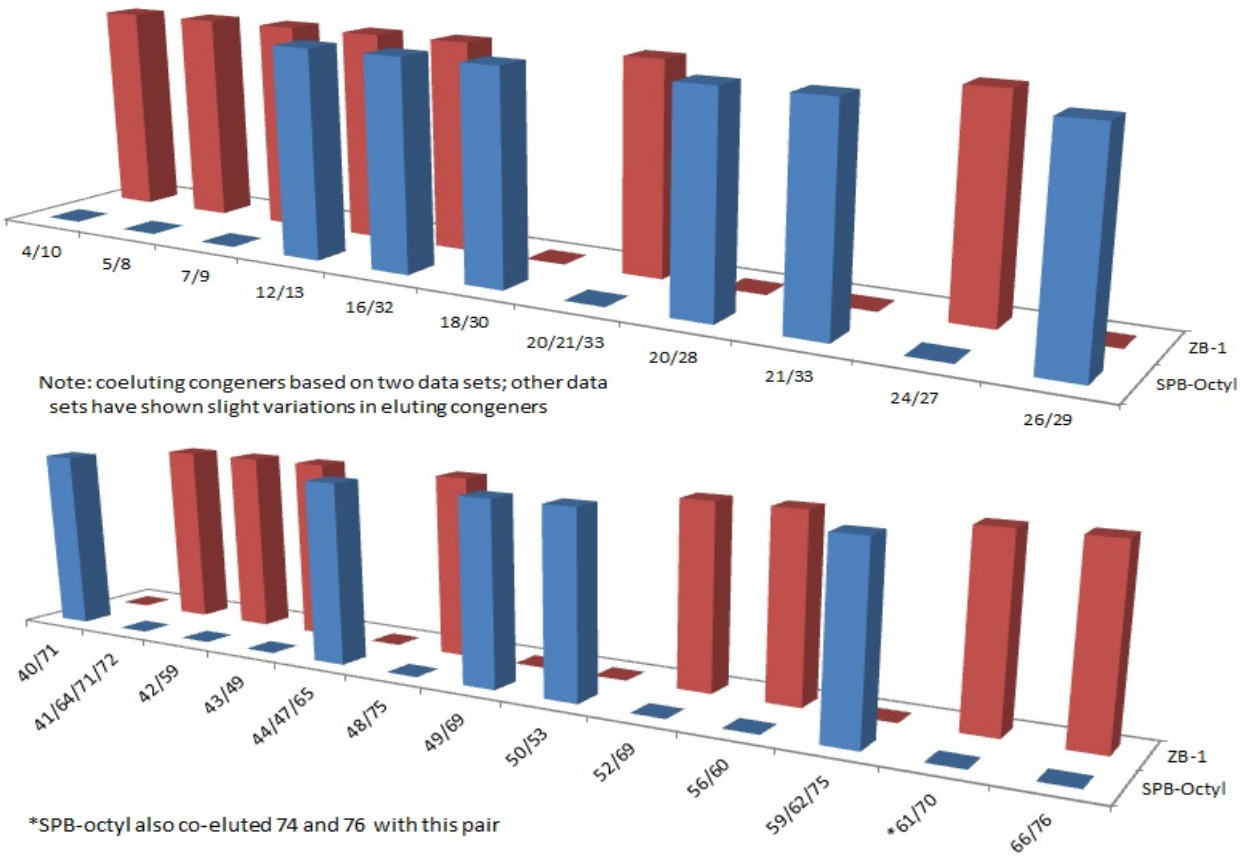
7.3.1 Laboratory Issues

Interlaboratory Variability: Columns

One of the issues related to PCB analysis by different laboratories is the use of different column types. Laboratories also vary in their ability to detect very low PCB concentrations. While EPA Method 1668 suggests the use of an SPB-octyl column, the method does not require it. The method specifies "any GC column or column system (2 or more columns) that provides unique resolution and identification of the Toxics for determination of a [PCB TEQ] using TEFs. Isomers may be unresolved so long as they have the same TEF and response factor and so long as these unresolved isomers are uniquely resolved from all other congeners."

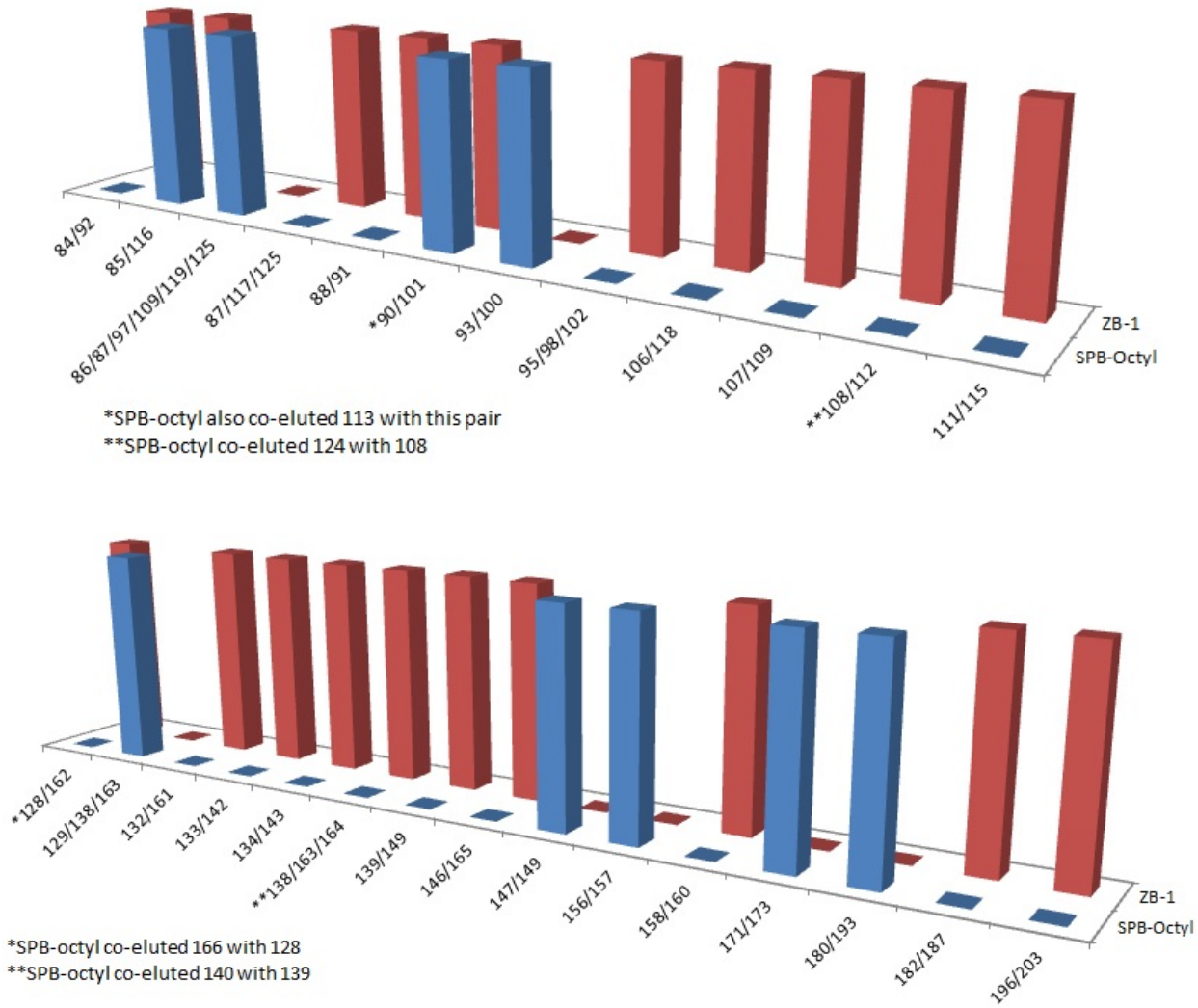
Use of different column types results in different co-eluting congener groups. Figure 7-10 shows the co-eluting congeners for two of the data sets included in this report (Leidos 2015a, 2015b). Samples collected during 2013 for the NPDES Inspection Sampling Support project were analyzed on an SPB-octyl column (blue bars). The samples collected for the project in 2014 and 2015 were analyzed on a ZB-1 column[24] (red bars). The bars in the graphs denote the presence or absence of a particular congener grouping for the respective column.

Figure 7-10. Co-Eluting Congeners by Analytical Column



[24] ZB-1 and DB-1 columns are considered comparable; ZB-1 is manufactured by Zebron/Phenomenex, while DB-1 is manufactured by Agilent Technologies. Both are non-polar 100 percent dimethylpolysiloxane columns.

Figure 7-10. Co-Eluting Congeners by Analytical Column



A full list of co-eluting congeners for studies reporting full-suite analyses is provided in Appendix B. A list of laboratories accredited in Washington State to perform PCB congener analysis by EPA Method 1668C, and the column they use during analysis, is provided in Table 7-2.

Table 7-2. Laboratories Accredited by Washington State to Perform EPA Method 1668C

Laboratory	Column Used by Laboratory		
	DB-1 / ZB-1	SPB-Octyl	SGE HE8
ALS Environmental – Burlington/Houston		●	
Axys Analytical		●	
Cape Fear Analytical[1]			
Eurofins Lancaster Laboratories Environmental		●	
Frontier Analytical Laboratory	●		

Table 7-2. Laboratories Accredited by Washington State to Perform EPA Method 1668C

Laboratory	Column Used by Laboratory		
	DB-1 / ZB-1	SPB-Octyl	SGE HE8
Maxxam Analytics International		●	
Pacific Rim Laboratories			●
SGS North America		●	
TestAmerica – Knoxville/Sacramento		●	
Vista Analytical Laboratory[2]	●	●	

[1] Multiple messages left with Cape Fear Analytical were not returned.
[2] Vista Analytical Laboratory indicated that they can use both DB-1 and SPB-Octyl columns but prefer DB-1 for congener separation. Although Vista Analytical Laboratory stated that they use DB-1 and SPB-Octyl columns for Method 1668C analysis when they were contacted in July 2015 regarding column used, the National Pollutant Discharge Elimination System Inspection Sampling Support samples collected during 2014/2015 were analyzed by this laboratory on a ZB-1 column.
EPA = U.S. Environmental Protection Agency.

Equipment Contamination

Because the latest PCB analysis techniques allow the detection of PCBs at very low levels (in the low picograms per liter [pg./L] range for water and picograms per gram range for soils, sediments, biosolids, tissue, and serum by HRMS[25]), and PCBs are ubiquitous, these compounds can be detected "in even the most pristine laboratory environments" (City of Spokane 2015).

A method blank is an analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank should be carried through the complete sample preparation and analytical procedure. The method blank is used to document contamination resulting from the analytical process. Per EPA Method 1668c, "All samples must be associated with an uncontaminated Method blank before the results for those samples may be reported or used for permitting or regulatory compliance purposes."

According to EPA Method 1668C, "Solvents, reagents, glassware, and other sample processing hardware may yield artifacts, elevated baselines, and/or lock-mass suppression causing misinterpretation of chromatograms… Environmentally abundant PCBs, as well as toxic congeners 105, 114, 118, 123, 156, 157, and 167 have been shown to be very difficult to completely eliminate from the laboratory at levels lower than the EMDLs in this Method, and baking of glassware in a kiln or furnace at 450 - 500°C may be necessary to remove these and other contaminants."

During the EPA interlaboratory study of Method 1668, congener detection rates in blank samples ranged from 8 to 33 percent, with most of the detected congeners being reported at very low concentrations relative to the concentrations reported in samples. According to EPA, "the relatively low frequency of detection of congeners in blanks by all laboratories is thought to be attributable to the failure by some laboratories to concentrate extracts to 20 µL [microlLiters] and to lesser PCB backgrounds in some laboratories."

[25] Advertised detection levels, Axys Analytical Services; available online at: <http://www.axysanalytical.com/services/organochlorine_legacy_compounds/pcb_polychlorinated_biphenyls/>.

The Spokane River source tracing study (Ecology 2012) had continual difficulty with clean laboratory blanks (i.e., method blanks) and field blanks. They were typically impacted with lower-chlorinated congeners, including PCB-11. However, this was not considered to be a concern for sediment samples because concentrations in sediment are usually much higher than the concentrations reported for the blanks. Note that the Ecology study of PCBs in general consumer products (Ecology 2014c) found PCB-11 in all 10 method blanks prepared during the study. PCB-208 was found in one method blank, PCB-209 in three method blanks, and PCB-206 in none of them.[26]

During the King County LDW Bulk Atmospheric Deposition Study (King County 2013b), the potential contribution of equipment contamination to detected samples ranged from 0.18 to 116 percent, with a median of 3.5 percent. The authors note that, because few equipment blanks were collected, the variability between equipment blanks is not well known. They also recommended that further collection and analysis of equipment blanks be performed to characterize this variability.

Dave Hope (Pacific Rim Laboratories), recommends the following procedures to reduce blank contamination levels in the laboratory (Hope 2015):

- Use carbon filtered water.
- Use disposable glassware (this is not always possible).
- Keep food samples away.

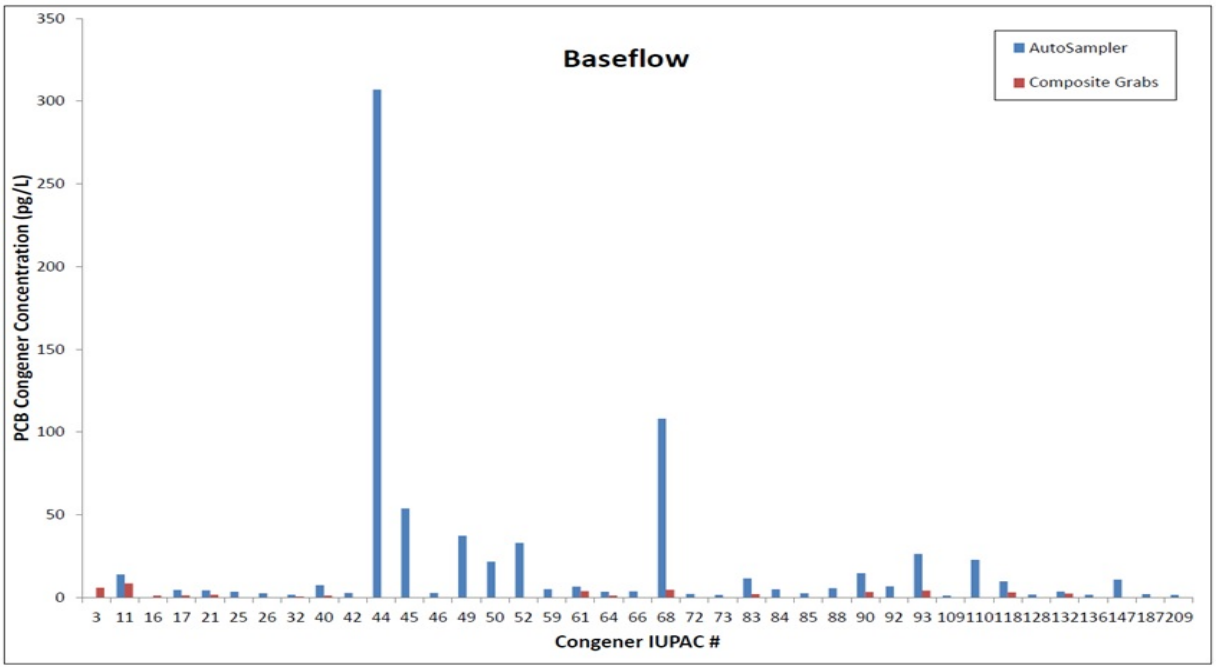
As discussed in Section 5.2, silicone tubing is a known source of PCB contamination in environmental samples. Pacific Rim Laboratories observed that PCB-68 was being detected on a regular basis in the samples they analyzed. Unpublished data developed in their laboratories indicate that this PCB congener is present in a variety of plastics, including polyethylene, polypropylene, and silicone. Others detected by Pacific Rim Laboratories during internal testing include PCB-7, PCB-28, PCB-31, PCB-47, PCB-51, and PCB-69 (Hope 2015). This laboratory has found that PCB-47, PCB-51, and PCB-68 presenting together is indicative of silicone tubing being used during sample collection (usually in conjunction with a peristaltic pump) (Hope 2012).

Similarly, an equipment blank contamination evaluation conducted by the King County Department of Natural Resources and Parks for the Upper and Middle Green River surface water study (King County 2015b) found “the environmentally-relevant total PCB concentrations and consistent PCB congener pattern in equipment blanks suggests PCB results in samples collected with autosamplers are biased high for congeners 44c[27], 45c[28] and 68. However, there is uncertainty around the degree of bias.” The King County researchers recommended that future sampling efforts include more direct comparison between composite grabs and autosampler collection methods, and that additional analysis of PCBs in the reverse-osmosis water and blanks for isolated Teflon and silicon tubing be conducted to better understand equipment blank results. Figure 7-11 shows the PCB congener profiles for method comparison base flow samples: ISCO® autosampler composite and cross-sectional composite grabs. PCB-44 and PCB-68 show up strongly in the samples taken by the autosampler. King County is currently conducting a PCB

[26] Other congeners may also have been detected, but this preliminary report focused only on these four congeners.
[27] Includes co-eluting congeners 47 and 65.
[28] Includes co-eluting congener 51.

Equipment Blank Study to further evaluate the potential bias and source of the PCB equipment contamination in the Green River samples collected by autosamplers (King County 2015c).

Figure 7-11. PCB Congener Profiles for Base Flow Method Comparison Samples



Source: King County 2015b.

Sample Preparation and Cleanup

There is a potential for loss of lower-chlorinated congeners during sample preparation. EPA Method 1668C indicates this loss is possible when extracts of tissues are concentrated by roto-evaporation.

Dr. Grace of Axys Analytical (Grace 2015b) notes that EPA Method 1668c analysis is highly dependent on the quality of cleanup steps taken before the actual sample analysis. Poor cleanup can lead to 'messy' samples that will have many interferences. Sensitivity will be decreased by sample interferences.

For tissue samples, the natural lipid content can interfere in the analysis for PCBs. The lipid contents of different species and portions of tissue can vary widely. Lipids may be present in sufficient quantity to overwhelm the column chromatographic cleanup procedures used for cleanup of sample extracts and must be removed via additional cleanup steps (EPA 2010b).

7.3.2 Data Interpretation and Reporting

This section includes a discussion of treatment of non-detected PCB congeners and Aroclors, including assumptions of 0, one-half of the reporting/detection limit, and the full reporting/detection limit. In addition, it discusses estimated maximum possible concentrations (EMPCs) reported by the laboratories, how these relate to detection limits, and issues associated with use of EMPC-flagged data.

PQLs

The practical quantitation limit (PQL) is defined in WAC 173-204-505(15) as: "The lowest concentration that can be reliably measured within specified limits of precision, accuracy, representativeness, completeness, and comparability during routine laboratory operating conditions, using department approved methods."

According to the *Sediment Cleanup User's Manual II* (Ecology 2015b), procedures for establishing PQLs must:

- Control false-positive and false-negative results.
- Consider and incorporate laboratory method blank results.
- Incorporate long-term variability.
- Include a demonstration of qualitative compound identification capability.

For Method 1668C, the detection limits and quantitation levels are usually dependent on the level of interferences and laboratory background levels rather than instrument limitations (EPA 2010b). The minimum levels of quantitation identified in the method are the levels at which PCBs can be determined with no interferences present: 20 to 100 pg/L in water, 2 to 10 nanograms per kilogram in solids.

As discussed in Section 7.1, PQLs for Aroclor analytical methods (e.g., EPA Method 8082) are generally in the ppb to ppm range. Regulatory criteria for soil and sediment are within this range; however, WQC are far lower (Table 4-1). To compare with the WQC, a more sensitive analytical method (e.g., EPA Method 1668C), which supports reporting of total PCBs in the ppq to ppt range, is required.

Treatment of Non-Detected PCB Congeners

Ecology's Technical Memorandum *Dioxins, Furans, and Dioxin-Like PCB Congeners: Addressing Non-Detects and Establishing PQLs for Ecological Risk Assessments in Upland Soil* notes that "when a laboratory reports a target analyte as ND or <, a numeric reporting limit is also provided. This means that the analyte is not present at or above the numeric reporting limit. However, the analyte could be present at a lower concentration. In fact, the laboratory may even have positively identified it - although not reported it - at that lower concentration (Scholz and Flory 1999). Many of the terms used by laboratories are interchangeable and several refer to the same thing, but some have significantly different meanings. It is helpful to understand these terms to make informed decisions based on laboratory results, when a compound is reported as ND. It is also important to use a consistent approach to describe: a) when reported laboratory data should be considered NDs, and b) how to interpret ND data" (Ecology 2015c).

Data sets that contain analyte values below the detection limit (BDL) are known as censored data sets. Censored data sets present difficulties for many standard estimation procedures and statistical tests. For example, the mean cannot be estimated unless numerical values are assigned to the BDL data. The values assigned to BDL data can, therefore, significantly impact the calculated mean of the data set (Ecology 1992).

Different methods have been used in the reporting of total PCB concentrations when some congeners are BDL. Such methods include an assumption that the actual value is zero (the

congener truly is not present in the sample), assumption that the congener is present at one-half of the reporting or detection limit, or assumption that the congener is present at the full reporting or detection limit. Researchers have noted that the calculation of statistics (e.g., upper confidence limits) using substitution methods (i.e., the replacement of non-detects by one-half or the full detection limits) do not perform well, even when the percentage of non-detected observations is low, such as less than 5 percent to 10 percent (reviewed in Singh and Singh 2013). According to a King County reviewer of an earlier version of this report, the use of one-half or full reporting or detection limits for non-detected data has typically only been done for TEQ calculation to evaluate the potential bias of how non-detects are treated in the TEQ calculation.

One technique used to handle data sets with non-detects is the Kaplan-Meier technique. The Kaplan-Meier technique may be used to adjust a mixture of detected and BDL data for the unknown concentrations of non-detect values. The technique leads to adjusted estimates for the mean and standard deviation of the underlying population (EPA 2009). This method can be used if sample size is at least 8 to 10, with up to 50 percent non-detects. Kaplan-Meier offers a way to adjust for significant fractions of non-detects without having to know the actual non-detect concentration values. Estimation of the Kaplan-Meier mean and standard deviation will tend to be slightly biased, typically with the mean on the high side and the standard deviation on the low side. This occurs because the Kaplan-Meier cumulative distribution function levels corresponding to distinct reporting limits are treated as if they were known measurements rather than the upper bounds on possible values. As long as the total proportion of censored measurements is not too high, the degree of bias will tend to be small. Larger biases are more likely whenever the detection rate is less than 50 percent.

The Kaplan-Meier estimation method has an added advantage over other methods as it can be used on data sets with multiple detection limits (Singh et al. 2006). EPA's ProUCL statistical software can employ this method for statistical calculations (Singh et al. 2013).

EMPCs

If an analyte is detected, and all of the identification criteria are met except for the mass ion abundance ratio (m/z ratio), this is considered an EMPC value. An EMPC is a worst-case estimate of the concentration. The congener-specific detection limit in a sample is at the estimated detection limit (EDL), which is expressed as 2.5 times the signal-to-noise ratio. Detecting a concentration above the EDL is not the only criterion needed to positively identify an analyte. The criteria needed to unambiguously identify a GC peak are as follows (Ecology 2015d, citing an EPA document: EPA-540-R-11-016):

- Retention times and relative retention times.
- Peak identification.
- Signal-to-noise ratio.
- Ion abundance ratios.
- Polychlorinated diphenyl ether interferences.

EMPC-flagged data indicate that there is uncertainty as to the true identity of an EMPC-qualified congener. This uncertainty could potentially lead to false positives. During data validation, EMPC-flagged results are typically qualified as not detected at the reported EMPC value.

Method Blank Contamination

EPA Method 1668C requires that "If any CB is found in the blank at greater than two times the minimum level (Table 2 [in the Method]) or one-third the regulatory compliance limit, whichever is greater; or if any potentially interfering compound is found in the blank at the minimum level for each CB given in Table 2 (assuming a response factor of 1 relative to the quantitation reference in Table 2 at that level of chlorination for a potentially interfering compound; i.e., a compound not listed in this Method), analysis of samples must be halted until the sample batch is re-extracted and the extracts re-analyzed, and the blank associated with the sample batch shows no evidence of contamination at these levels. All samples must be associated with an uncontaminated Method blank before the results for those samples may be reported or used for permitting or regulatory compliance purposes." Note, however, that it is not possible for all method blanks to be completely free of contamination, and achieving less than 20 pg/L per congener in laboratory blanks is considered excellent (Hope 2011).

Laboratories maintain control charts for each congener that track the mean and standard deviations of the concentrations of that congener in the blanks. This information is used to assess the confidence level of the data and to identify if there is significant contamination in the laboratory environment so that action may be taken to address it.

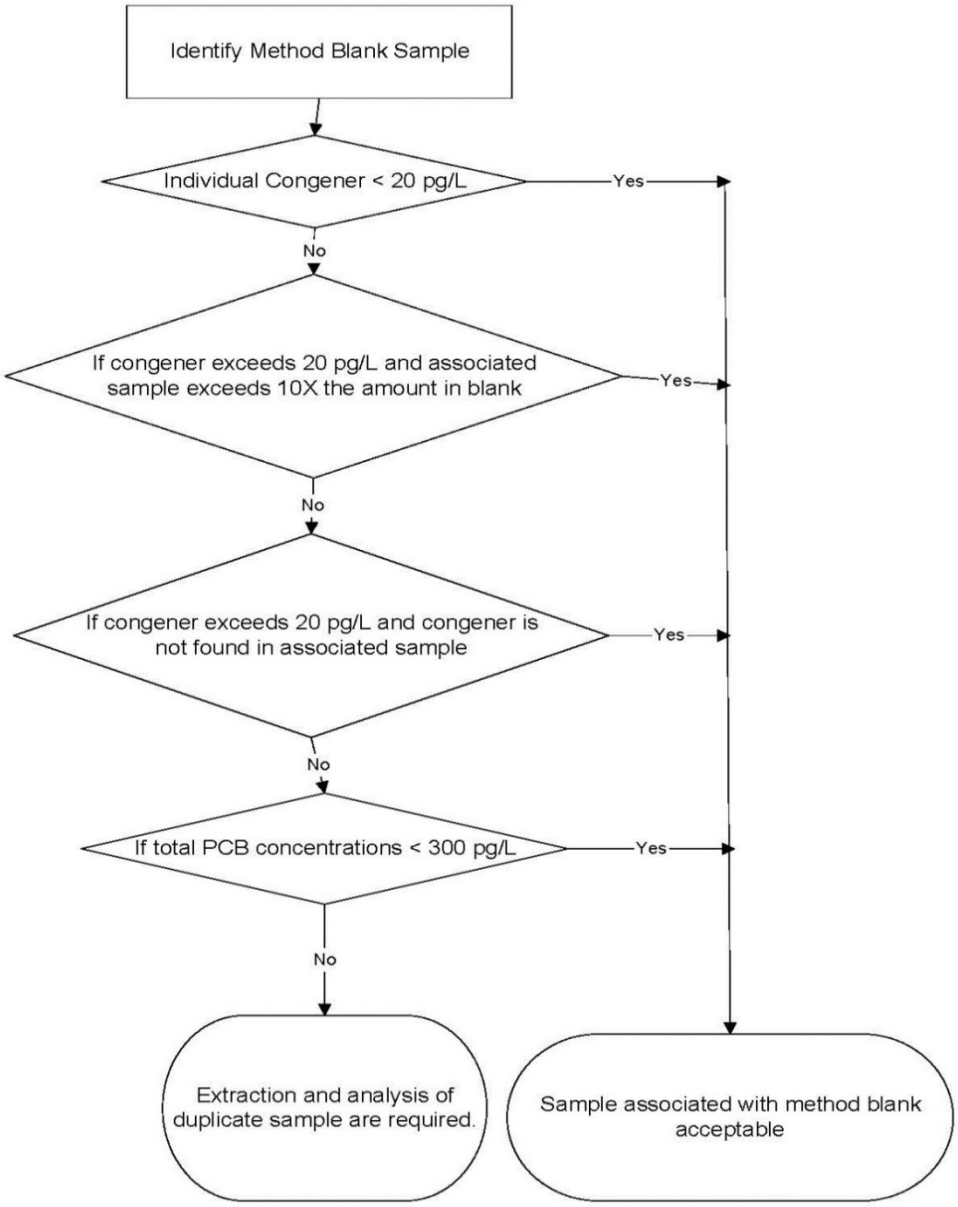
The Delaware River Basin Commission established a protocol for handling water sample data when there is demonstrated method blank contamination. A set of decision rules (Figure 7-12) is applied that results in either the sample associated with the method blank being considered acceptable or in the requirement for the laboratory to extract and analyze a duplicate of the sample.

The Spokane River Regional Toxics Task Force has also developed draft decision rules for the treatment of samples with corresponding method blank contamination (Figure 7-13).

Ecology's *Sediment Cleanup User's Manual* (Ecology 2015b) states that 1 method blank should be run per sample batch or every 20 samples, whichever is more frequent, or when there is a change in reagents. If the analyte concentration is less than or equal to the PQL, then the laboratory is required to eliminate or greatly reduce laboratory contamination due to glassware, reagents, or analytical system, and to redigest and reanalyze affected samples (Table 5-3). Samples that contain less than five times the amount found in the blank are flagged as non-detect.

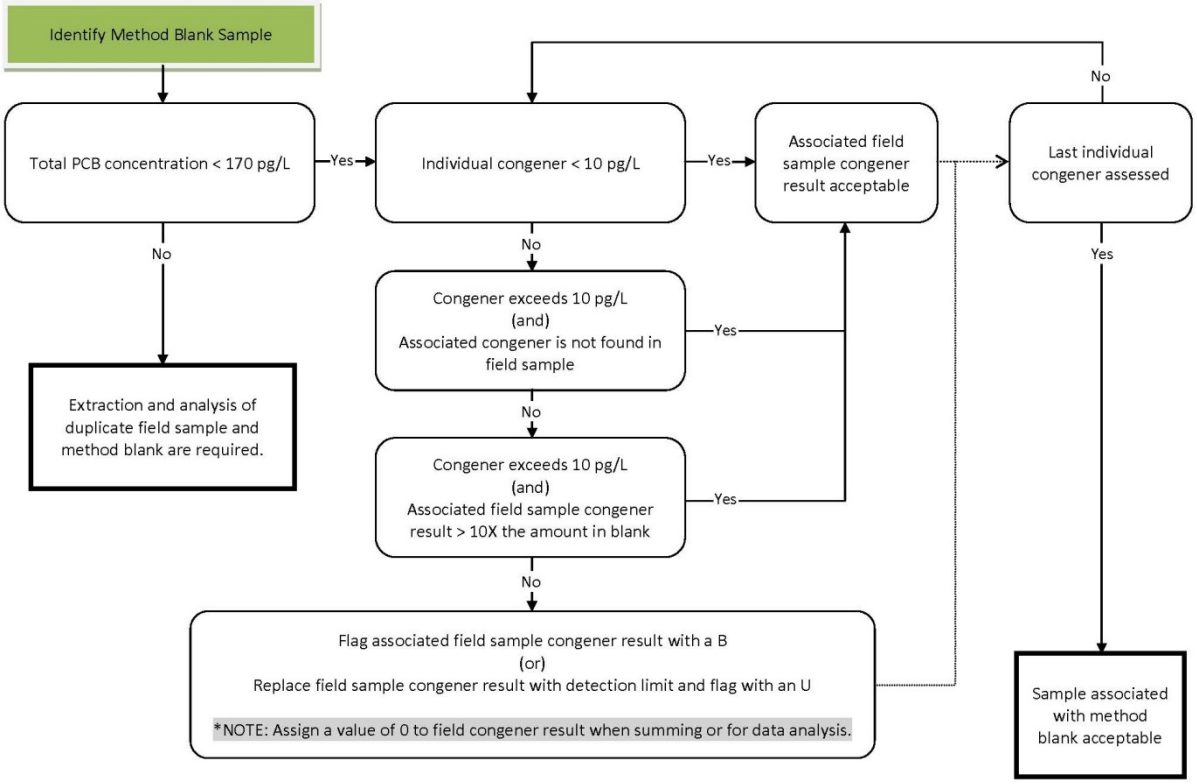
The King County LDW Bulk Atmospheric Deposition Study experienced method blank contamination in all batches analyzed for PCBs (King County 2015a). As many as 28 congeners were detected in method blanks, and 1 or more mono- or dichlorinated congeners were detected in most method blanks. For this study, environmental sample detections were qualified as non-detect by the contract validator whenever sample concentrations were within five times the method blank concentration. Using the "5X" rule reduces the potential for false positives but raises the opportunities for false negatives. This potentially resulted in some low bias for congeners detected above the method blank concentration but within five times the method blank.

Figure 7-12. Delaware River Basin Commission Method Blank Decision Rules



Source: Delaware River Basin Commission website. Available online at: <http://www.state.nj.us/drbc/library/documents/PCB-MethodBlankRules.pdf>.

Figure 7-13. Spokane River Method Blank Contamination Decision Rules



Source: Available online at: <http://srrttf.org/wp-content/uploads/2013/10/Method-Blank-Contamination-Decision-Rules_Draft_10-16-2013.pdf>.

Chromatograph Interpretation

Hughes et al. (2015) raise concerns regarding interpretation of congener peaks and errors that can arise in the assignment of concentrations to co-eluting congeners during post-processing calculation of congener concentrations. They note that some researchers assume co-eluting congeners occur in equal proportions. Others split co-eluting peaks based on the average distribution of congeners in Aroclor mixtures. Both methods are problematic.

Splitting congener concentrations in equal portions becomes an issue because not all co-eluting congeners occur in the same concentrations. This method automatically under-represents the more highly concentrated congener while simultaneously over-representing the less highly concentrated congener.

With regard to splitting congener concentrations by Aroclor ratios, Hughes et al. (2015) note: "Peak concentration splits according to Aroclor ratios presuppose that congeners absent from the commercial Aroclor mixtures are also absent from the sample to be analyzed. Thus, if transformation of a PCB molecule produces a congener that is not found in the commercial Aroclors, and if this congener shares a peak with a congener that is present in the commercial Aroclors, the former congener will not be reported. This is because when a congener that is not present in Aroclors is detected by the GC-ECD method, its mass within the co-eluting peak will

be assigned to the congener that is present in Aroclor(s). Thus, certain congeners, here termed ghost congeners, that are not present in Aroclors, cannot be reported in field samples analyzed by this method." Using field samples from a contaminated sediment site, these researchers found "significant underestimation of total PCB concentration in sediment samples can occur if Aroclor-based splits are used for weathered samples."

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8.0 Summary of Available Data

8.1 Data Acquisition

PCB data were queried from the Ecology EIM database on March 26, April 23, and June 16, 2015. The query search parameters were WRIA = 09-Duwamish Green, County = King, and Result Parameter Group = Polychlorinated biphenyls. These initial searches resulted in a total of 266 studies. Many of these studies did not contain any congener data and were eliminated. The remaining studies were checked against their respective EIM location maps. A total of 34 studies contained at least some PCB congener data for samples that were located within the study area. Of these 34 studies, 3 were excluded, as described in Section 8.3.

The following five data sets were not found in EIM but were added to the database separately in response to reviewer comments on the draft version of this PCB Congener Report:

- East Waterway Supplemental RI/FS database, dated June 5, 2013.
- Data for surface water, sediment, and suspended solids samples collected by the U.S. Geological Survey (USGS) during 2014/2015 in support of the Green River Loading Study.
- King County's Bulk Atmospheric Deposition Study (2011/2012) and Supplemental Bulk Atmospheric Deposition Study (2013).
- King County CSO sampling conducted between 2007 and 2010.

Additional PCB congener data for the study area may be present; for example, one reviewer of an earlier draft of this report noted that additional congener data may be available in the LDW RI/FS database. A review of the LDW RI/FS database for PCB congener sampling results that have not been uploaded to EIM was beyond the scope of the current project phase.

8.2 Data Sets Included in Polychlorinated Biphenyl Congener Database

The studies described below reported chemical analysis data for PCB congeners. PCB Aroclor data collected during these studies were also included in the PCB Congener Database. A total of 39 data sets have been identified to date, of which 34 were obtained from the EIM database. Three of these data sets were considered unusable, as described in Section 8.3.

The 36 remaining data sets were grouped into 8 categories:

- Data sets in support of the LDW RI/FS;
- Data sets in support of the East Waterway RI/FS;
- Data sets in support of Green-Duwamish River loading studies;
- Surface water, atmospheric deposition, and CSO data collected by King County in the Green-Duwamish watershed;
- Storm drain system sampling data;

- Other fish/shellfish sampling studies;
- Other sediment sampling studies;
- Soil/solids sampling data.

In the summary descriptions below, the name of the study is followed by the sampling year(s) in parentheses. Studies are presented in chronological order from oldest to the most recent. Some studies described below had geographic boundaries that extended beyond the LDW/Green River study area. The numbers of locations sampled listed in the summary tables below are for samples taken in the study area only.

With the exception of three studies identified in Section 8.3, which appeared to list PCB congeners in error, no attempt was made to exclude data sets from the PCB Congener Database during this phase of the study. Little information was available in EIM to assess the quality of the data associated with these studies; additional work is needed to review original source documents. Phase 2 of this project should include development of criteria for inclusion/exclusion of data prior to use of this data set for future data evaluations and modeling. Criteria to be considered may include age of the data, key congeners, appropriate detection limits, and data validation level.

A summary of sample totals by environmental medium is presented in Section 8.4.

8.2.1 Lower Duwamish Waterway Remedial Investigation/Feasibility Study

Five studies were identified in EIM that included PCB congener data collected specifically to support the RI/FS for the LDW Superfund Site. Samples collected include tissue (fish, crab, clam, and benthic invertebrates) and surface and subsurface sediment.

A detailed comparison of data with the LDW RI/FS database was not performed. Additional PCB congener data that have not been uploaded to EIM may have been collected in support of the LDW RI/FS.

LDW RI – Fish and Crab (2004)

Study Code / EIM Study Identifier	9 / Fishtion
Sample Matrix	Tissue – fish and crab
Number of Locations Sampled	116 locations (all LDW)
Number of Samples	139 samples: 47 – full suite of congeners, 139 – Aroclors
Number of Congener Locations/Samples	47 locations / 47 samples
PCB Analysis Method	Congeners: EPA 1668A; Aroclors: EPA 8082A
Laboratory that Conducted Analyses	Congeners: Axys Analytical Services, Sidney, British Columbia; Aroclors: Analytical Resources, Inc., Seattle, Washington
Submitting Organization	Windward Environmental for LDWG

The purpose of this study was to investigate the nature and extent of contamination to support ecological and human health risk assessment. Six species of fish (English sole, starry flounder, Pacific staghorn sculpin, pile perch, shiner perch, and striped seaperch) and two species of crab (Dungeness and slender crab) were collected in August and September 2004. A total of 139

samples were analyzed for PCBs as Aroclors. A subset of 47 samples, including at least 1 from each species, was also analyzed for the full suite of PCB congeners (Windward 2005a, 2005c).

LDW RI – Sediment, Clams, and Benthic Invertebrates (2004)

Study Code / EIM Study Identifier	26 / LDWRITHIC
Sample Matrix	Sediment, Tissue – clam and benthic invertebrates
Number of Locations Sampled	35 locations (all LDW)
Number of Samples	68 samples: 35 sediment samples (16 – full suite of congeners, 35 – Aroclors), 33 tissue samples (16 – full suite of congeners, 33 – Aroclors)
Number of Congener Locations/Samples	17 locations / 32 samples
PCB Analysis Method	Congeners: EPA 1668; Aroclors: EPA 8082A
Laboratory that Conducted Analyses	Congeners: Axys Analytical Services, Sidney, British Columbia; Aroclors: Columbia Analytical Services, Kelso, Washington
Submitting Organization	Windward Environmental for LDWG

The purpose of this study was to investigate the nature and extent of contamination to support ecological and human health risk assessment. A total of 14 clam samples (soft-shell clam) were analyzed for Aroclors, and 8 of these samples were also analyzed for the full suite of PCB congeners. A total of 19 benthic invertebrate (amphipod) samples were analyzed for Aroclors, and 8 of these were also analyzed for the full suite of PCB congeners. Surface sediment sample data are also included in this data set: 35 surface sediment samples were analyzed for Aroclors, and 16 of these were also analyzed for the full suite of PCB congeners. Samples were collected in August and September of 2004 (Windward 2005e).

LDW RI – Fish Collection (2005)

Study Code / EIM Study Identifier	18 / LDWFISH5
Sample Matrix	Tissue – Fish
Number of Locations Sampled	65 locations (all LDW)
Number of Samples	65 samples: 6 – full suite of congeners, 65 – Aroclors
Number of Congener Locations/Samples	6 locations / 6 samples
PCB Analysis Method	Congeners: EPA 1668A; Aroclors: EPA 8082, Revision 0
Laboratory that Conducted Analyses	Congeners: Axys Analytical Services, Sidney, British Columbia; Aroclors: Analytical Resources, Inc., Seattle, Washington
Submitting Organization	Windward Environmental for LDWG

The purpose of this study was to investigate PCB concentrations in fish in the Lower Duwamish River. According to the data entered into EIM, a total of 65 locations were sampled during August and September 2005, with 1 sample collected at each location. All samples were analyzed for PCBs as Aroclors, and a subset of six samples (English sole and shiner perch) was also analyzed for the full suite of congeners. EIM incorrectly identifies the sample matrix for these samples as “sediment.”

LDW RI – Surface Sediment Rounds 1 and 2 (2005)

Study Code / EIM Study Identifier	24 and 25 / LDWRRUN1 and LDWRRUN2
Sample Matrix	Sediment
Number of Locations Sampled	163 locations (157 in LDW, 6 Green-Duwamish River)
Number of Samples	171 samples: 33 – subset of congeners, 171 – Aroclors
Number of Congener Locations/Samples	33 locations (LDW) / 33 samples
PCB Analysis Method	Congeners: EPA 1668A; Aroclors: EPA 8082, Revision 0
Laboratory that Conducted Analyses	Congeners: Axys Analytical Services, Sidney, British Columbia; Aroclors: Analytical Resources, Inc., Seattle, Washington
Submitting Organization	Windward Environmental for LDWG

The purpose of this study was site monitoring for a contaminated site investigation. Sampling was conducted between January and March 2005. A total of 163 locations were sampled over the course of the 2 sampling phases. A total of 171 samples were analyzed for PCBs as Aroclors. A total of 33 of these samples were also analyzed for a subset of 17 PCB congeners (listed below). This congener set includes the WHO 12 most toxic dioxin-like congeners plus 5 additional congeners (Windward 2005b, 2005d).

PCB-66	PCB-110	PCB-129	PCB-180
PCB-77	PCB-114	PCB-153	PCB-189
PCB-81	PCB-118	PCB-156	
PCB-90	PCB-123	PCB-167	
PCB-105	PCB-126	PCB-169	

8.2.2 East Waterway Supplemental Remedial Investigation/Feasibility Study

Study Code/EIM Study Identifier	6 / not in EIM
Sample Matrix	Sediment
Number of Locations Sampled	311 locations (all East/West Waterway)
Number of Samples	482 samples: 257 sediment samples (17 – full suite of congeners, 244 – Aroclors); 57 surface water samples (57 – full suite of congeners), and 168 fish/shellfish tissue samples (29 – full suite of congeners, 3 – subset of congeners, 168 – Aroclors)
Number of Congener Locations/Samples	22 locations (East Waterway) / 36 samples
PCB Analysis Method	Congeners: EPA 1668A; Aroclors: EPA 8082, Revision 0
Laboratory that Conducted Analyses	Congeners: Analytical Perspectives, Wilmington, North Carolina; Aroclors: Columbia Analytical Services, Kelso, Washington
Submitting Organization	Windward Environmental for the East Waterway Group

Data were obtained from the East Waterway Operable Unit Supplemental RI/FS database, provided by Windward Environmental (McGroddy 2015). The database includes surface water,

sediment, and fish/shellfish tissue samples collected from a total of 311 locations between March 1995 and August 2009.

A total of 257 surface/subsurface sediment samples were collected, and 17 samples were analyzed for the full suite of 209 congeners, while a total of 244 samples were analyzed for Aroclors. All 57 surface water samples that have been collected were analyzed for the full suite of PCB congeners. Tissue samples analyzed for PCB congeners include fish (brown rockfish, English sole, and shiner surfperch), crab (red rock crab and Dungeness crab), and clams (butter clam, cockle, and geoduck). Twenty-nine tissues samples were analyzed for the full suite of PCB congeners, 3 were analyzed for a subset of congeners, and all 168 tissue samples were analyzed for Aroclors (Windward 2010a).

Three English sole samples collected in December 1995 were analyzed for the following subset of 23 congeners:

PCB-52	PCB-118	PCB-151	PCB-180
PCB-75	PCB-126	PCB-153	PCB-183
PCB-77	PCB-128	PCB-169	PCB-187
PCB-81	PCB-138	PCB-170	PCB-195
PCB-101	PCB-141	PCB-174	PCB-206
PCB-105	PCB-149	PCB-177	

8.2.3 Lower Duwamish Waterway and Green River Contaminant Loading Studies

Lower Duwamish River Upstream Sediment Analysis (2008)

Study Code / EIM Study Identifier	27 / LDWUPSED2010
Sample Matrix	Sediment
Number of Locations Sampled	27 locations (1 in LDW, 26 in Green-Duwamish River)
Number of Samples	27 samples: 7 – full congener suite, 27 – Aroclors
Number of Congener Locations/Samples	7 locations (Green-Duwamish River) / 7 samples
PCB Analysis Method	Congeners: EPA 1668A; Aroclors: EPA SW8082, Revision 0
Laboratory that Conducted Analyses	Aroclors: TestAmerica, Tacoma, Washington; and Analytical Resources, Inc., Seattle, Washington; Congeners: Analytical Perspectives, Wilmington, North Carolina
Submitting Organization	Windward Environmental for EPA

This study consisted of reanalysis of a subset of samples collected by Ecology in 2008 in support of Ecology's contaminant loading study for the LDW site. The goal of the 2008 sampling effort was to better understand the distribution of chemicals in surface sediments upstream of RM 4.9. A total of 88 surface sediment samples (including 2 duplicates) were collected during the initial study in 2008; these were analyzed for Aroclors only. Samples were collected in the Duwamish River from just south of the Boeing Developmental Center to about S 124th Street. Sampling was conducted in April and May 2008. In May 2009, 27 samples were reanalyzed for

PCBs as Aroclors, and a subset of 7 samples was analyzed for the full suite of PCB congeners. These 27 samples were included in the PCB Congener Database.

Green River Loading Study – Phases 1 and 2 (2013-2015)

Study Code / EIM Study Identifier	12 and 13 / GRNRVLD13 and GRNRVLD14 plus recent data not in EIM
Sample Matrix	Sediment, surface water, and suspended solids
Number of Locations Sampled	3 locations (Green-Duwamish River)
Number of Samples	88 samples: 22 sediment samples (20 – full suite of congeners, 16 – Aroclors); 39 surface water (37 – full suite of congeners, 37 – Aroclors); 27 suspended sediment (26 – full suite of congeners, 14 – Aroclors)
Number of Congener Locations/Samples	3 locations / 84 samples
PCB Analysis Method	Congeners: EPA 1668A; Aroclors: EPA SW8082A Revision 1
Laboratory that Conducted Analyses	Congeners: Axys Analytical Services, Sidney, British Columbia, Canada; Aroclors: Analytical Resources, Inc., Seattle, Washington
Submitting Organization	USGS for Ecology

The purpose of this study was to assess sediment and chemical loading from the Green River to the LDW. During the first phase of the project, 24 samples (12 sediment, 8 surface water, and 4 suspended solids samples) were collected at and near the Foster Links Golf Course cart bridge between February and June 2013. Six water samples and 10 sediment samples collected in 2013 were analyzed for the full suite of PCB congeners.

A single water sample with PCB data was included in the GRNRVLD2014 data set. Recent (2014) data from the second phase of the study were obtained from Ecology; these data were not in EIM at the time this report was prepared. A total of 64 samples collected between January 2014 and March 2015 are included in the PCB Congener Database; these include 10 sediment samples, 31 surface water samples, and 23 suspended solids samples. Full suite congener data were available for all but one suspended solids sample from August 2014, for which only total homolog data were provided.

8.2.4 Studies Conducted by King County

King County's Department of Natural Resources and Parks has conducted several studies that included sampling for PCB congeners. These studies include surface water, CSO, and air deposition data, and were included in the PCB Congener Database as described below.

King County Water Sampling (2005-2008)

Study Code / EIM Study Identifier	23 / LDW-KC-Waters
Sample Matrix	Surface water
Number of Locations Sampled	4 locations (1 in LDW, 2 in Green River, 1 in East/West Waterway)
Number of Samples	51 samples (all full suite PCB congeners)
Number of Congener Locations/Samples	4 locations (see above) / 51 samples
PCB Analysis Method	Congeners: EPA 1668A
Laboratories that Conducted Analyses	Congeners: Axys Analytical Services, Sidney, British Columbia (2005-2008); King County Environmental Laboratory, Seattle, Washington (2008 only)
Submitting Organization	King County Department of Natural Resources and Parks and Windward Environmental (for King County)

Four locations were sampled as part of this effort: one location south of the Spokane Street Bridge to the west of the Harbor Island marina; one at the South Park Bridge at 16th Avenue S; one in the Duwamish River at the East Marginal Way S Bridge; and one in the Green River at Fort Dent Park. Samples were collected in 2005 during the months of August, September, November, and December. Multiple sampling events at each station were conducted between August 2005 and August 2008. Samples were collected at 1 meter above the river bottom and 1 meter below the water surface. All samples were analyzed for the full suite of PCB congeners.

EIM also includes study KC20LING; the data from this study were incorrectly identified as sediment and these data are also included in LDW-KC-Waters. Therefore, KC20LING was not considered further.

King County CSO Sampling (2007-2010)

Study Code / EIM Study Identifier	38 / not in EIM
Sample Matrix	Water
Number of Locations Sampled	7 locations (4 in LDW, 3 in East Waterway)
Number of Samples	51 samples (all full suite PCB congeners)
Number of Congener Locations/Samples	7 locations (see above) / 51 samples
PCB Analysis Method	Congeners: EPA 1668A
Laboratories that Conducted Analyses	Congeners: Axys Analytical Services, Sidney, British Columbia
Submitting Organization	King County Department of Natural Resources and Parks

This study included collection of samples that represent CSOs in the Duwamish River Basin between September 2007 and January 2010 (King County 2011). Samples were collected at partially to near-full conditions of the combined sewers at seven locations. Four locations are in the LDW (Michigan Street Regulator, West Michigan Regulator, Brandon St. CSO, and Duwamish Siphon Forebay) and three are in the East Waterway (Hanford #2 CSO, Lander II Regulator, and Kingdome Regulator). A total of 51 water samples were collected (including field duplicates); all were analyzed for the full suite of PCB congeners.

King County Green River Watershed Surface Water (2011-2012)

Study Code / EIM Study Identifier	14 / not in EIM
Sample Matrix	Surface water
Number of Locations Sampled	6 locations (all Green River)
Number of Samples	55 samples (55 – full suite of congeners)
Number of Congener Locations/Samples	6 locations / 55 samples
PCB Analysis Method	Congeners: EPA 1668C
Laboratory that Conducted Analyses	Congeners: Axys Analytical Services, Sidney, British Columbia
Submitting Organization	King County Department of Natural Resources and Parks

The purpose of this study was to better understand the relative contribution of COCs for the LDW from upstream areas in the Green River. It included collection and analysis of surface water samples from four major tributaries to the Green River (Newaukum, Soos, and Mill Creeks and the Black River), as well as at two locations on the main stem Green River: an upstream location at Flaming Geyser State Park (upriver of the tributary sampling sites), and a downstream location at Foster Links Golf Course (downstream of the tributaries). At each of the six locations, three composite samples were collected during the dry season to represent base flow conditions, while six composite samples were collected during storm events. All samples were analyzed for the full suite of PCB congeners (King County 2014).

King County Upper and Middle Green River Surface Water (2013-2014)

Study Code / EIM Study Identifier	15 / not in EIM
Sample Matrix	Surface water
Number of Locations Sampled	3 locations (all Green River)
Number of Samples	24 samples (24 – full suite of congeners)
Number of Congener Locations/Samples	3 locations / 24 samples
PCB Analysis Method	Congeners: EPA 1668C
Laboratory that Conducted Analyses	Congeners: Axys Analytical Services, Sidney, British Columbia
Submitting Organization	King County Department of Natural Resources and Parks

The purpose of this effort was to better understand the relative concentrations of key LDW COCs in the upper and middle reaches of the Green River, which are farther away from developed areas and contaminant sources. This study included analysis of water samples collected from three locations. Two sites were located approximately 20 miles above the Howard Hansen Dam; one on the main stem of the Green River (RM 85), and a second on a major tributary, Sunday Creek (RM 82). A third site was located below the Dam in the middle reach of the Green River at Kanaskat-Palmer State Park at RM 56. At the Kanaskat-Palmer location, three composite samples were collected during the dry season to represent base flow conditions, while five composite samples were collected during storm events. At each of the two locations upstream of the Dam, three composite base flow and three composite storm event samples were collected. Samples were analyzed for PCB congeners (King County 2015b).

King County LDW Bulk Atmospheric Deposition Study (2011-2012)

Study Code / EIM Study Identifier	16 / not in EIM
Sample Matrix/Matrices	Air deposition
Number of Locations Sampled	6 locations (3 in LDW, 3 in Green-Duwamish River)
Number of Samples	49 samples (49 – full suite of congeners)
Number of Congener Locations/Samples	6 locations (see above), 49 samples
PCB Analysis Method	Congeners: EPA 1668A (August and September 2011); EPA 1668C (October 2011 through October 2012)
Laboratory that Conducted Analyses	Axys Analytical Services, Sidney, British Columbia
Submitting Organization	King County Department of Natural Resources and Parks

The purpose of this study was to evaluate how the atmospheric deposition of pollutants in the Green-Duwamish River Basin varies with land use type and proximity to various levels of urbanization. The objectives of this study were to compare the measurements of bulk deposition (dry particulates and precipitation) at a small number of stations in areas of different land uses within the Green-Duwamish River Basin and to provide information to assist in understanding atmospheric sources to the LDW.

Five stations representing various land uses located in the Lower Duwamish Valley and the Green River watershed were sampled for bulk atmospheric deposition (wet and dry deposition) of metals and organics between July 2011 and October 2012. Two stations were located in the urban areas of the LDW: the Duwamish and South Park stations. The Duwamish station represents the most industrial area; whereas, the South Park station represents a mixture of industrial/commercial and residential land uses. Of the remaining stations, one station was in an urban residential neighborhood (Beacon Hill), one station was located in a suburban/commercial area (Kent), and one station was located in the rural area of Enumclaw (Mud Mountain). A sixth station was added in the Kent area towards the end of the study period for paired comparison with the original Kent station; microscale effects (deposition rate differences due to differences in local sources and conditions) were suspected in Kent after interim review of the dioxin/furan congener data. A total of 47 samples were collected, with 8 and 10 samples collected at the Duwamish and South Park stations, respectively (King County 2013b).

King County LDW Supplemental Bulk Atmospheric Deposition Study (2013)

Study Code / EIM Study Identifier	39 / not in EIM
Sample Matrix/Matrices	Air deposition
Number of Locations Sampled	3 locations (LDW)
Number of Samples	15 samples (15 – full suite of congeners)
Number of Congener Locations/Samples	3 locations / 15 samples
PCB Analysis Method	Congeners: EPA 1668C
Laboratory that Conducted Analyses	Congeners: Axys Analytical Services, Sidney, British Columbia
Submitting Organization	King County Department of Natural Resources and Parks

This study supplements the 2011/2012 Bulk Atmospheric Deposition Study described above; it was designed in response to a recommendation from the 2012 study to collect additional PCB and dioxin/furan congener data to increase the ability to detect temporal trends and relationships with weather parameters and air concentrations of fine particulate matter. The 2012 report also recommended adding a third sampling location in the Duwamish River Valley to evaluate variability between stations.

This Supplemental Air Deposition Study includes monitoring at three stations in the LDW valley, two of which were previously monitored during the 2011/2012 study, and one station that was new in 2013 (King County 2015a). All samples were analyzed for the full suite of PCB congeners and were collected between April and November 2013. Samples were also collected at the Beacon Hill station but were not analyzed for PCBs.

8.2.5 Storm Drain System Studies

NPDES Inspection Sampling Support (2013-2015)

Study Code / EIM Study Identifier	20 and 21 / LDWISS
Sample Matrix	Storm drain solids and water
Number of Locations Sampled	80 locations (all LDW)
Number of Samples	97 samples: 57 storm drain solids (30 – full suite of congeners, 57 – Aroclors); 40 storm drain water (40 – full suite of congeners, 2 – Aroclors)
Number of Congener Locations/Samples	70 locations / 70 samples (30 storm drain solids, 40 water)
PCB Analysis Method	Congeners: EPA 1668C; Aroclors: EPA SW8082A
Laboratory that Conducted Analyses	Congeners: Vista Analytical Laboratory, El Dorado Hills, California (2014/2015 samples) and SGS Environmental Services, Wilmington, North Carolina (2013 samples); Aroclors: TestAmerica, Tacoma, Washington (2014/2015 samples) and Analytical Resources, Inc., Seattle, Washington (2013 samples)
Submitting Organization	Leidos for Ecology

The purpose of this study was to investigate the presence of contaminants in stormwater and storm drain solids at NPDES-permitted facilities discharging to the LDW. All 57 solids samples were analyzed for Aroclors; the 30 solids samples collected during 2014/2015 were analyzed for PCB congeners. All 40 water samples were analyzed for the full suite of PCB congeners, and 2 water samples collected during the 2013 effort were analyzed for Aroclors (Leidos 2015a, 2015b). Samples were collected from storm drain structures at 80 locations representing 24 permitted facilities.

S 96th Street/Hamm Creek Sediment Trap and Creek Sampling (2015)

Study Code / EIM Study Identifier	22 / LDWISS
Sample Matrix	Storm drain solids and water
Number of Locations Sampled	6 locations (LDW)
Number of Samples	10 samples: 6 storm drain solids (6 – full suite of congeners, 6 – Aroclors); 4 water (4 – full suite of congeners, 4 – Aroclors)
Number of Congener Locations/Samples	6 locations / 10 samples (see above)
PCB Analysis Method	Congeners: EPA 1668C; Aroclors: EPA SW8082A
Laboratory that Conducted Analyses	Congeners: Vista Analytical Laboratory, El Dorado Hills, California; Aroclors: TestAmerica, Tacoma, Washington
Submitting Organization	Leidos for Ecology

The purpose of this study was to characterize the water, sediment, and storm drain solids in the S 96th Street storm drain and Hamm Creek drainage subbasins. The S 96th Street storm drain subbasin includes the north and middle forks of Hamm Creek, and the Hamm Creek subbasin includes the south fork of Hamm Creek. Sediment trap solids and grab water samples were collected from conveyance structures in the S 96th Street drainage basin. Creek sediment, base flow, and storm event grab samples were collected from the north fork of Hamm Creek. Sediment trap solids, creek sediments, and storm event grab samples were collected from the south fork of Hamm Creek. All samples were analyzed for PCBs as Aroclors and congeners (Leidos 2015c).

8.2.6 Other Fish/Shellfish Studies

Several fish/shellfish studies were identified in EIM that contained PCB congener data and samples within the study area. Samples were collected between 1995 and 2012. Most of these studies analyzed a subset of PCB congeners. The studies are described briefly below.

NOAA Chinook Salmon Bioaccumulation Study (1989-1990)

Study Code / EIM Study Identifier	31 / NOAASALM
Sample Matrix	Fish tissue (Chinook salmon)
Number of Locations Sampled	1 location (LDW)
Number of Samples	11 samples (homologs)
Number of Congener Locations/Samples	1 location / 11 samples
PCB Analysis Method	Not identified in EIM
Laboratory that Conducted Analyses	Not identified in EIM
Submitting Organization	NOAA

This data set was downloaded from EIM (EIM Study Identifier NAASALM). The purpose of this bioaccumulation study was identified in EIM as sediment toxicity assessment. Chinook salmon were collected from four locations in King, Pierce, and Snohomish Counties between May 1989 and June 1990 (Varanasi et al. 1993); one of these locations was in the Duwamish River. Samples were identified as stomach contents, liver, or hepatopancreas. A total of 11 samples were analyzed for PCBs as homologs.

USFWS Bioaccumulation Study (1998)

Study Code / EIM Study Identifier	35 / USFHERON
Sample Matrix	Tissue (amphipod)
Number of Locations Sampled	1 location (LDW)
Number of Samples	6 samples (6 – subset of congeners)
Number of Congener Locations/Samples	1 location / 6 samples
PCB Analysis Method	Not identified in EIM
Laboratory that Conducted Analyses	Not identified in EIM
Submitting Organization	USFWS

The purpose of this U.S. Fish and Wildlife Service (USFWS) study was to examine bioaccumulation. Only one sample location was associated with this study in the EIM data query. The sample location is probably incorrect because it is inland, west of the main roadway, and the matrix was tissue from gammarid amphipods, which are aquatic species. The actual location was probably somewhere in the wetlands/intertidal area adjacent to Herring's House to the east. Six samples were collected in April 1998 and analyzed for a subset of 15 congeners (listed below).

PCB-77	PCB-126	PCB-157
PCB-101	PCB-128	PCB-169
PCB-105	PCB-138	PCB_170
PCB-110	PCB-153	PCB-180
PCB-118	PCB-156	PCB-189

Elliott Bay/Duwamish River Fish Tissue and Bioaccumulation Investigation (1995)

Study Code / EIM Study Identifier	8 / EVS95
Sample Matrix	Fish tissue
Number of Locations Sampled	8 locations (3 – LDW, 3 – West Waterway, 2 – East Waterway)
Number of Samples	8 samples (8 – subset of congeners, 8 – Aroclors)
Number of Congener Locations/Samples	8 locations (see above) / 8 samples
PCB Analysis Method	Not identified in EIM
Laboratory that Conducted Analyses	Not identified in EIM
Submitting Organization	TerraStat Consulting Group, Snohomish, Washington

The purpose of this study was to investigate the nature and extent of contamination to support human health risk assessment. Eight fish samples were collected from the LDW around Harbor Island to just south of Slip 1, approximately adjacent to United Western Supply. At least one set of sample location coordinates is incorrect, as they map on the land (EVS95-EW-01184). Based on its location name and the location names of the two samples that map in the East Waterway, it is likely this sample was also in the East Waterway, but its latitude was entered incorrectly in the database; the longitude for this sample is aligned with the East Waterway.

The EIM data query indicates the samples were composites of skin-off fillets from English sole collected in December 1995. Each sample was analyzed for PCBs as Aroclors and a subset of 23 individual congeners (listed below).

PCB-52	PCB-118	PCB-151	PCB-180
PCB-75	PCB-126	PCB-153	PCB-183
PCB-77	PCB-128	PCB-169	PCB-187
PCB-81	PCB-138	PCB-170	PCB-195
PCB-101	PCB-141	PCB-174	PCB-206
PCB-105	PCB-149	PCB-177	

LDW Injury Assessment – Chinook Salmon and Shiner Perch (2000)

Study Code / EIM Study Identifier	30 / NOAA-sal
Sample Matrix	Fish tissue (Chinook salmon)
Number of Locations Sampled	4 locations (2 – LDW, 2 – Green-Duwamish River)
Number of Samples	55 samples (55 – subset of congeners)
Number of Congener Locations/Samples	4 locations (see above) / 55 samples
PCB Analysis Method	Not identified in EIM
Laboratory that Conducted Analyses	Not identified in EIM
Submitting Organization	TerraStat Consulting Group, Snohomish, Washington

The purpose of this study was to investigate the nature and extent of contamination to support ecological and human health risk assessment. Four locations were sampled in May 2000: two in the LDW (with one at Kellogg Island and one in Slip 4), and two upstream locations. There appears to be some error in the coordinates reported for sample locations; the Slip 4 location coordinates provided in EIM are inland on the south side of the slip. A total of 55 Chinook salmon samples were collected and analyzed: 23 at Kellogg Island, 12 in Slip 4, and 20 at the 2 upstream sites. Only Chinook salmon samples were included in the PCB data set, including 48 whole body and 7 digestive system (gut content) samples. A subset of 14 congeners was analyzed (listed below).

PCB-66	PCB-110	PCB-153	PCB-180
PCB-77	PCB-118	PCB-156	PCB-189
PCB-101	PCB-126	PCB-157	
PCB-105	PCB-138	PCB-169	

PSAMP Groundfish Contaminant Survey – PCB Reanalysis (2007)

Study Code / EIM Study Identifier	17 / LDWEnglishSole2007
Sample Matrix	Fish tissue (English sole)
Number of Locations Sampled	1 location (trawl) (LDW)
Number of Samples	6 samples (6 – full suite of congeners, 6 – Aroclors)
Number of Congener Locations/Samples	1 location / 6 samples
PCB Analysis Method	Congeners: EPA 1668A; Aroclors: EPA SW8082, Revision 0
Laboratory that Conducted Analyses	Congeners: Axys Analytical Services, Sidney, British Columbia; Aroclors: Analytical Resources, Inc., Seattle, Washington
Submitting Organization	Windward Environmental for LDWG

Samples were initially collected by the Washington Department of Fish and Wildlife (WDFW) as part of the Puget Sound Ambient Monitoring Program (PSAMP). English sole were collected in May 2007 from several trawls in greater Elliott Bay (WDFW 2007, as cited in Ecology 2008a). Skinless fillets from 20 individual fish were combined into 6 composite samples from each of the trawl sites. A subset of PCB congeners was measured in each of these tissue samples. This data set was not found in EIM.

As part of the LDW RI, the LDWG reanalyzed six of these tissue samples (from a single location) for Aroclors and the full suite of congeners. A formal report summarizing the reanalysis of these samples was never formally prepared, but the data were submitted to EIM and have been included in the PCB Congener Database.

Fish, Crab, and Clam Tissue Collection and Chemical Analyses in the LDW (2007)

Study Code / EIM Study Identifier	19 / LDWFishCrabClam2007
Sample Matrix	Fish/shellfish tissue
Number of Locations Sampled	10 locations (LDW)
Number of Samples	86 samples: 17 – full suite of congeners; 86 – Aroclors
Number of Congener Locations/Samples	3 locations / 17 samples
PCB Analysis Method	Congeners: EPA 1668A; Aroclors: EPA SW8082, Revision 0
Laboratory that Conducted Analyses	Congeners: Axys Analytical Services, Sidney, British Columbia; Aroclors: Analytical Resources, Inc., Seattle, Washington
Submitting Organization	Windward Environmental for LDWG

The purpose of this study was to provide additional insight into PCB concentrations in tissue of six aquatic species found in the LDW. Data had been collected previously in 1997, 2004, 2005, and 2006. In combination, these data sets provide a time series of PCB concentrations in fish and crab tissues collected from the LDW and inform the design and interpretation of future monitoring studies. All 87 of the composite samples were analyzed for PCBs as Aroclors, and a subset of 17 samples was also analyzed for the full suite of PCB congeners. Congener analyses included the following species (number of samples in parentheses): slender crab (two, Dungeness crab (three), shiner perch (six), English sole (six). None of the starry flounder or soft-shell clam samples was analyzed for congeners.

WDFW PSEMP – Toxic Contaminants in Dungeness Crab and Spot Prawn from Puget Sound (2011)

Study Code / EIM Study Identifier	3 / C1200226
Sample Matrix	Shellfish tissue
Number of Locations Sampled	1 location (trawl) – LDW
Number of Samples	1 sample (subset of congeners)
Number of Congener Locations/Samples	1 location / 1 sample
PCB Analysis Method	Congeners: NOAA-NMFS-NWFSC-59 (Organic Contaminants in Sediments and Tissues by Extraction, Cleanup, and Gas Chromatography/Mass Spectrometry)
Laboratory that Conducted Analyses	Congeners: NOAA NW Fisheries Science Center Environmental Laboratory, Seattle, Washington
Submitting Organization	WDFW

This study was conducted and managed by the WDFW Puget Sound Environmental Monitoring Program (PSEMP). This study was a broad-scale, Puget Sound-wide assessment of toxic contaminants in Dungeness crab and spot prawn. The purpose of the study was to evaluate the geographic extent and magnitude of toxic contaminants in these two crustacean species in Puget Sound and to provide contaminant data to the Washington State Department of Health for a human health risk assessment.

One trawl was conducted in the LDW, and a single Dungeness crab sample was collected; no spot prawns were collected during this trawl. A total of 46 PCB congeners (listed below) were analyzed from the edible muscle portion of the crab (WDFW 2014).

PCB-17	PCB-74	PCB-132/153	PCB-180
PCB-18	PCB-82	PCB-138/163/164	PCB-183
PCB-28	PCB-87	PCB-149	PCB-191
PCB-31	PCB-95	PCB-151	PCB-194
PCB-33	PCB-99	PCB-156	PCB-195
PCB-44	PCB-90/101	PCB-158	PCB-199
PCB-49	PCB-105	PCB-159/182/187	PCB-205
PCB-52	PCB-110	PCB-170	PCB-206
PCB-66	PCB-118	PCB-171	PCB-208
PCB-70	PCB-128	PCB-177	PCB-209

8.2.7 Other Sediment Studies

LDW Sediment Characterization (1997)

Study Code / EIM Study Identifier	29 / NOAA97
Sample Matrix	Sediment
Number of Locations Sampled	326 locations (304 – LDW, 22 – Green-Duwamish River)
Number of Samples	326 samples (326 – subset of congeners)
Number of Congener Locations/Samples	326 locations (see above) / 326 samples
PCB Analysis Method	Congeners and Aroclors: Methylene chloride extraction followed by high-performance liquid chromatography with photodiode array detection.
Laboratory that Conducted Analyses	NWFSC-ECD
Submitting Organization	NOAA

The purpose of this study was to characterize sediment contamination in the LDW. Surface sediment samples (0 to 10 cm) were collected from 326 locations in the LDW and upstream areas during September through November 1997 (NOAA 1998). Samples were analyzed for a subset of 15 congeners (listed below).

PCB-77	PCB-126	PCB-157
PCB-101	PCB-128	PCB-169
PCB-105	PCB-138	PCB_170
PCB-110	PCB-153	PCB-180
PCB-118	PCB-156	PCB-189

Lower Duwamish River – EPA Site Inspection (1998)

Study Code / EIM Study Identifier	28 / LODRIV98
Sample Matrix	Sediment
Number of Locations Sampled	299 locations (292 – LDW, 1 – West Waterway, 6 – Green-Duwamish River)
Number of Samples	332 (332 – subset of congeners, 332 – Aroclors)
Number of Congener Locations/Samples	299 locations (see above) / 332 samples
PCB Analysis Method	Not identified in EIM
Laboratory that Conducted Analyses	Not identified in EIM
Submitting Organization	Roy F. Weston for EPA

The study was performed to provide sediment data for EPA's site investigation process, which evaluated actual or potential hazards at the LDW, relative to other sites, for the purpose of identifying remedial action priorities. The data report indicates that 300 unique locations were sampled, but only 299 sampling locations are included in the data queried from the EIM database. A total of 332 surface and subsurface sediment samples were collected in the lower 6 miles of the Duwamish River in August and September 1998. Each sample was analyzed for PCBs as Aroclors and a subset of 27 individual congeners, listed below (Weston 1999).

PCB-18	PCB-101	PCB-138	PCB-180
PCB-28	PCB-105	PCB-153	PCB-187
PCB-42	PCB-114	PCB-156	PCB-189
PCB-52	PCB-118	PCB-157	PCB-195
PCB-66	PCB-123	PCB-167	PCB-206
PCB-77	PCB-126	PCB-169	PCB-209
PCB-81	PCB-128	PCB-170	

PSAMP and NOAA NS&T (1998)

Study Code / EIM Study Identifier	33 / PSAMPNOA
Sample Matrix	Sediment
Number of Locations Sampled	7 locations (3 – LDW, 3 – East Waterway, 1 – West Waterway)
Number of Samples	7 (7 – subset of congeners, 7 – Aroclors)
Number of Congener Locations/Samples	7 locations (see above) / 7 samples
PCB Analysis Method	EPA SW8081/8082 combined method
Laboratory that Conducted Analyses	Ecology Manchester Environmental Laboratory, Port Orchard, Washington
Submitting Organization	Not identified in EIM

In 1997, Ecology entered into a Cooperative Agreement with NOAA's NS&T Program to jointly examine measures of sediment quality throughout Puget Sound. This 3-year monitoring effort consisted of focused studies, with 100 stations sampled annually in north, central, and south Puget Sound using a stratified random sampling approach.

A total of seven sediment samples were collected within the LDW and East/West Waterway, one from each of seven locations, during June and July 1998. Each sample was analyzed for PCBs as Aroclors and a subset of 20 congeners (listed below).

PCB-8	PCB-66	PCB-126	PCB-180
PCB-18	PCB-77	PCB-128	PCB-187
PCB-28	PCB-101	PCB-138	PCB-195
PCB-44	PCB-105	PCB-153	PCB-206
PCB-52	PCB-118	PCB-170	PCB-209

Sediment Characterization of Duwamish River Navigation Channel (1999)

Study Code / Study Identifier	34 / PSDDDA99
Sample Matrix	Sediment
Number of Locations Sampled	20 locations (LDW)
Number of Samples	20 samples (3 – subset of congeners, 20 – Aroclors)
Number of Congener Locations/Samples	3 locations / 3 samples
PCB Analysis Method	Congeners: EPA 1668A; Aroclors: EPA SW8082, Revision 0
Laboratory that Conducted Analyses	Not identified in EIM
Submitting Organization	Not identified in EIM

The purpose of this study was to monitor PCBs at maintenance dredging areas in the LDW. A single sediment sample was collected from each of 20 locations. Samples were collected in August 2009; collection depths were listed as 0 to 4 feet or 4 to 8 feet. All 20 sediment samples were analyzed for PCBs as Aroclors. Three of these samples were also analyzed for a subset of PCB congeners, including the 12 WHO dioxin-like PCBs (listed below) plus congeners PCB-170 and PCB-180. The LDW RI noted that, although the area was proposed for dredging, no dredging had been conducted since the samples were collected (Windward 2010b).

PCB-11	PCB-118	PCB-157	PCB-180
PCB-81	PCB-123	PCB-167	PCB-189
PCB-105	PCB-126	PCB-169	
PCB-114	PCB-156	PCB-170	

EMAP Coastal Sampling (2002)

Study Code / EIM Study Identifier	7 / EMAP_1999-2002
Sample Matrix	Sediment, fish tissue
Number of Locations Sampled	1 location (East Waterway)
Number of Samples	2 samples (subset of congeners)
Number of Congener Locations/Samples	1 location / 2 samples
PCB Analysis Method	EPA SW8082 (as listed in EIM)
Laboratory that Conducted Analyses	Ecology Manchester Environmental Laboratory, Port Orchard, Washington
Submitting Organization	Hart Crowser for EPA

EPA's Environmental Monitoring and Assessment Program (EMAP) coastal sampling was conducted under the National Coastal Condition Assessment, previously National Coastal Assessment. The purpose was to survey the condition of the U.S. coastal resources by creating an integrated, comprehensive monitoring program among the coastal states. One location on the east side of Harbor Island was sampled; one surface sediment sample and one English sole tissue sample were collected in September 2002. The samples were analyzed for a subset of 21 individual congeners (listed below).

PCB-8	PCB-101	PCB-169
PCB-18	PCB-105	PCB-170
PCB-28	PCB-118	PCB-180
PCB-44	PCB-126	PCB-187
PCB-52	PCB-128	PCB-195
PCB-66	PCB-138	PCB-206
PCB-77	PCB-153	PCB-209

T117 Early Action Area Non-Time-Critical Removal Action (2003-2006 and 2008-2011)

Study Code / EIM Study Identifier	11 / G0800557
Sample Matrix	Sediment, groundwater, soil
Number of Locations Sampled	302 locations (LDW)
Number of Samples	913 samples: 183 sediment (2 – subset of congeners, 183 – Aroclors); 136 groundwater (136 – Aroclors); 594 soil (594 – Aroclors)
Number of Congener Locations/Samples	2 locations / 2 samples
PCB Analysis Method	Congeners: EPA 1668A; Aroclors: EPA SW8082 Revision 0 and Revision 1
Laboratory that Conducted Analyses	Congeners: Axys Analytical Services, Sidney, British Columbia Canada; Aroclors: Analytical Resources, Inc., Seattle, Washington; Columbia Analytical Services, Kelso, Washington
Submitting Organization	Port of Seattle

The purpose of this study was the investigation and cleanup of PCB-contaminated soil and sediment as a Comprehensive Environmental Response, Compensation, and Liability Act action for LDW source control. The T117 Early Action Area consists of: (1) sediment-aquatic portion of the site within the LDW; (2) upland area that was the site of historical upland activities; and (3) street and residential yards east of 14th Avenue S bounded by Dallas Avenue S and S Donovan Street. The groundwater samples were collected over several months during each year from 2008 through 2011. Soil and sediment samples were collected from 2003 through 2006 and in 2008 and included surface and subsurface samples.

PCB results were reported as congeners for only two of the hundreds of samples collected in this study. Two sediment samples, 1 collected in 2004 and 1 collected in 2005, were analyzed for a subset of 18 congeners (listed below). All other sediment samples and all of the soil and groundwater samples were analyzed for PCBs as Aroclors.

PCB-66	PCB-110	PCB-129	PCB-169
PCB-77	PCB-114	PCB-153	PCB-180
PCB-81	PCB-118	PCB-156	PCB-189
PCB-90	PCB-123	PCB-157	
PCB-105	PCB-126	PCB-167	

Urban Waters Initiative, Sediment Quality in Elliott Bay and LDW (2007)

Study Code / EIM Study Identifier	36 / UWI2007
Sample Matrix	Sediment
Number of Locations Sampled	7 locations (3 – LDW, 3 – East Waterway, 1 – West Waterway)
Number of Samples	8 samples (8 – subset of congeners, 8 – Aroclors)
Number of Congener Locations/Samples	7 locations (see above) / 8 samples
PCB Analysis Method	EPA SW8081/8082 combined method (as listed in EIM)
Laboratory that Conducted Analyses	Ecology Manchester Environmental Laboratory, Port Orchard, Washington
Submitting Organization	Ecology Environmental Assessment Program

The purpose of the 2007 Urban Waters Initiative study was to gauge the long-term effectiveness of collective toxics management efforts in Elliot Bay and the LDW. The objectives were to assess the current conditions in the Bay, particularly the overall extent of sediment contamination, and to determine whether there had been changes in sediment quality over time. Eight sediment samples were collected and analyzed for PCBs as Aroclors. Each sample was also analyzed for a subset of 21 congeners, listed below (Ecology 2009a).

PCB-8	PCB-101	PCB-169
PCB-18	PCB-105	PCB-170
PCB-28	PCB-118	PCB-180
PCB-44	PCB-126	PCB-187
PCB-52	PCB-128	PCB-195
PCB-66	PCB-138	PCB-206
PCB-77	PCB-153	PCB-209

PSAMP Spatial/Temporal Monitoring (2009)

Study Code / EIM Study Identifier	32 / PSAMP_SP
Sample Matrix	Sediment
Number of Locations Sampled	4 locations (2 – LDW, 2 – East/West Waterway)
Number of Samples	4 samples (4 – subset of congeners, 4 – Aroclors)
Number of Congener Locations/Samples	4 locations (see above) / 4 samples
PCB Analysis Method	EPA SW8081/8082 combined (as listed in EIM)
Laboratory that Conducted Analyses	Ecology Manchester Environmental Laboratory, Port Orchard, Washington
Submitting Organization	Ecology Environmental Assessment Program

A total of four surface sediment samples (0 to 3 cm), one at each of four locations, were collected in June 2009 as part of the PSAMP. The samples were analyzed for PCBs as Aroclors and for a subset of 21 individual congeners (listed below).

PCB-8	PCB-101	PCB-169
PCB-18	PCB-105	PCB-170
PCB-28	PCB-118	PCB-180
PCB-44	PCB-126	PCB-187
PCB-52	PCB-128	PCB-195
PCB-66	PCB-138	PCB-206
PCB-77	PCB-153	PCB-209

Duwamish Waterway, East Waterway, and West Waterway Subsurface Sediment Characterization (2012)

Study Code / EIM Study Identifier	**5 / DUWSU12**
Sample Matrix	Sediment
Number of Locations Sampled	27 locations (16 – LDW, 11 – East/West Waterway)
Number of Samples	70 samples (9 – full suite of congeners, 70 – Aroclors)
Number of Congener Locations/Samples	9 locations / 9 samples (6 – LDW, 1 – East Waterway, 2 – West Waterway)
PCB Analysis Method	Congeners: EPA 1668A; Aroclors: EPA SW8082A, Revision 1
Laboratory that Conducted Analyses	Congeners: ALS Laboratory Group, Burlington, Ontario; Aroclors: Analytical Resources, Inc., Seattle, Washington
Submitting Organization	U.S. Army Corps of Engineers, Seattle, Washington District

The purpose of this study was to determine the level of contamination in subsurface sediments of the LDW, East Waterway, and West Waterway; compare analytical techniques for PCB analysis; and monitor the Confined Aquatic Disposal site in the West Waterway. A total of 70 sediment samples were collected from 27 locations in the East/West Waterways and LDW in October 2012. Samples included 1 surface sample and 26 sediment cores, sampled at 2 to 5 depth intervals each. All samples were analyzed for Aroclors; a subset of nine samples (from nine sediment cores) was analyzed for the full suite of PCB congeners.

Urban Waters Initiative, Sediment Quality in Elliott Bay (2013)

Study Code /EIM Study Identifier	37 / UWI2013
Sample Matrix	Sediment
Number of Locations Sampled	7 locations (3 – LDW, 3 – East Waterway, 1 – West Waterway)
Number of Samples	8 samples (8 – subset of congeners, 8 – Aroclors)
Number of Congener Locations/Samples	7 locations (see above) / 8 samples
PCB Analysis Method	EPA SW8081/8082, combined method
Laboratory that Conducted Analyses	Ecology Manchester Environmental Laboratory, Port Orchard, Washington
Submitting Organization	Ecology Environmental Assessment Program

The purpose of the 2013 Urban Waters Initiative study was to gauge the long-term effectiveness of collective toxics management efforts in Elliott Bay. The objectives were to assess the current conditions in the study area, particularly the overall extent of sediment contamination, and to determine whether there had been changes in sediment quality over time.

Seven sediment samples plus one field duplicate were collected over a 3-day period in June. Three samples were collected in the LDW, three were collected in the East Waterway, and one sample was collected in the West Waterway. The samples were analyzed for PCBs as Aroclors and for a subset of 21 individual congeners, listed below. Five of these congeners (PCB-77, PCB-105, PCB-118, PCB-126, and PCB-169) are considered dioxin-like. Congeners PCB-77, PCB-126, and PCB-169 generally occurred in Aroclors in only trace amounts.

PCB-8	PCB-101	PCB-169
PCB-18	PCB-105	PCB-170
PCB-28	PCB-118	PCB-180
PCB-44	PCB-126	PCB-187
PCB-52	PCB-128	PCB-195
PCB-66	PCB-138	PCB-206
PCB-77	PCB-153	PCB-209

8.2.8 Soil/Other Solids Data

LDW Site, Boeing Split Samples (1989-2015)

Study Code / EIM Study Identifier	2 / Boeing Split
Sample Matrix	Soil, sediment, storm drain solids, and other solids
Number of Locations Sampled	125 locations (LDW)
Number of Samples	164 samples (10 – full suite of congeners, 154 – Aroclors)
Number of Congener Locations/Samples	8 locations / 10 samples (including 2 field replicates)
PCB Analysis Method	Congeners and homologs: EPA 1668A; Aroclors: EPA SW8082, Revision 0
Laboratory that Conducted Analyses	Congeners: Columbia Analytical Services, Kelso, Washington; Aroclors: Analytical Resources Inc., Seattle, Washington
Submitting Organization	Integral Consulting for City of Seattle

This study was performed to support litigation between the City of Seattle and Boeing. Types of samples in the data set include 8 sediment samples, 119 soil samples from various depths, 9 solids samples from catch basins, and 25 “other solids” samples (e.g., caulking material and solid material from the Georgetown Steam Plant condenser pit and furnace effluent vents). A total of 154 samples were analyzed for Aroclors, and an additional 8 samples were analyzed for the full PCB congener suite. Samples were collected between May 1989 and April 2015. The sampling appears to have been concentrated in the area of the Georgetown Steam Plant and buildings along the northwest perimeter of the site. Three of the sample locations were at the head of Slip 4. The eight locations for which congener analysis was performed were located at or very near the fence line separating North Boeing Field from the Georgetown Steam Plant property. These include three soil sampling locations (sampled in May 2006) and five samples of “other solids” (surface debris, sampled in July 2009).

8.3 Data Sets Not Included in the Polychlorinated Biphenyl Congener Database

Some of the studies containing PCB congener data that were initially downloaded from EIM or found on the EPA Superfund website have not been retained for this report. The rationale for each study’s exclusion is provided below.

8801 Site Storm Drain Solids (EIM Study Identifier FS2072A, 2008-2009)

This data set (Database Study Code 10, EIM Study Identifier FS2072A) was downloaded from EIM and was initially considered for inclusion because it included congener data. The purpose of this study was to investigate solids in a stormwater system. Four samples were collected; two each at two catch basin locations. PCBs were analyzed as Aroclors and a single congener, PCB-209. This may be an error in the EIM database; the study was, therefore, eliminated.

U.S. Coast Guard Pier 36 (EIM Study Identifier CGS31682, 2002)

This data set (Database Study Code 4, EIM Study Identifier CGS36182) was downloaded from EIM and was initially considered for inclusion because it included congener data. The purpose of this study was to support a Dredged Material Management Program recency extension. It includes one sediment sample that was analyzed for Aroclors and one congener – PCB-16. Because it is considered unlikely that only a single congener would be tested/reported, it is possible that when the data were being entered into the database, PCB-16 was accidentally selected instead of Aroclor 1016, which was not among the Aroclors included in the data. The study was, therefore, eliminated.

Burlington Northern Railroad-Scenic (EIM Study Identifier BNRR-SCE, 2001)

This data set (Database Study Code 1, EIM Study Identifier BNRR-SCE) was downloaded from EIM and was initially considered for inclusion because it included congener data. This study was an initial site investigation. The EIM database lists three different locations sampled during the study, but all three plot in the same location and the location notes state “Plant entrance” for each. A single soil sample was collected at each location, and the PCB analysis reported as Aroclors and a single congener, PCB-209. The study was, therefore, eliminated.

8.4 Summary of Available Data

A total of 36 studies that included at least 1 sample analyzed for PCB congeners within the study area have been identified. PCB results from these studies (including congener and Aroclor data) have been compiled in a PCB Congener Database. The database includes PCB results for 3,427 samples collected from a total of 1,989 sampling locations, including 1,555 locations in the LDW, 349 locations in the East/West Waterways, and 79 locations in the Green-Duwamish River. Of the 3,427 total samples:

- 18 percent (645 samples) were analyzed for the full suite of PCB congeners,
- 23 percent (798 samples) were analyzed for a subset of PCB congeners,
- <1 percent (12 samples) were analyzed for total PCB homologs,
- 77 percent (2,641 samples) were analyzed for Aroclors.

Table 8-1 summarizes PCB congener data availability by environmental medium; information is presented by geographic area (LDW, East/West Waterway, or Green River) and by analysis type (full congener suite or subset of congeners). Sample counts may include duplicate and replicate samples. Locations for samples collected in the LDW and East/West Waterway are shown on Figures 8-1 through 8-7. Locations for samples collected in the Green River are shown on Figure 8-8.

Table 8-1. Summary of Available PCB Congener Data – Green-Duwamish Watershed

Geographic Area	No. of Studies with this Data Type	No. Locations Sampled	No. of Samples	Years Covered
All Samples				
Full Suite of Congener Data				
LDW	13	142	249	2004 – 2015
Green River	8	25	229	2005 – 2015
East/West Waterway	3	33	115	2005 – 2012
Subsets of Congeners				
LDW	14	651	723	1995 – 2013
Green River	4	30	49	1997 – 2012
East/West Waterway	8	24	26	1995 – 2012
Sediment Samples				
Full Suite of Congener Data				
LDW	2	22	22	2004 – 2012
Green River	3	9	27	2008 – 2015
East/West Waterway	2	18	20	2009 – 2012
Subsets of Congeners				
LDW	10	645	678	1997 – 2013
Green River	2	28	28	1997 – 1998
East/West Waterway	6	16	18	1998 – 2013
Tissue Samples				
Full Suite of Congener Data				
LDW	5	69	92	2004 – 2007
Green River	0	0	0	NA
East/West Waterway	1	17	29	2008
Subsets of Congeners				
LDW	5	7	45	1995 – 2011
Green River	1	2	20	2000
East/West Waterway	1	8	8	1995
Surface Water Samples				
Full Suite of Congener Data				
LDW	1	1	10	2005
Green River	5	12	147	2005 – 2015
East/West Waterway	2	7	66	2005 – 2009
Subsets of Congeners				
LDW	0	0	0	NA
Green River	1	1	1	2012

Table 8-1. Summary of Available PCB Congener Data – Green-Duwamish Watershed

Geographic Area	No. of Studies with this Data Type	No. Locations Sampled	No. of Samples	Years Covered
East/West Waterway	0	0	0	NA
Suspended Solids Samples				
Full Suite of Congener Data				
LDW	0	0	0	NA
Green River	2	2	26	2013 – 2015
East/West Waterway	0	0	0	NA
Subsets of Congeners				
LDW	0	0	0	NA
Green River	0	0	0	NA
East/West Waterway	0	0	0	NA
Storm Drain Solids Samples				
Full Suite of Congener Data				
LDW	2	32	33	2014 – 2015
Green River	1	2	3	2014
East/West Waterway	0	0	0	NA
Subsets of Congeners				
LDW	0	0	0	NA
Green River	0	0	0	NA
East/West Waterway	0	0	0	NA
Storm Drain Water Samples				
Full Suite of Congener Data				
LDW	3	38	42	2013 – 2015
Green River	1	2	2	2014
East/West Waterway	0	0	0	NA
Subsets of Congeners				
LDW	0	0	0	NA
Green River	0	0	0	NA
East/West Waterway	0	0	0	NA
Air Deposition Samples				
Full Suite of Congener Data				
LDW	2	4	40	2011 – 2013
Green River	1	3	24	2011 – 2012
East/West Waterway	0	0	0	NA
Subsets of Congeners				
LDW	0	0	0	NA

Table 8-1. Summary of Available PCB Congener Data – Green-Duwamish Watershed

Geographic Area	No. of Studies with this Data Type	No. Locations Sampled	No. of Samples	Years Covered
Green River	0	0	0	NA
East/West Waterway	0	0	0	NA
Soil Samples				
Full Suite of Congener Data				
LDW	1	3	5	2006
Green River	0	0	0	NA
East/West Waterway	0	0	0	NA
Subsets of Congeners				
LDW	0	0	0	NA
Green River	0	0	0	NA
East/West Waterway	0	0	0	NA
Other Solids Samples				
Full Suite of Congener Data				
LDW	1	5	5	2009
Green River	0	0	0	NA
East/West Waterway	0	0	0	NA
Subsets of Congeners				
LDW	0	0	0	NA
Green River	0	0	0	NA
East/West Waterway	0	0	0	NA

LDW = Lower Duwamish Waterway.
NA = Not applicable.
No. = Number.
PCB = Polychlorinated biphenyl.

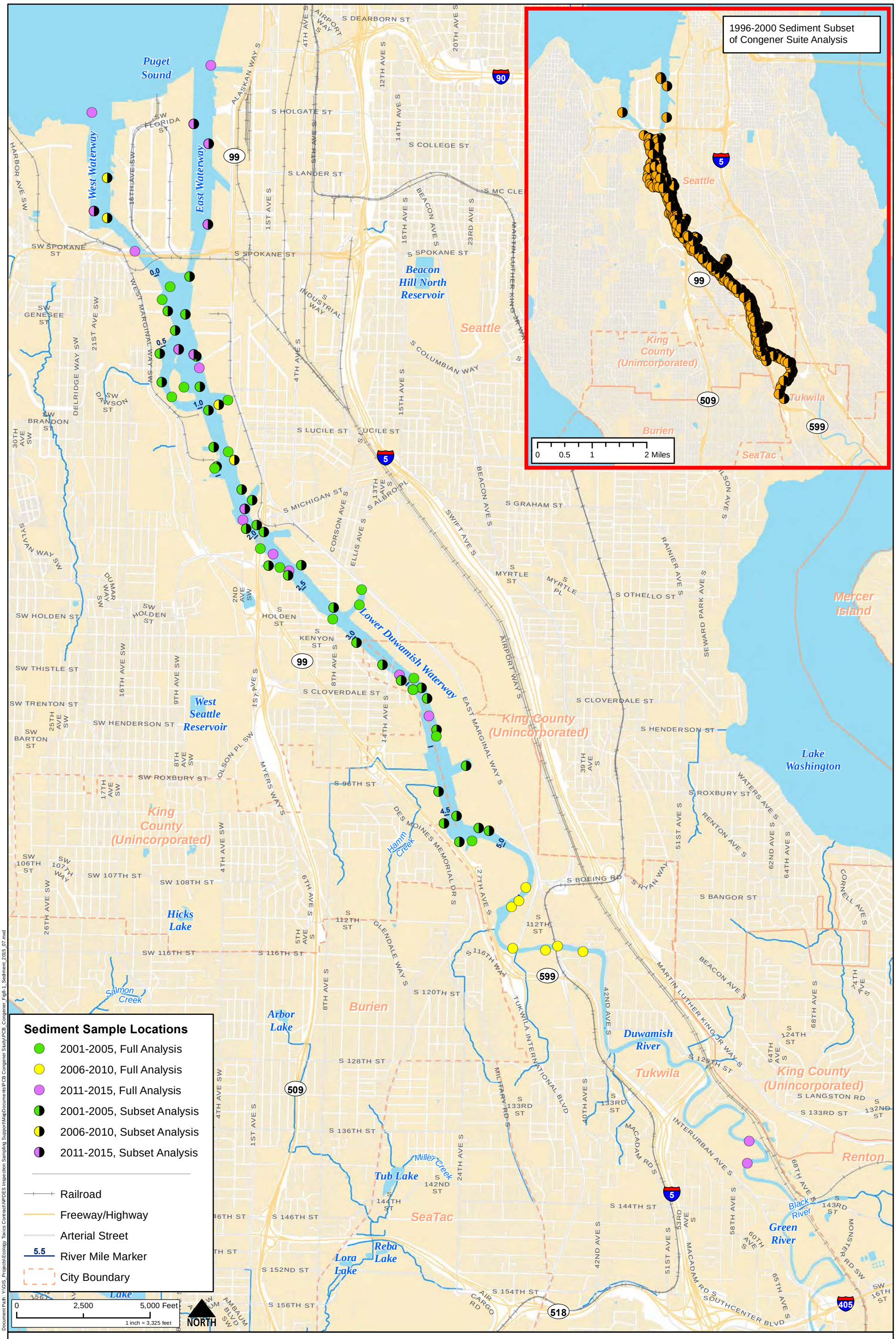

Figure 8-1. Green-Duwamish Sediment Sampling Locations: PCB Congeners

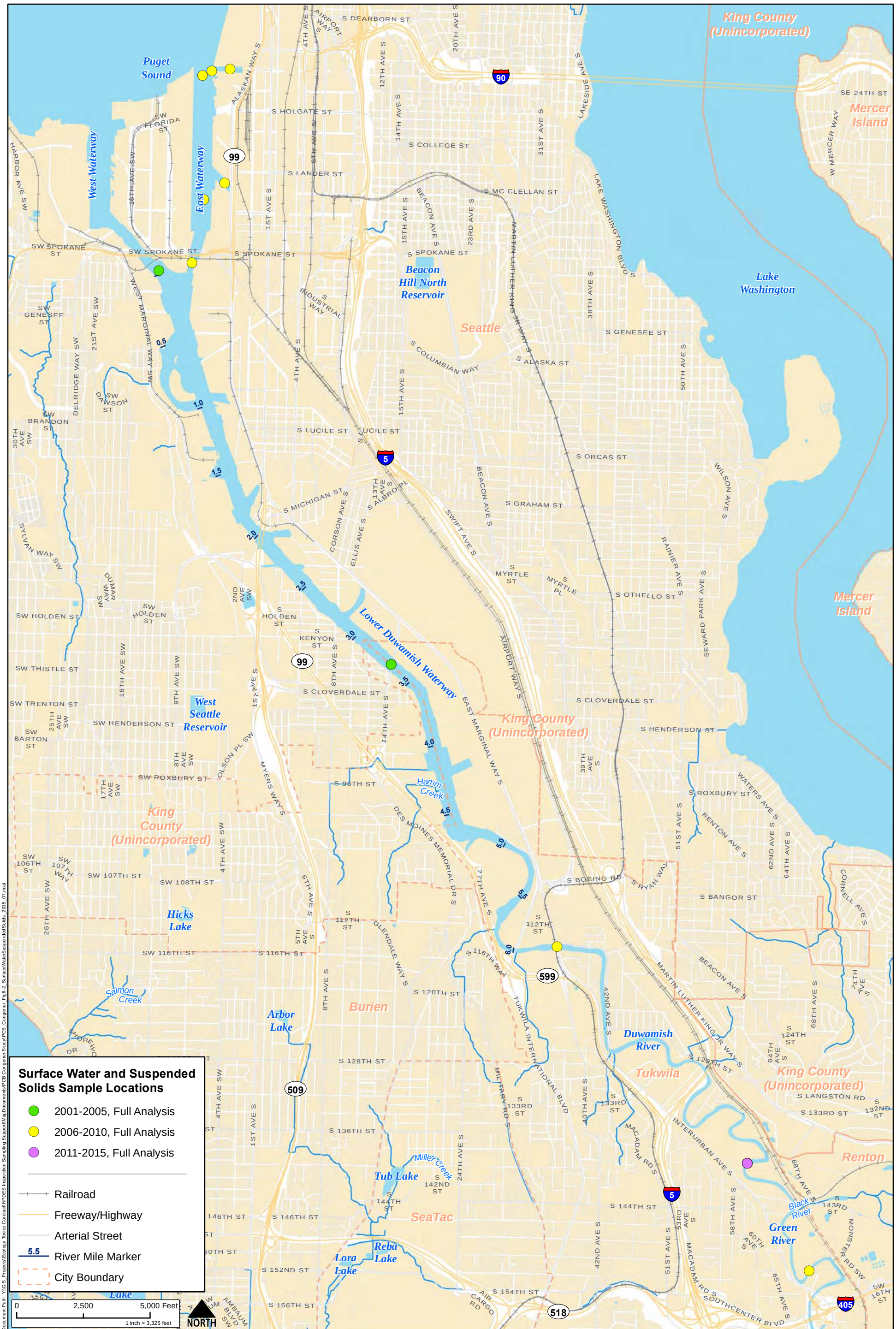

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Figure 8-2. Green-Duwamish Surface Water and Suspended Solids Sampling Locations: PCB Congeners

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Figure 8-3. Green-Duwamish Soil and Other Solids Sampling Locations: PCB Congeners

Figure 8-4. Green-Duwamish Tissue Sampling Locations: PCB Congeners

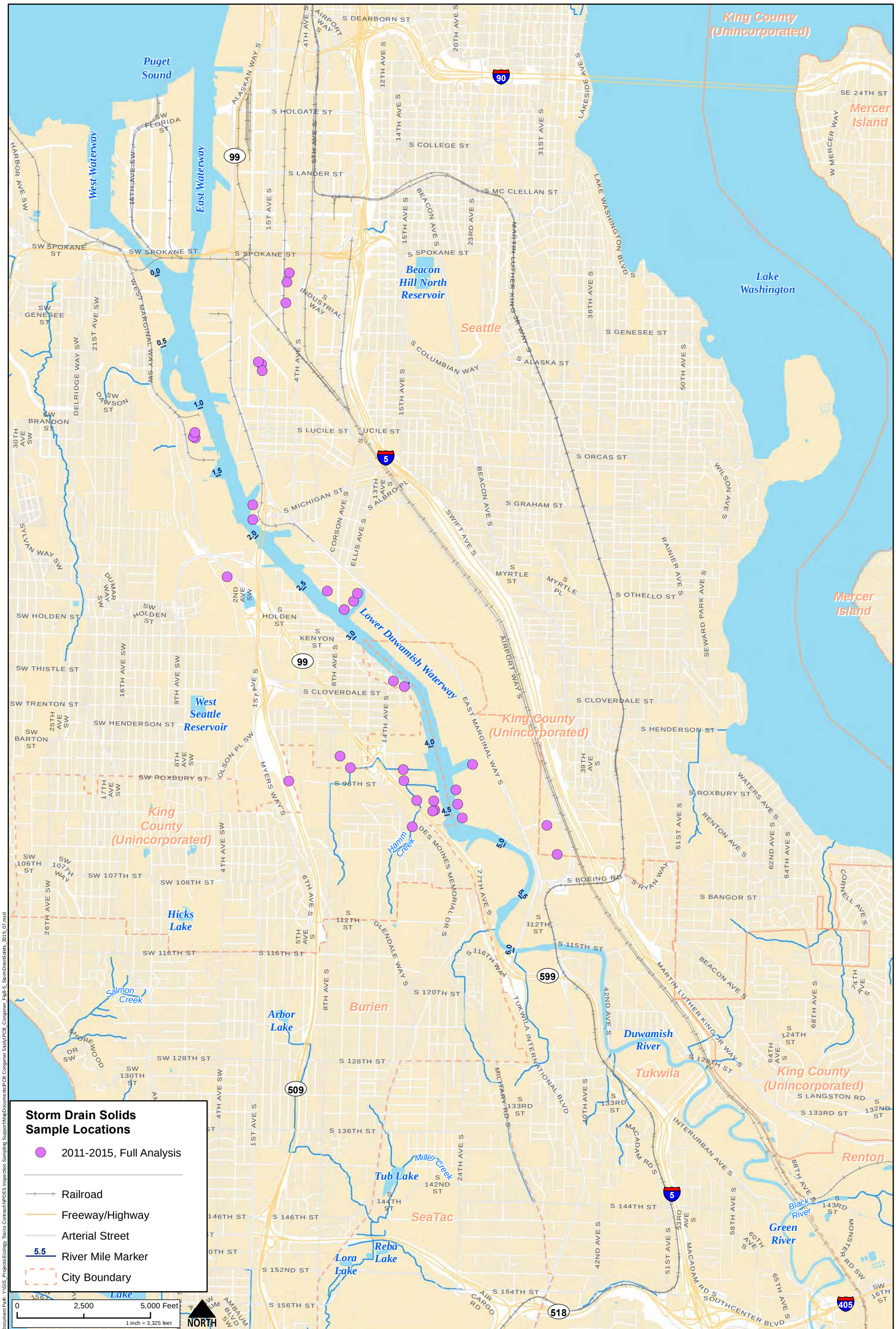

Figure 8-5. Green-Duwamish Storm Drain Solids Sampling Locations: PCB Congeners

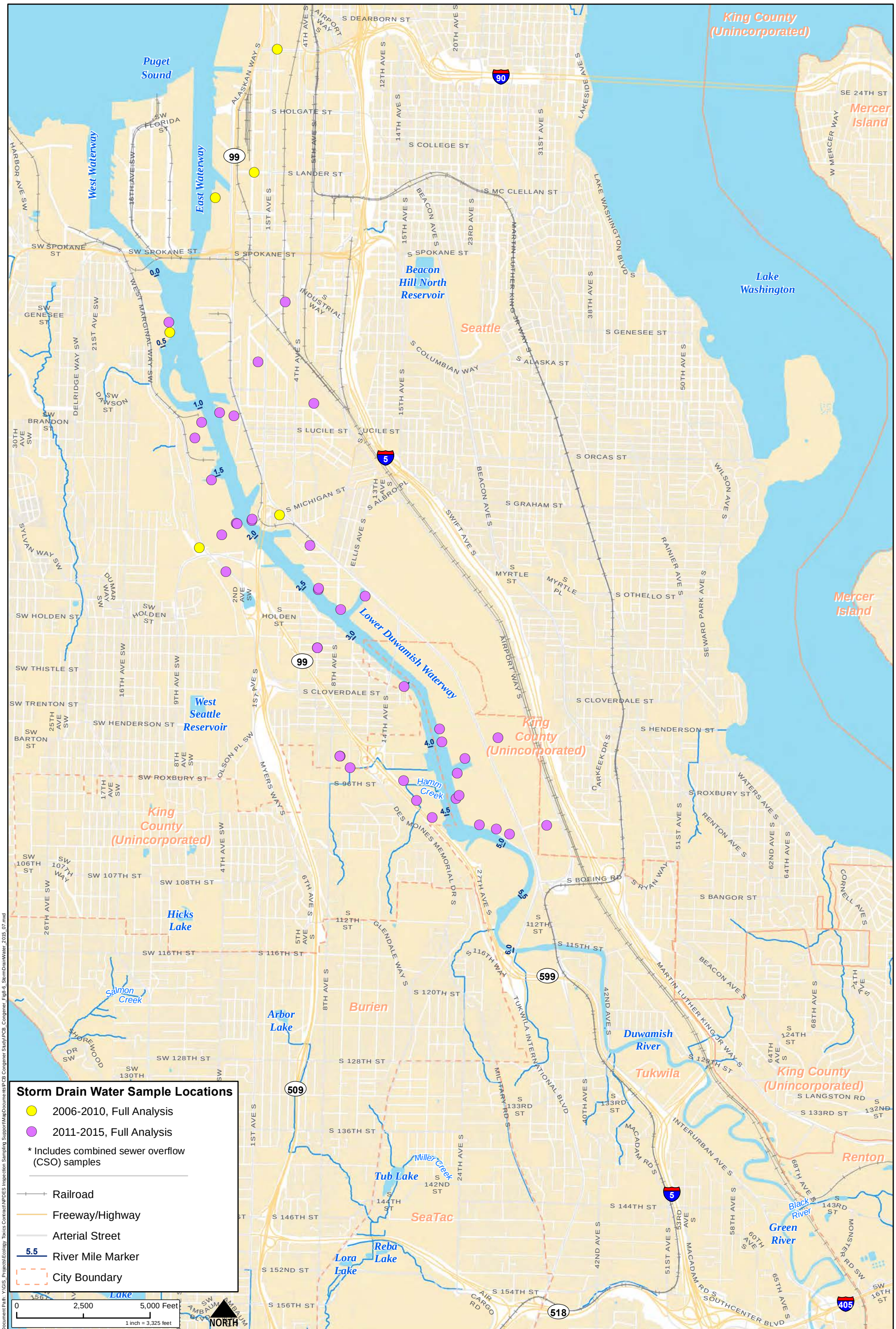

Figure 8-6. Green-Duwamish Storm Drain Water Sampling Locations: PCB Congeners

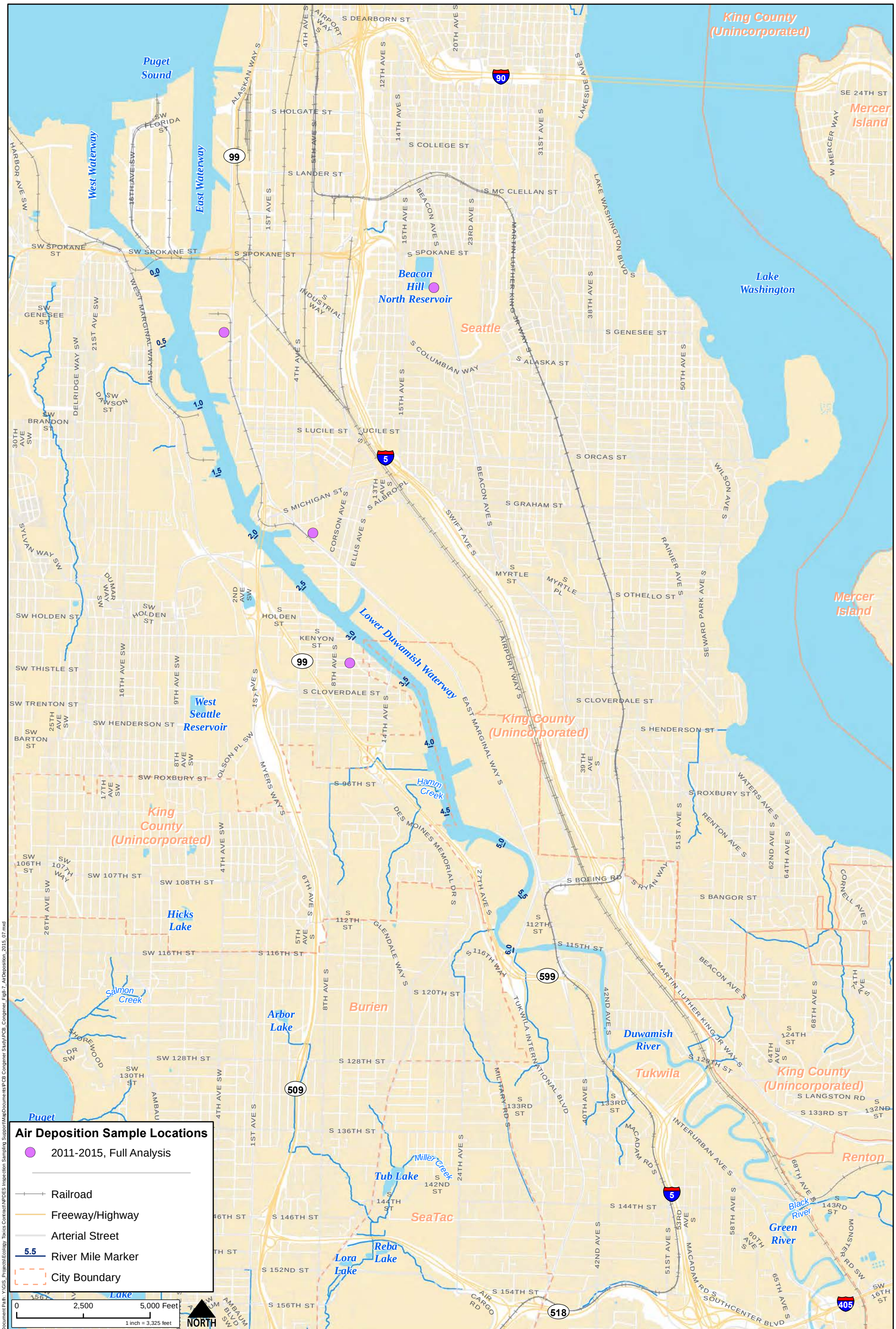

Figure 8-7. Green-Duwamish Air Deposition Sampling Locations: PCB Congeners

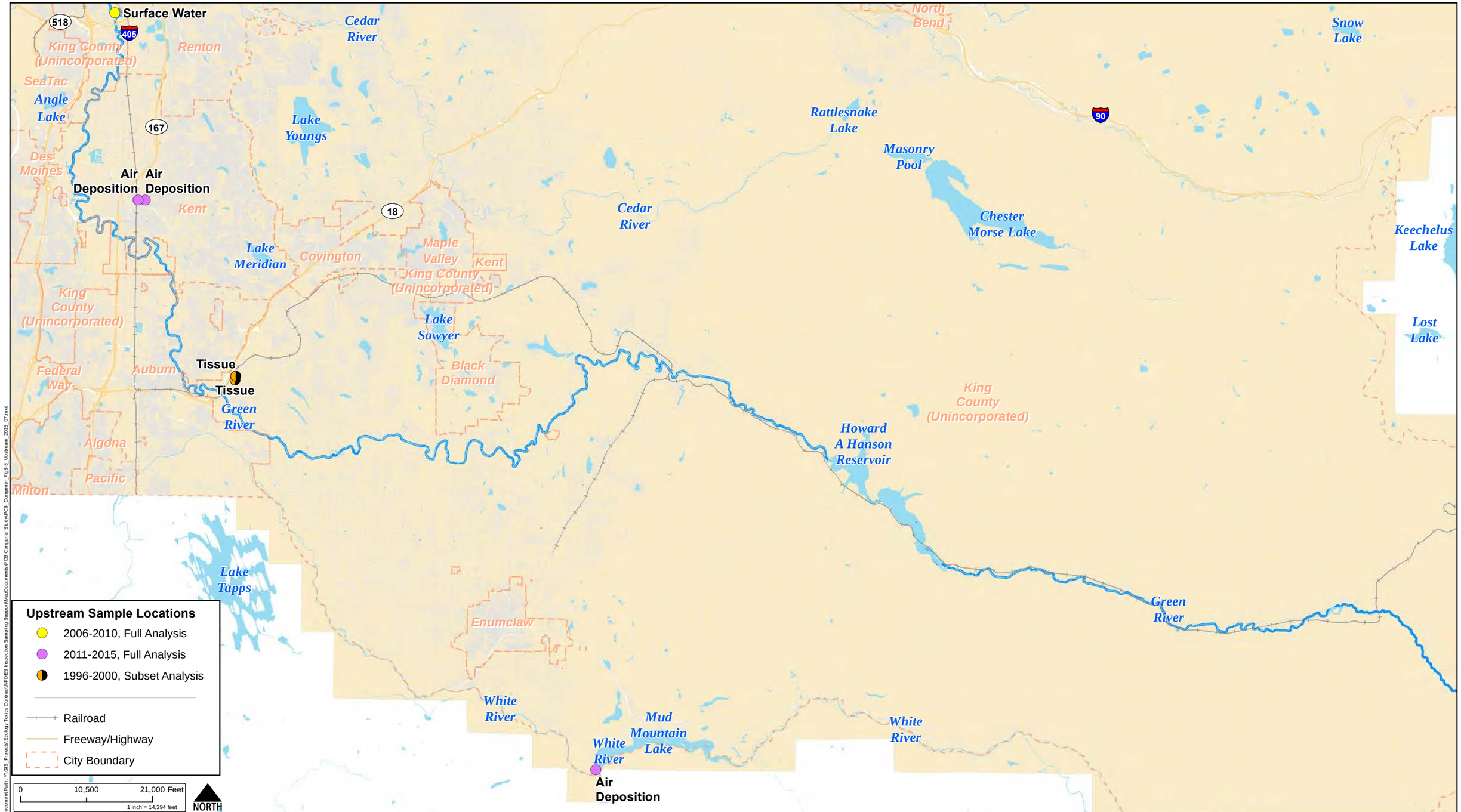

Figure 8-8. Green-Duwamish Upstream Sampling Locations: PCB Congeners

8.5 Recommendations for Data Management

One objective of this report is to make recommendations regarding data management so that PCB data sets collected in the future are comparable to each other and can be used for trend monitoring and modeling efforts. Dr. Lisa Rodenburg (Rutgers State University) recommended following applicable guidance set up by the Delaware River Basin Commission (DRBC) to assist NPDES permittees in complying with sampling requirements. The DRBC guidance includes a list of acceptable laboratories, requirements for the column to be used during analysis (SPB-octyl), how data must be analyzed, data output formats, data codes, naming conventions, how to report non-detects, etc. Implementation of these guidelines led to the generation of comparable data sets from the NPDES permittees in Delaware (see: <http://www.state.nj.us/drbc/quality/toxics/pcbs/monitoring.html>).

Relevant DRBC guidance is provided in Appendix C. This guidance was considered in the development of data recommendations for the LDW described below.

8.5.1 Require Use of an Ecology-Approved Laboratory

Ecology maintains a list of accredited laboratories approved to perform analysis of PCBs as congeners (see: <https://fortress.wa.gov/ecy/laboratorysearch/>). It should be a goal for PCB analyses going forward that any congener work be conducted by one of the approved laboratories.

8.5.2 Recommend a Standard Column for Polychlorinated Biphenyl Congener Analyses

For total PCB analysis, a specific analytical column does not need to be recommended. However, as described in Section 7.3.1, each analytical column or column combination generates data with specific congeners and co-eluting congeners. In the interest of generating comparable congener data going forward, a goal would be to use the same analytical column for all work. However, recommending a particular column for LDW work could be an issue for public agencies that might not be able to limit which laboratories can do the work by specifying the analytical column in bid (King County reviewer comment). Further, prescribing which column should be used could have economic implications for the analytical laboratories conducting congener analyses (King County reviewer comment).

8.5.3 Use Naming Conventions

IUPAC numbers for the 209 PCB congeners (Table 3-1) should be used for all data reporting concerning congeners.

8.5.4 Develop Standardized Data Codes

Many of the terms used by laboratories are interchangeable, and several refer to the same thing, but some have significantly different meanings (Ecology 2015d).

Table 8-2 provides an example of standardized data codes (see the DRBC website; available online at: <http://www.state.nj.us/drbc/quality/toxics/pcbs/monitoring.html>). LDW-specific

data codes and descriptions should be developed in consultation with participating agencies and other interested parties that generate PCB congener data in the LDW.

Table 8-2. Data Qualifier Codes

Qualifier Flag	Description
J	The reported result is an estimate. The value is less than the minimum calibration level but greater than the estimated detection limit (EDL)
U	The analyte was not detected in the sample at the estimated detection limit (EDL)
E	Exceeds calibration range
D	Dilution data. Result was obtained from the analysis of a dilution
B	Analyte found in sample and associated blank
C	Co-eluting congener
Cxx	Co-elutes with the indicated congener, data is reported under the lowest IUPAC congener. 'Xx' denotes the IUPAC number with the lowest numerical designated congener.
NR	Analyte not reported because of problems in sample preparation or analysis
V	Surrogate recovery is not within method control limits
X	Results from reinjection/repeat/recolumn data
EMPC	Estimated maximum possible concentration. Indicates that a peak is detected but did not meet all met the method required criteria.

Source: Delaware River Basin Commission. Available online at: <http://www.state.nj.us/drbc/quality/toxics/pcbs/monitoring.html>.

8.5.5 Develop Blank Contamination Decision Rules

In concert with the development of standardized data codes, decision rules should be developed that prescribe how method blank contamination is handled with regard to data acceptability and reporting. Example decision rules from the DBRC and Spokane River Regional Toxics Task Force (draft rules) are provided in Section 7.3.2.

8.5.6 Other Quality Assurance/Quality Control Issues

As noted by a reviewer from Seattle Public Utilities, there are a number of quality assurance/QC issues that are specific to congener analysis that should be considered as more congener data are generated. For congeners analyzed for by Method 1668, there can be ion abundance outliers that lead to results being reported as an EMPC. These can be difficult to handle in data management as they represent an elevated detection limit that is different from the detection limit and reporting limit in the electronic data deliverable. There may also be lock-mass interferences that can bias results. Both of these cases are flagged by the laboratory, but the data user needs to know how to interpret them. For congeners analyzed by other methods (GC-ECD or GC/LRMS), co-eluting non-target congeners can lead to possible false positives.

8.5.7 Develop Standard Data Output Formats

One goal for collection of LDW PCB congener data is that data reports be standardized. As noted by a King County commenter, it may be difficult to get all laboratories to provide data in the same format. However, this should be a goal that is pursued to make data comparability more transparent and to improve both the ease and reliability of database uploading of new data. Also, it should be expected that some database work would be required. As part of this report effort, a Microsoft Access database containing LDW PCB congener data was created. This database was created by examining the data fields in the Ecology EIM database, the electronic data deliverables examples provided by the DRBC, and the various compiled LDW PCB data sets to establish relationships among and between the data types. Given that various agencies already have data collections/databases, generating a standardized format will require coordination among the various agencies.

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9.0 References

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Appendix A
Lower Duwamish Waterway Source Control: Bulk Atmospheric Deposition Study Polychlorinated Biphenyl Congener Profiles

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Figure A-1. PCB Congener Profile – Beacon Hill Station

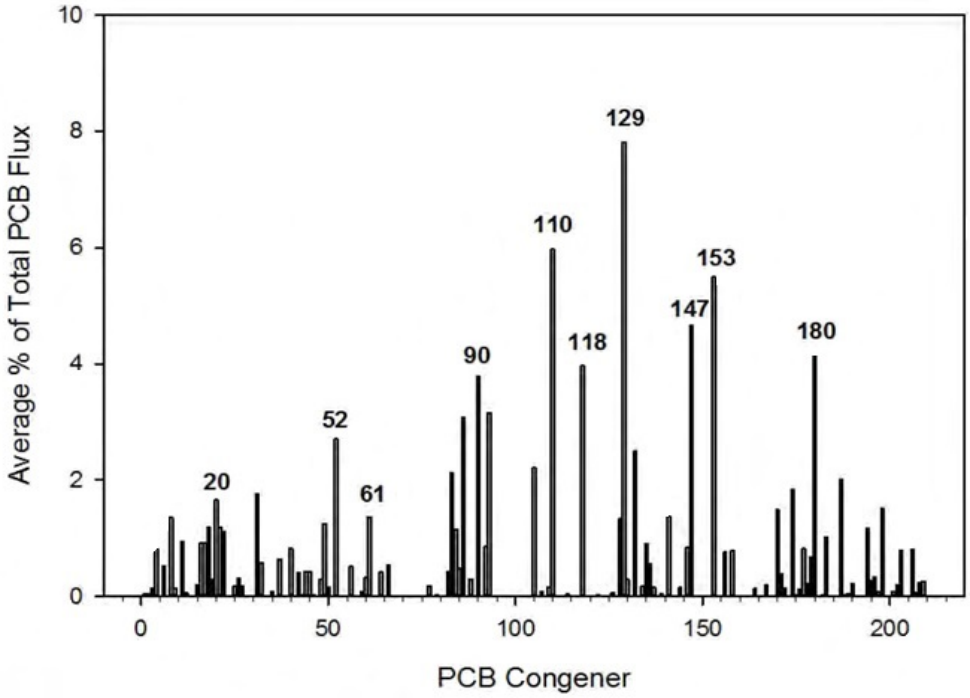


Figure A-2. PCB Congener Profile – Duwamish Station

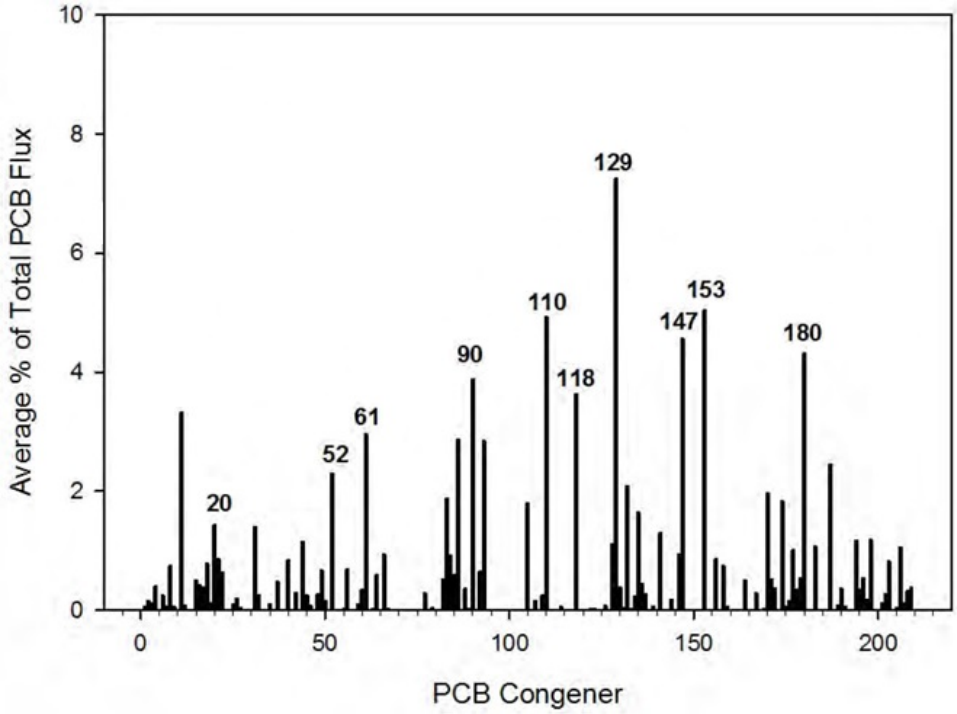


Figure A-3. PCB Congener Profile – Enumclaw Station

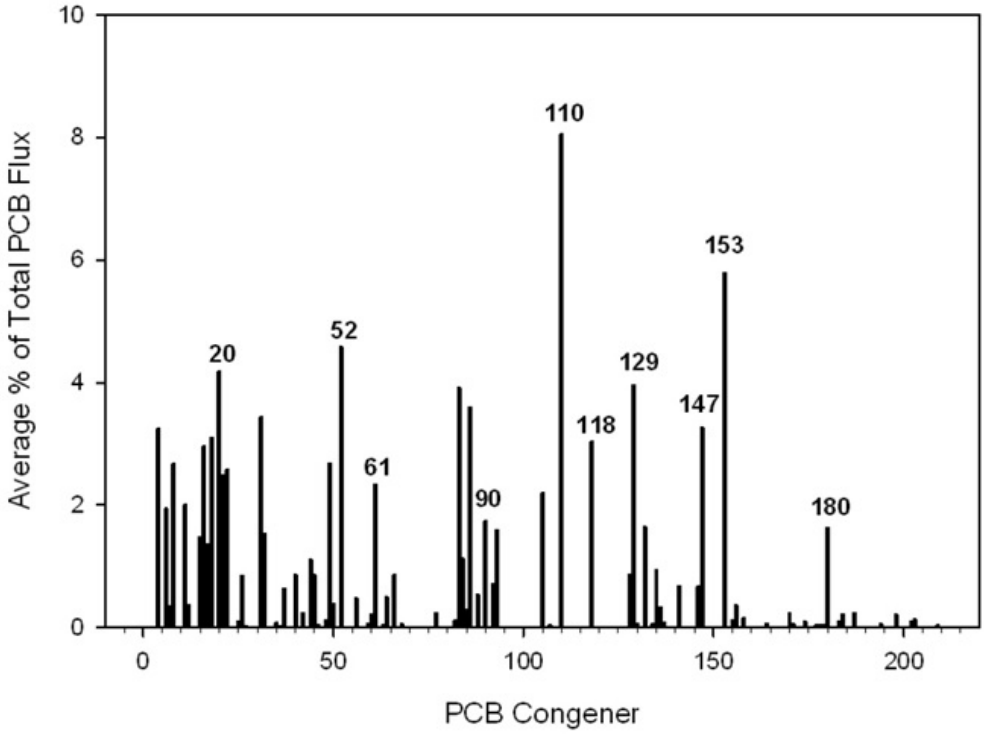


Figure A-4. PCB Congener Profile – Georgetown Station

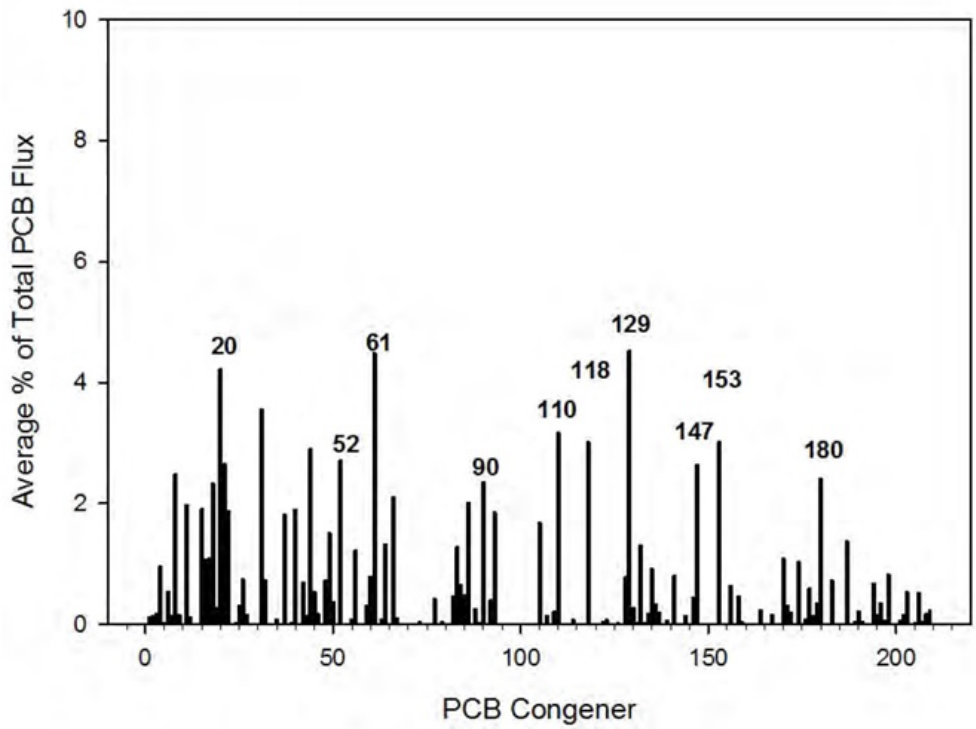


Figure A-5. PCB Congener Profile – Kent Senior Center Station

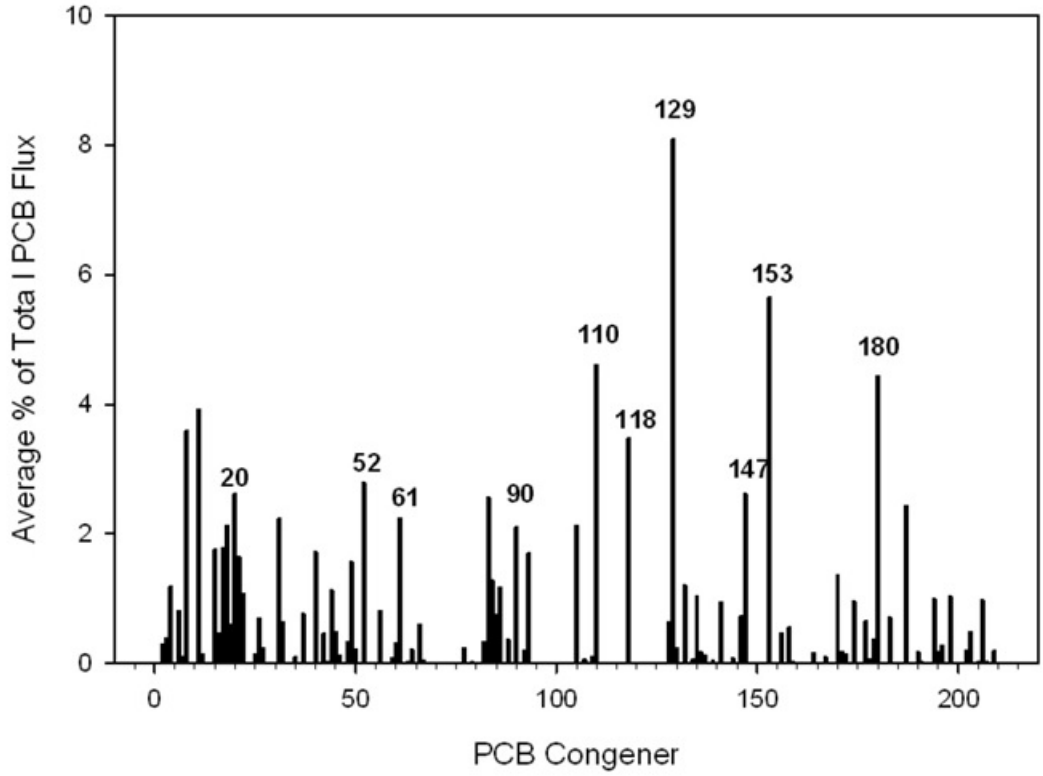


Figure A-6. PCB Congener Profile – Kent Station

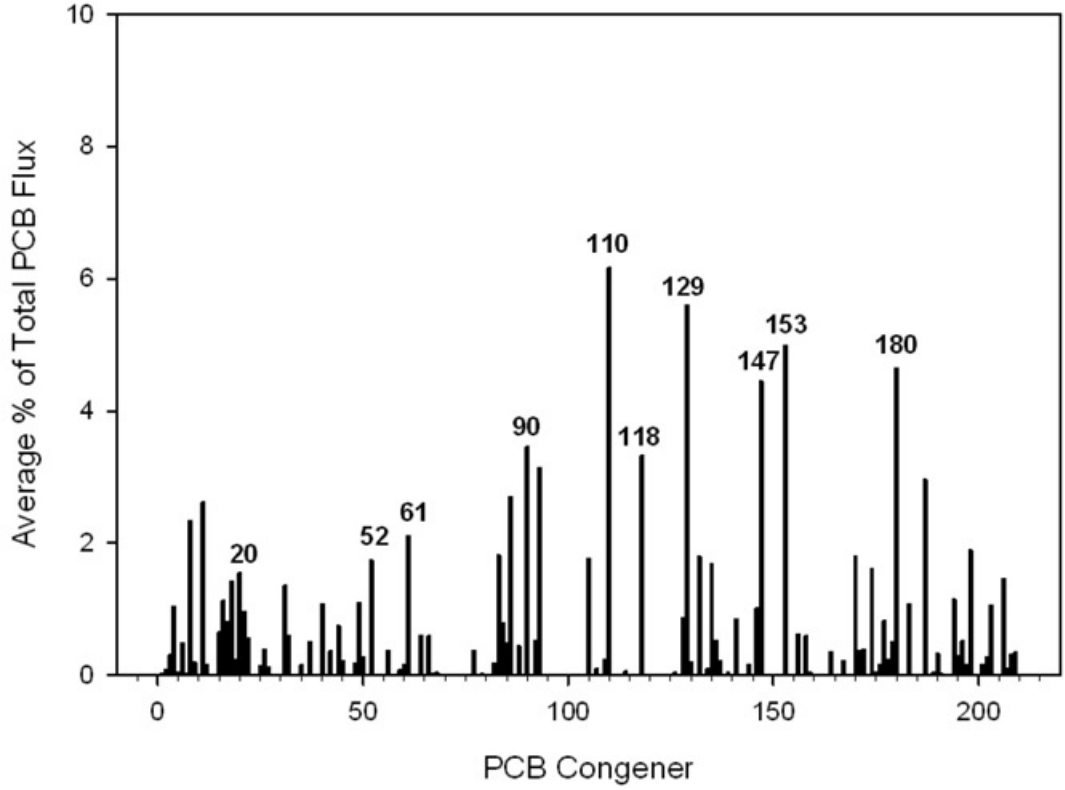
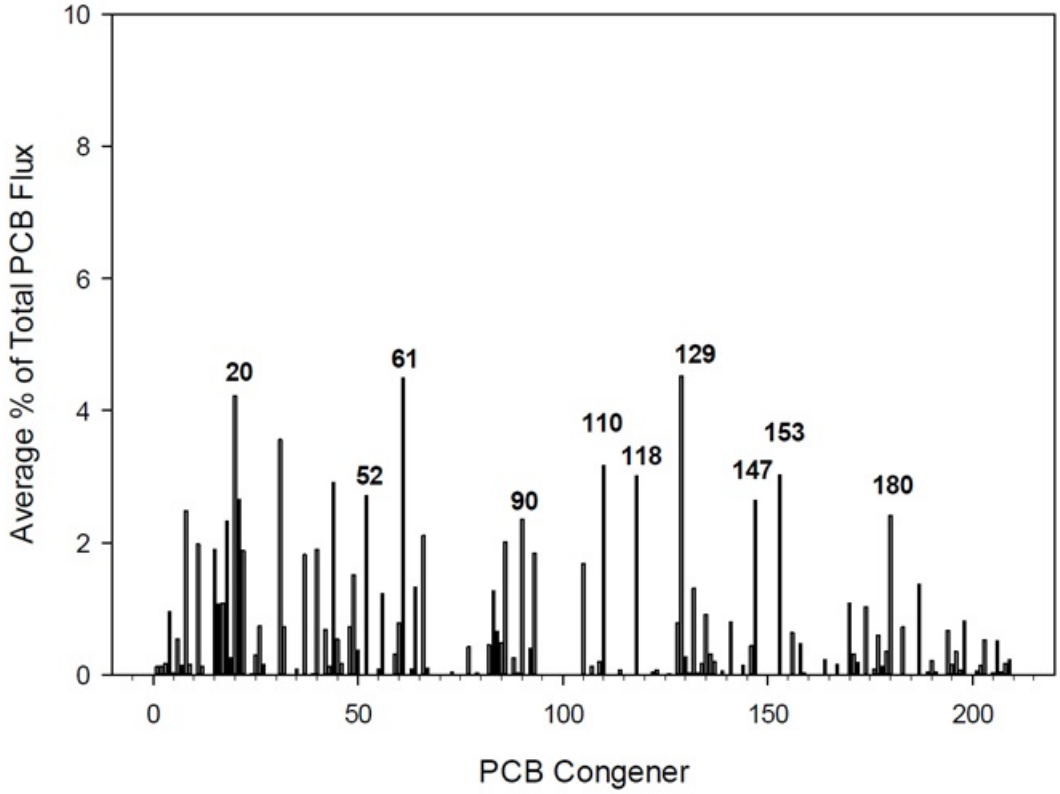


Figure A-7. PCB Congener Profile – South Park Station



Appendix B
List of Co-eluting Congeners in Lower Duwamish Waterway/Green River Data Set by Study Code

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Study Code (Environmental Medium)					
19 (tissue)	2 (soil/solids) 5 (sediment)	20 (SD water)	9 (tissue) 12 (sediment, SW, SS) 13 (sediment, SW, SS) 14 (SW) 15 (SW) 16 (air deposition) 17 (tissue) 18 (tissue) 23 (SW) 26 (sediment, tissue) 38 (SD water) 39 (air deposition)	6 (sediment, tissue) 27 (sediment)	21 (SD solids, SD water) 22 (SD solids, SD water)
					4/10
					5/8
					7/9
12/13	12/13	12/13	12/13	12/13	12/13
					16/32
18/30	18/30	18/30	18/30	18/30	
20/28	20/28	20/28	20/28	20/28	20/21/33
21/33	21/33	21/33	21/33	21/33	
					24/27
26/29	26/29	26/29	26/29	26/29	
40/41/71	40/41/71	40/71	40/41/71	40/71	
					41/64/71/72
					42/59
	43/73				43/49
44/47/65	44/47/65	44/47/65	44/47/65	44/47/65	
45/51	45/51		45/51		
					48/75
49/69	49/69	49/69	49/69	49/69	
50/53	50/53	50/53	50/53	50/53	
					52/69

Study Code (Environmental Medium)					
					56/60
59/62/75	59/62/75	59/62/75	59/62/75	59/62/75	
61/70/74/76	61/70/74/76	61/70/74/76	61/70/74/76	61/70/74/76	61/70
					66/76
83/99	83/99		83/99		
					84/92
85/116/117	85/116	85/116	85/116/117	85/116	85/116
86/87/97/109/119/125	86/87/97/109/119/125	86/87/97/109/119/125	86/87/97/108/119/125	86/87/97/109/119/125	
					87/117/125
88/91	88/91		88/91		88/91
90/101/113	90/101/113	90/101/113	90/101/113	90/101/113	90/101
93/95/98/100/102	93/100	93/100	93/95/98/100/102	93/100	
					95/98/102
	98/102				
					106/118
107/124			107/124	107/124	107/109
	108/124	108/124			108/112
110/115	110/115		110/115		
					111/115
128/166	128/166	128/166	128/166	128/166	128/162
129/138/160/163	129/138/163	129/138/163	129/138/160/163	129/138/163	
					132/161
					133/142
134/143			134/143		134/143
135/151/154	135/151	135/151	135/151/154	135/151	
					138/163/164
139/140	139/140	139/140	139/140	139/140	139/149
					146/165
147/149	147/149	147/149	147/149	147/149	
153/168	153/168	153/168	153/168	153/168	

Study Code (Environmental Medium)					
156/157	156/157	156/157	156/157	156/157	
					158/160
171/173	171/173	171/173	171/173	171/173	
180/193	180/193	180/193	180/193	180/193	
					182/187
183/185			183/185		
					196/203
197/200			197/200		
198/199	198/199	198/199	198/199	198/199	

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Appendix C
Delaware River Basin Commission Guidance

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Monitoring for PCBs

More Information

Link to PCB Information on U.S. EPA Region III's Web Site

Link to U.S. EPA's PCB Transformer Registration Database

Presentations from the 2013 NJWEA Conference Watershed Management Session - PCB Remediation Efforts

Introduction

The United States Environmental Protection Agency (U.S. EPA) Regions II and III established Stage 1 Total Maximum Daily Loads (TMDLs) for polychlorinated biphenyls (PCBs) for Zones 2 through 5 of the Delaware Estuary on December 15, 2003. The U.S. EPA has provided that the Stage 1 TMDLs will be replaced by more refined Stage 2 TMDLs.

At the request of U.S. EPA Regions II and III and the States of New Jersey, Delaware, and Pennsylvania, DRBC is in the process of developing the Stage 2 TMDLs. Point source discharges are one of the several PCB source categories being evaluated as part of this effort.

In order to better characterize loadings of PCBs to the estuary and to develop and calibrate the additional PCB homolog models, additional data must be collected.

Summary of Sampling and Analytical Requirements

The following information has been assembled to assist permittees in completing their PCB sampling requirements:

1. Analysis will be conducted utilizing DRBC project-specific modifications (pdf 125 KB; revised 2/3/05*) of U.S. EPA Method 1668, Revision A (pdf 688 KB)
2. Sample Labeling and Identification Protocols (pdf 50 KB)
3. Sample Size:
    1. Two 2-liter (nominal)** samples to be collected in PCB proofed amber glass jars***
    2. The entire volume of one 2-liter amber glass jar will be extracted for analysis. The second jar will be archived for possible future analysis.
4. Sample collection techniques (pdf 65 KB)
5. Analysis will conducted for 209 PCB congeners (pdf 25 KB) utilizing the following data qualifier flags (pdf 5 KB) and convention for reporting coeluting congeners (pdf 36 KB)
6. Sample extracts will be concentrated to 20 ul
7. Analysis will be conducted utilizing a SPB-octyl column
8. Calibration will consist of a minimum of five levels utilizing a 0.5 ng/ml standard for the low concentration calibration
9. Sample must meet method blank contamination decision rules (pdf 30 KB)
10. Sample must meet rinsate blank contamination decision rules (pdf 60 KB)
11. Results must be reported to the Estimated Detection Limit (EDL) (pdf 7 KB)
12. Results are to be reported utilizing a common Electronic Data Deliverable (EDD) (pdf 41 KB; revised 1/13/2009****) and hardcopy data deliverable protocols (pdf 104 KB; revised 2/18/2005*****)

** Storage temperature is now <6°C and preservation with sulfuric acid has been removed.*
*** In some instances, collection of precisely a 2-liter volume may not be possible. If the sample size falls below 2 liters, the Estimated Detection Limits (EDLs) will become elevated above project objectives. If the volume exceeds 2 liters, the additional volume poses difficulties in extracting the sample properly. The sample size collected can acceptably range from 1.8 liters to 2.1 liters, but should be as close as possible to 2 liters.*
**** Amber glass containers are specified in the U.S. EPA regulatory methods for organic contaminants, including PCBs.*
***** Changes made to: Chain_Custody_EDD file (Formatting Issues Addressed), Location_Table_EDD file (New Jersey locations not within the Delaware River Basin were added, as well as locations in the non-tidal Schuylkill River Basin, and For Chain_Custody and Location_Table EDDs previously submitted, there is no requirement to resubmit those files. Changes will be made by the DRBC to existing files.*
****** Change made to Section 1.1 F) 2.c. on page four.*

Data Glossary (pdf 40 KB)

Listing of Laboratories Known to Perform Low Level PCB Congener Analyses (pdf 96 KB; updated Feb. 2015)

Workshop Presentations and Related Resolutions

PCB Point Source Discharger Sampling Workshop: January 11 and 20, 2005

This workshop, sponsored by the Delaware Estuary TMDL Coalition, was held at the Camden County Municipal Utilities Authority (CCMUA) Auditorium Building in Camden, N.J. Below are the powerpoint presentations given:

- Introduction (pdf 144 KB)
- Review of Sampling Protocols (pdf 99 KB)
- Need for Additional Point Source Monitoring (pdf 134 KB)
- PCB Analysis by U.S. EPA Method 1668A, Including Project Analytical Quality Control Requirements (pdf 654 KB)

Related Resolutions

- DRBC Resolution Concerning the Collection of Additional Data to Further Characterize Point and Non-Point Source Discharges of Toxic Contaminants, including PCBs (Dec. 2003)
- DRBC Resolution Regarding Additional PCB Sampling (Apr. 2002)
- DRBC Resolution to Control Estuary Toxics (Jul. 2000)

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P.O. Box 7360, West Trenton, NJ 08628-0360
Phone (609)883-9500; Fax (609)883-9522
Thanks to NJ for hosting the DRBC website

Last Modified: 02/25/2015

DRBC – Reporting Rules for Coeluting Congeners

Reporting Rules for coeluting congeners

1. If a congener coelutes with another congener, qualify the result with CXXX (where XXX is the lowest numbered target in the coelution). No value will be entered into the concentration field for CXXX.
2. The CXXX designation will be identified in the data qualifier flag column.
3. If the congener is the lowest numbered congener in a coelution, then it will be identified with a C in the data qualifier flag column.

DRBC – List of PCB Congeners

23.0 Tables and Figures

Table 1. Names, International Union of Pure and Applied Chemistry (IUPAC) numbers, and CAS Registry numbers for native and labeled chlorinated biphenyl (CB) congeners determined by isotope dilution and internal standard HRGC/HRMS.

CB congener[1]	IUPAC number	CAS registry number	Labeled analog	IUPAC analog	CAS registry number
2-MoCB	1	2051-60-7	$^{13}C_{12}$-2-MoCB[2]	1L	234432-85-0
3-MoCB	2	2051-61-8			
4-MoCB	3	2051-62-9	$^{13}C_{12}$-4-MoCB[2]	3L	208263-77-8
2,2'-DiCB	4	13029-08-8	$^{13}C_{12}$-2,2'-DiCB[2]	4L	234432-86-1
2,3-DiCB	5	16605-91-7			
2,3'-DiCB	6	25569-80-6			
2,4-DiCB	7	33284-50-3			
2,4'-DiCB[3]	8	34883-43-7			
2,5-DiCB	9	34883-39-1	$^{13}C_{12}$-2,5-DiCB[4]	9L	250694-89-4
2,6-DiCB	10	33146-45-1			
3,3'-DiCB	11	2050-67-1			
3,4-DiCB	12	2974-92-7			
3,4'-DiCB	13	2974-90-5			
3,5-DiCB	14	34883-41-5			
4,4'-DiCB	15	2050-68-2	$^{13}C_{12}$-4,4'-DiCB[2]	15L	208263-67-6
2,2',3-TrCB	16	38444-78-9			
2,2',4-TrCB	17	37680-66-3			
2,2',5-TrCB[3]	18	37680-65-2			
2,2',6-TrCB	19	38444-73-4	$^{13}C_{12}$-2,2',6-TrCB[2]	19L	234432-87-2
2,3,3'-TrCB	20	38444-84-7			
2,3,4-TrCB	21	55702-46-0			
2,3,4'-TrCB	22	38444-85-8			
2,3,5-TrCB	23	55720-44-0			
2,3,6-TrCB	24	55702-45-9			
2,3',4-TrCB	25	55712-37-3			
2,3',5-TrCB	26	38444-81-4			
2,3',6-TrCB	27	38444-76-7			
2,4,4'-TrCB[3]	28	7012-37-5	$^{13}C_{12}$-2,4,4'-TriCB[5]	28L	208263-76-7
2,4,5-TrCB	29	15862-07-4			
2,4,6-TrCB	30	35693-92-6			
2,4',5-TrCB	31	16606-02-3			
2,4',6-TrCB	32	38444-77-8			
2',3,4-TrCB	33	38444-86-9			
2',3,5-TrCB	34	37680-68-5			
3,3',4-TrCB	35	37680-69-6			
3,3',5-TrCB	36	38444-87-0			
3,4,4'-TrCB	37	38444-90-5	$^{13}C_{12}$-3,4,4'-TrCB[2]	37L	208263-79-0
3,4,5-TrCB	38	53555-66-1			
3,4',5-TrCB	39	38444-88-1			
2,2',3,3'-TeCB	40	38444-93-8			

CB congener[1]	IUPAC number	CAS registry number	Labeled analog	IUPAC analog	CAS registry number
2,2',3,4-TeCB	41	52663-59-9			
2,2',3,4'-TeCB	42	36559-22-5			
2,2',3,5-TeCB	43	70362-46-8			
2,2',3,5'-TeCB[3]	44	41464-39-5			
2,2',3,6-TeCB	45	70362-45-7			
2,2',3,6'-TeCB	46	41464-47-5			
2,2',4,4'-TeCB	47	2437-79-8			
2,2',4,5-TeCB	48	70362-47-9			
2,2',4,5'-TeCB	49	41464-40-8			
2,2',4,6-TeCB	50	62796-65-0			
2,2',4,6'-TeCB	51	68194-04-7			
2,2',5,5'-TeCB[3]	52	35693-99-3	$^{13}C_{12}$-2,2',5,5'-TeCB[4]	52L	208263-80-3
2,2',5,6'-TeCB	53	41464-41-9			
2,2',6,6'-TeCB	54	15968-05-5	$^{13}C_{12}$-2,2',6,6'-TeCB[2]	54L	234432-88-3
2,3,3',4'-TeCB	55	74338-24-2			
2,3,3',4'-TeCB	56	41464-43-1			
2,3,3',5-TeCB	57	70424-67-8			
2,3,3',5'-TeCB	58	41464-49-7			
2,3,3',6-TeCB	59	74472-33-6			
2,3,4,4'-TeCB	60	33025-41-1			
2,3,4,5-TeCB	61	33284-53-6			
2,3,4,6-TeCB	62	54230-22-7			
2,3,4',5-TeCB	63	74472-34-7			
2,3,4',6-TeCB	64	52663-58-8			
2,3,5,6-TeCB	65	33284-54-7			
2,3',4,4'-TeCB[3]	66	32598-10-0			
2,3',4,5-TeCB	67	73575-53-8			
2,3',4,5'-TeCB	68	73575-52-7			
2,3',4,6-TeCB	69	60233-24-1			
2,3',4',5-TeCB	70	32598-11-1			
2,3',4',6-TeCB	71	41464-46-4			
2,3',5,5'-TeCB	72	41464-42-0			
2,3',5',6-TeCB	73	74338-23-1			
2,4,4',5-TeCB	74	32690-93-0			
2,4,4',6-TeCB	75	32598-12-2			
2',3,4,5-TeCB	76	70362-48-0			
3,3',4,4'-TeCB[3,6]	77	32598-13-3	$^{13}C_{12}$-3,3',4,4'-TeCB[2,7]	77L	105600-23-5
3,3',4,5-TeCB	78	70362-49-1			
3,3',4,5'-TeCB	79	41464-48-6			
3,3',5,5'-TeCB	80	33284-52-5			
3,4,4',5-TeCB[6]	81	70362-50-4	$^{13}C_{12}$-3,4,4',5-TeCB[7]	81L	208461-24-9
2,2',3,3',4-PeCB	82	52663-62-4			
2,2',3,3',5-PeCB	83	60145-20-2			
2,2',3,3',6-PeCB	84	52663-60-2			
2,2',3,4,4'-PeCB	85	65510-45-4			
2,2',3,4,5-PeCB	86	55312-69-1			

CB congener[1]	IUPAC number	CAS registry number	Labeled analog	IUPAC analog	CAS registry number
2,2',3,4,5'-PeCB	87	38380-02-8			
2,2',3,4,6-PeCB	88	55215-17-3			
2,2',3,4,6'-PeCB	89	73575-57-2			
2,2',3,4',5-PeCB	90	68194-07-0			
2,2',3,4',6-PeCB	91	68194-05-8			
2,2',3,5,5'-PeCB	92	52663-61-3			
2,2',3,5,6-PeCB	93	73575-56-1			
2,2',3,5,6'-PeCB	94	73575-55-0			
2,2',3,5',6-PeCB	95	38379-99-6			
2,2',3,6,6'-PeCB	96	73575-54-9			
2,2',3',4,5-PeCB	97	41464-51-1			
2,2',3',4,6-PeCB	98	60233-25-2			
2,2',4,4',5-PeCB	99	38380-01-7			
2,2',4,4',6-PeCB	100	39485-83-1			
2,2',4,5,5'-PeCB[3]	101	37680-73-2	$^{13}C_{12}$-2,2',4,5,5'-PeCB[4]	101L	104130-39-4
2,2',4,5,6'-PeCB	102	68194-06-9			
2,2',4,5,'6-PeCB	103	60145-21-3			
2,2',4,6,6'-PeCB	104	56558-16-8	$^{13}C_{12}$-2,2',4,6,6'-PeCB[2]	104L	234432-89-4
2,3,3',4,4'-PeCB[3,6]	105	32598-14-4	$^{13}C_{12}$-2,3,3',4,4'-PeCB[7]	105L	208263-62-1
2,3,3',4,5-PeCB	106	70424-69-0			
2,3,3',4',5-PeCB	107	70424-68-9			
2,3,3',4,5'-PeCB	108	70362-41-3			
2,3,3',4,6-PeCB	109	74472-35-8			
2,3,3',4',6-PeCB	110	38380-03-9			
2,3,3',5,5'-PeCB	111	39635-32-0	$^{13}C_{12}$-2,3,3',5,5'-PeCB[5]	111 L	235416-29-2
2,3,3',5,6-PeCB	112	74472-36-9			
2,3,3',5',6-PeCB	113	68194-10-5			
2,3,4,4',5-PeCB[6]	114	74472-37-0	$^{13}C_{12}$-2,3,4,4',5-PeCB[7]	114 L	208263-63-2
2,3,4,4',6-PeCB	115	74472-38-1			
2,3,4,5,6-PeCB	116	18259-05-7			
2,3,4',5,6-PeCB	117	68194-11-6			
2,3',4,4',5-PeCB[3,6]	118	31508-00-6	$^{13}C_{12}$-2,3',4,4',5-PeCB[7]	118 L	104130-40-7
2,3',4,4',6-PeCB	119	56558-17-9			
2,3',4,5,5'-PeCB	120	68194-12-7			
2,3',4,5,'6-PeCB	121	56558-18-0			
2',3,3',4,5-PeCB	122	76842-07-4			
2',3,4,4',5-PeCB[6]	123	65510-44-3	$^{13}C_{12}$-2',3,4,4',5-PeCB[7]	123L	208263-64-3
2',3,4,5,5'-PeCB	124	70424-70-3			
2',3,4,5,6'-PeCB	125	74472-39-2			
3,3',4,4',5-PeCB[3,6]	126	57465-28-8	$^{13}C_{12}$-3,3',4,4',5-PeCB[2,7]	126L	208263-65-4
3,3',4,5,5'-PeCB	127	39635-33-1			
2,2',3,3',4,4'-HxCB[3]	128	38380-07-3			
2,2',3,3',4,5-HxCB	129	55215-18-4			
2,2',3,3',4,5'-HxCB	130	52663-66-8			
2,2',3,3',4,6-HxCB	131	61798-70-7			
2,2',3,3',4,6'-HxCB	132	38380-05-1			

CB congener[1]	IUPAC number	CAS registry number	Labeled analog	IUPAC analog	CAS registry number
2,2',3,3',5,5'-HxCB	133	35694-04-3			
2,2',3,3',5,6-HxCB	134	52704-70-8			
2,2',3,3',5,6'-HxCB	135	52744-13-5			
2,2',3,3',6,6'-HxCB	136	38411-22-2			
2,2',3,4,4',5-HxCB	137	35694-06-5			
2,2',3,4,4',5'-HxCB[3]	138	35065-28-2	$^{13}C_{12}$-2,2',3,4,4',5'-HxCB[4]	138L	208263-66-5
2,2',3,4,4',6-HxCB	139	56030-56-9			
2,2',3,4,4',6'-HxCB	140	59291-64-4			
2,2',3,4,5,5'-HxCB	141	52712-04-6			
2,2',3,4,5,6-HxCB	142	41411-61-4			
2,2',3,4,5,6'-HxCB	143	68194-15-0			
2,2',3,4,5',6-HxCB	144	68194-14-9			
2,2',3,4,6,6'-HxCB	145	74472-40-5			
2,2',3,4',5,5'-HxCB	146	51908-16-8			
2,2',3,4',5,6-HxCB	147	68194-13-8			
2,2',3,4',5,6'-HxCB	148	74472-41-6			
2,2',3,4',5',6-HxCB	149	38380-04-0			
2,2',3,4',6,6'-HxCB	150	68194-08-1			
2,2',3,5,5',6-HxCB	151	52663-63-5			
2,2',3,5,6,6'-HxCB	152	68194-09-2			
2,2',4,4',5,5'-HxCB[3]	153	35065-27-1			
2,2',4,4',5',6-HxCB	154	60145-22-4			
2,2',4,4',6,6'-HxCB	155	33979-03-2	$^{13}C_{12}$-2,2',4,4',6,6'-HxCB[2]	155L	234432-90-7
2,3,3',4,4',5-HxCB[6]	156	38380-08-4	$^{13}C_{12}$-2,3,3',4,4',5-HxCB[7]	156L	208263-68-7
2,3,3',4,4',5'-HxCB[6]	157	69782-90-7	$^{13}C_{12}$-2,3,3',4,4',5'-HxCB[7]	157L	235416-30-5
2,3,3',4,4',6-HxCB	158	74472-42-7			
2,3,3',4,5,5'-HxCB	159	39635-35-3			
2,3,3',4,5,6-HxCB	160	41411-62-5			
2,3,3',4,5',6-HxCB	161	74472-43-8			
2,3,3',4',5,5'-HxCB	162	39635-34-2			
2,3,3',4',5,6-HxCB	163	74472-44-9			
2,3,3',4',5',6-HxCB	164	74472-45-0			
2,3,3',5,5',6-HxCB	165	74472-46-1			
2,3,4,4',5,6-HxCB	166	41411-63-6			
2,3',4,4',5,5'-HxCB[6]	167	52663-72-6	$^{13}C_{12}$-2,3',4,4',5,5'-HxCB[7]	167L	208263-69-8
2,3',4,4',5',6-HxCB	168	59291-65-5			
3,3',4,4',5,5'-HxCB[3,6]	169	32774-16-6	$^{13}C_{12}$-3,3',4,4',5,5'-HxCB[2,7]	169L	208263-70-1
2,2',3,3',4,4',5-HpCB[3]	170	35065-30-6			
2,2'3,3',4,4',6-HpCB	171	52663-71-5			
2,2',3,3',4,5,5'-HpCB	172	52663-74-8			
2,2',3,3',4,5,6-HpCB	173	68194-16-1			
2,2',3,3',4,5,6'-HpCB	174	38411-25-5			
2,2',3,3',4,5',6-HpCB	175	40186-70-7			
2,2',3,3',4,6,6'-HpCB	176	52663-65-7			
2,2',3,3',4',5,6-HpCB	177	52663-70-4			
2,2',3,3',5,5',6-HpCB	178	52663-67-9	$^{13}C_{12}$-2,2',3,3',5,5',6-HpCB[5]	178L	232919-67-4

CB congener[1]	IUPAC number	CAS registry number	Labeled analog	IUPAC analog	CAS registry number
2,2',3,3',5,6,6'-HpCB	179	52663-64-6			
2,2',3,4,4',5,5'-HpCB[3]	180	35065-29-3			
2,2',3,4,4',5,6-HpCB	181	74472-47-2			
2,2',3,4,4',5,6'-HpCB	182	60145-23-5			
2,2',3,4,4',5',6-HpCB	183	52663-69-1			
2,2',3,4,4',6,6'-HpCB	184	74472-48-3			
2,2',3,4,5,5',6-HpCB	185	52712-05-7			
2,2',3,4,5,6,6'-HpCB	186	74472-49-4			
2,2',3,4',5,5',6-HpCB[3]	187	52663-68-0			
2,2',3,4',5,6,6'-HpCB	188	74487-85-7	$^{13}C_{12}$-2,2',3,4',5,6,6'-HpCB[2]	188L	234432-91-8
2,3,3',4,4',5,5'-HpCB[6]	189	39635-31-9	$^{13}C_{12}$-2,3,3',4,4',5,5'-HpCB[2,7]	189L	208263-73-4
2,3,3',4,4',5,6-HpCB	190	41411-64-7			
2,3,3',4,4',5',6-HpCB	191	74472-50-7			
2,3,3',4,5,5',6-HpCB	192	74472-51-8			
2,3,3',4',5,5',6-HpCB	193	69782-91-8			
2,2',3,3',4,4',5,5'-OcCB	194	35694-08-7	$^{13}C_{12}$-2,2',3,3',4,4',5,5'-OcCB[4]	194L	208263-74-5
2,2',3,3',4,4',5,6-OcCB[3]	195	52663-78-2			
2,2',3,3',4,4',5,6'-OcCB	196	42740-50-1			
2,2',3,3',4,4',6,6'-OcCB	197	33091-17-7			
2,2',3,3',4,5,5',6-OcCB	198	68194-17-2			
2,2',3,3',4,5,5',6'-OcCB	199	52663-75-9			
2,2',3,3',4,5,6,6'-OcCB	200	52663-73-7			
2,2',3,3',4,5',6,6'-OcCB	201	40186-71-8			
2,2',3,3',5,5',6,6'-OcCB	202	2136-99-4	$^{13}C_{12}$-2,2',3,3',5,5',6,6'-OcCB[2]	202L	105600-26-8
2,2',3,4,4',5,5',6-OcCB	203	52663-76-0			
2,2',3,4,4',5,6,6'-OcCB	204	74472-52-9			
2,3,3',4,4',5,5',6-OcCB	205	74472-53-0	$^{13}C_{12}$-2,3,3',4,4',5,5',6-OcCB[2]	205L	234446-64-1
2,2',3,3',4,4',5,5',6-NoCB[3]	206	40186-72-9	$^{13}C_{12}$-2,2',3,3',4,4',5,5',6-NoCB[2]	206L	208263-75-6
2,2',3,3',4,4',5,6,6'-NoCB	207	52663-79-3			
2,2',3,3',4,5,5',6,6'-NoCB	208	52663-77-1	$^{13}C_{12}$-2,2',3,3',4,5,5',6,6'-NoCB[2]	208L	234432-92-9
DeCB[3]	209	2051-24-3	$^{13}C_{12}$-DeCB[2]	209L	105600-27-9

1. Abbreviations for chlorination levels
   - MoCB = monochlorobiphenyl
   - DiCB = dichlorobiphenyl
   - TrCB = trichlorobiphenyl
   - TeCB = tetrachlorbiphenyl
   - PeCB = pentachlorobiphenyl
   - HxCB = hexachlorobiphenyl
   - HpCB = heptachlorobiphenyl
   - OcCB = octachlorobiphenyl
   - NoCB = nonachlorobiphenyl
   - DeCB = decachlorobiphenyl
2. Labeled level of chlorination (LOC) window-defining congener
3. National Oceanic and Atmospheric Administration (NOAA) congener of interest
4. Labeled injection internal standard
5. Labeled clean-up standard

6. World Health Organization (WHO) toxic congener
7. Labeled analog of WHO toxic congener

DRBC – Data Glossary

Data Glossary

The purpose of this data glossary is to provide a reference for terms used in the analytical analysis of polychlorinated biphenyl (PCB) congeners, utilizing high resolution gas chromatography/high resolution mass spectrometry/ (HRGC/HRMS) methods. The definitions and equations provided are specific to HRGC/HRMS methods used in the analysis of chlorinated biphenyl congeners.

Acceptance criteria - specific limits placed on characteristics of an item, process, or service defined in requirements documents.

Analyte - Any of 209 chlorinated biphenyl (CB) congeners.

Bias - the systematic or persistent distortion of a measurement process that causes errors in one direction (i.e., the expected sample measurement is different from the sample's true value).

Calibration standard (CAL) - A solution prepared from a secondary standard and/or stock solutions and used to calibrate the response of the HRGC/HRMS instrument.

Calibration verification standard (VER) - The mid-point calibration standard (CS-3) that is used to verify calibration.

CB—chlorinated biphenyl congener. One of the 209 individual chlorinated biphenyl congeners determined using Method 1668A.

CAS # (CASRN)---- Chemical Abstracts Service Registry Number. CAS Registry Numbers are unique identifiers for chemical compounds.

Congener Number---A numbering system from 1-209, which uniquely identifies each of the 209 chlorinated biphenyl congeners. This numbering system was formerly referred to as the BZ and IUPAC number and remains identical to the numbering system published by Ballschmiter et al., 1992 (BZ #'s).

Data quality assessment (DQA) - a statistical and scientific evaluation of the data set to determine the validity and performance of the data collection design and statistical test, and to determine the adequacy of the data set for its intended use.

Data quality objectives (DQOs) - qualitative and quantitative statements that clarify study objectives, define the appropriate type of data, and specify tolerable levels of potential decision errors that will be used as the basis for establishing the quality and quantity of data needed to support decisions.

Data usability - the process of ensuring or determining whether the quality of the data produced meets the intended use of the data.

Data validation – an analyte- and sample-specific process that extends the evaluation of data beyond method, procedural, or contractual compliance (i.e., data verification) to determine the analytical quality of a specific data set.

Data validation qualifier – code applied to the data by a data validator to indicate a verifiable or potential data deficiency or bias.

Data verification – the process of evaluating the completeness, correctness, and conformance/compliance of a specific data set against the method, procedural, or contractual requirements.

Equipment Blank or Rinsate Blank - A blank consisting of analyte-free media which has been used to rinse the sampling equipment. It is collected after completion of equipment decontamination and prior to sampling. This blank is useful in documenting adequate decontamination of sampling equipment and determining the impact of any contamination that may be present on the associated investigation samples.

Estimated detection limit (EDL) — The sample specific estimated detection limit (EDL) is the concentration of a given analyte required to produce a signal with a peak height of at least 2.5 times the background signal level. This concentration is determined by measuring the noise height of the two quantitation ions for a given congener at the region of the SICP where the congener is expected to elute, converting this height into area based on the associated internal standard area, and taking the internal standard concentration, internal standard area, initial calibration average RRF, minimum signal-to-noise factor, and sample weight/volume into account.

Estimated minimum level (EML)—The lowest concentration at which a CB can be measured reliably with common laboratory interferences present. EMLs for Method 1668A are provided in Table 2 of the Method. EMLs should be routinely achievable by laboratories running the method.

False negative or false acceptance decision error - the error that occurs when a decision maker accepts a result as true when it is actually false. Also referred to as a Type II error.

False positive or false rejection decision error - the error that occurs when a decision maker rejects a result when it actually is true. Also referred to as a Type I error.

Field blank—An aliquot of reagent water or other reference matrix that is placed in a sample container in the laboratory or the field, and treated as a sample in the following respects, including exposure to sampling site conditions, storage, preservation, and all analytical procedures. However, a field blank is not used to rinse the sampling equipment. The purpose of the field blank is to determine if the field or sample transporting procedures and environments have potentially contaminated the associated investigation samples.

Field Replicates - Independent samples that are collected as close as possible to the same point in space and time. They are two separate samples taken from the same source, stored in separate containers, and analyzed independently. These replicates are useful in documenting the overall precision of the sampling and analytical process. This definition is presented to distinguish a field replicate from a field duplicate. A field duplicate is a split of a well mixed homogenized sample.

High Resolution Gas Chromatography (HRGC) - A gas chromatograph is an instrument which is used to separate the components of a mixture. A high resolution GC provides the capability of separating similar substances in complex mixtures.

High Resolution Mass Spectrometry (HRMS) - A mass spectrometer is an instrument that measures the masses of individual molecules or molecular fragments that have been converted into ions, i.e., molecules or fragments that have been electrically charged. Mass spectrometers use the difference in mass-to-charge ratio (m/z) of ionized molecules or fragments to separate them from each other.

Internal standard—a labeled compound used as a reference for quantitation of other labeled compounds and for quantitation of native CB congeners other than the congener of which it is a labeled analog. See Internal standard quantitation.

Internal standard quantitation—A means of determining the concentration of (1) a naturally occurring (native) compound by reference to a compound other than its labeled analog and (2) a labeled compound by reference to another labeled compound.

IPR—Initial precision and recovery; four aliquots of a reference matrix spiked with the analytes of interest and labeled compounds and analyzed to establish the ability of the laboratory to generate acceptable precision and recovery. An IPR is performed prior to the first time this Method is used and any time the Method or instrumentation is modified.

Isotope Dilution - An analytical technique where a compound is determined in reference to the same compound in which one or more atoms has been isotopically enriched. This technique results in extremely accurate identification and quantitation. It also allows for determination of matrix effects on a sample specific basis.

Isotope dilution quantitation—A means of determining a naturally occurring (native) compound by reference to the same compound in which one or more atoms has been isotopically enriched. In Method 1668A, all 12 carbon atoms in the biphenyl molecule are enriched with carbon-13 to produce 13C12-labeled analogs of the chlorinated biphenyls. The 13C12-labeled CBs are spiked into each sample and allow identification and correction of the concentration of the native compounds in the analytical process.

Laboratory control sample (LCS)—See Ongoing precision and recovery standard (OPR) - A known matrix spiked with compound(s) representative of the target analytes. This is used to document laboratory performance

Laboratory blank—See Method blank

Laboratory reagent blank—See Method blank

Matrix Duplicate - An intralaboratory split sample that is used to document the precision of a method in a given sample matrix.

Matrix Spike - An aliquot of sample spiked with a known concentration of target analyte(s). The spiking occurs prior to sample preparation and analysis. A matrix spike is used to document the bias of a method in a given sample matrix.

Matrix Spike Duplicate - Intralaboratory split samples spiked with identical concentrations of target analyte(s). The spiking occurs prior to sample preparation and analysis. They are used to document the precision and bias of a method in a given sample matrix.

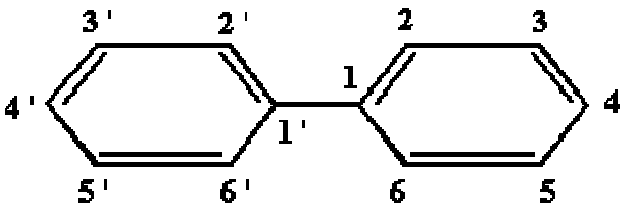
Method blank—an aliquot of reagent water that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with samples. The Method blank is used to determine if analytes or interferences are present in the laboratory environment, the reagents, or the apparatus.

Minimum level of quantitation (ML)—The level at which the entire analytical system must give a recognizable signal and acceptable calibration point for the analyte. It is equivalent to the concentration of the lowest calibration standard, assuming that all Method-specified sample weights, volumes, and cleanup procedures have been employed. According to Method 1668A, laboratories may establish MLs lower than EMLs: MLs may be established as low as the lowest calibration point provided that the concentration of the congener in a minimum of 10 blanks for a sample medium (e.g., water, soil, sludge, tissue) is significantly below the EML. Significant means that the ML for the congener is no less than the average (mean) plus 2 standard deviations above the level in the minimum of 10 blanks. The blanks must be analyzed during the same period that the sample is analyzed, ideally over an approximately 1-month period.

Ongoing Precision and Recovery Standard (OPR) - A method blank spiked with known quantities of analytes. The OPR is analyzed exactly like a sample. Its purpose is to assure that the results produced by the laboratory remain within the limits specified in the method for precision and recovery.

Polychlorinated Biphenyl (PCB) - PCB (or PCBs) is a category, or family, of chemical compounds formed by the addition of chlorine (Cl) to biphenyl ($C_{12}H_{10}$), which is a dual-ring structure comprising two 6-carbon benzene rings linked by a single carbon-carbon bond. The nature of an "aromatic" (benzene) ring allows a single attachment to each

carbon. This means that there are 10 possible positions for chlorine substitution (replacing the hydrogens in the original biphenyl).



A generalized PCB structure showing bonding locations

PCB Congener - Any single, unique, chemical compound in the PCB category is called a "Congener". The name of a congener specifies the total number of chlorine substituents and the position of each chlorine. For example: 4,4'-Dichlorobiphenyl is a congener comprising the biphenyl structure with two chlorine substituents, one on each of the two carbons at the "4" (also called "para") positions of the two rings. There are 209 possible unique PCB congeners.

PCB Homolog - "Homologs" are subcategories of PCBs, representing all congeners having an equal numbers of chlorine substituents. For example, the "Tetrachlorobiphenyls" (or "Tetra-PCBs" or "Tetra-CBs" or just "Tetras") are all PCB congeners with exactly 4 chlorine substituents in any arrangement. The number of congeners in each homolog group are given in the following table:

PCB Homologs			
Homolog	**Abbreviation**	**Cl Substituents**	**PCB Congeners**
Monochlorobiphenyl	MoCB	1	3
Dichlorobiphenyl	DiCB	2	12
Trichlorobiphenyl	TrCB	3	24
Tetrachlorobiphenyl	TeCB	4	42
Pentachlorobiphenyl	PeCB	5	46
Hexachlorobiphenyl	HxCB	6	42
Heptachlorobiphenyl	HpCB	7	24
Octachlorobiphenyl	OcCB	8	12
Nonachlorobiphenyl	NoCB	9	3
Decachlorobiphenyl	DeCB	10	1

PCB Mixture - With few exceptions, PCB was manufactured as a complex mixture of congeners, through progressive chlorination of batches of biphenyl until a certain target percentage of chlorine by weight was achieved. Commercial mixtures with higher percentages of chlorine contained higher proportions of the more heavily chlorinated congeners. While PCB was manufactured and sold under many names, the most common

were the "Aroclor" series (the Monsanto trade name), in many of which a numerical identifier included the percentage of chlorine (e.g., "Aroclor 1254", with 54 percent chlorine).

Preparation blank—See Method blank

Quality assurance (QA) - an integrated system of management activities involving planning, implementation, documentation, assessment, reporting, and quality improvement to ensure that a process, item, or service is of the type and quality needed and expected by the client.

Quality assurance project plan - a formal document describing in comprehensive detail the necessary QA, QC, and other technical activities that must be implemented to ensure that the results of the work performed will satisfy the stated performance criteria.

Quality control (QC) - the overall system of technical activities that measures the attributes and performance of a process, item, or service against defined standards to verify that they meet the stated requirements established by the customer; operational techniques and activities that are used to fulfill requirements for quality.

Quality control check sample (QCS)—A sample containing all or a subset of the analytes at known concentrations. The QCS is obtained from a source external to the laboratory or is prepared from a source of standards different from the source of calibration standards. It is used to check laboratory performance with test materials prepared external to the normal preparation process.

Reagent water—water demonstrated to be free from the analytes of interest and potentially interfering substances at the method detection limit for the analyte.

Relative standard deviation (RSD)—The standard deviation times 100 divided by the mean. Also termed "coefficient of variation."

SPE—Solid-phase extraction; an extraction technique in which an analyte is extracted from an aqueous sample by passage over or through a material capable of reversibly adsorbing the analyte. Also termed liquid-solid extraction.

Signal-to-noise ratio (S/N)—The height of the signal as measured from the mean (average) of the noise to the peak maximum divided by the width of the noise.

Trip Blank - A sample of analyte-free reagent water or other media placed in a sample container in the laboratory and taken from the laboratory to the sampling site and returned to the laboratory unopened. The trip blank is stored and preserved in the same manner as samples and undergoes all sample analytical procedures. A trip blank is used to document potential contamination attributable to shipping and field handling procedures. This type of blank is often used for volatile organics samples.

Unique GC resolution or uniquely resolved—Two adjacent chromatographic peaks in which the height of the valley is less than 40 percent of the height of the shorter peak. This is an indication of the ability of the instrument to separate two or more similar compounds.

VER—See Calibration verification.

Validation - confirmation by examination and provision of objective evidence that the particular requirements for a specific intended use are fulfilled. In design and development, validation concerns the process of examining a product or result to determine conformance to user needs.

Verification - confirmation by examination and provision of objective evidence that specified requirements have been fulfilled. In design and development, verification concerns the process of examining a result of a given activity to determine conformance to the stated requirements for that activity.

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DRBC – Description of Data Qualifiers

Description of Data Qualifiers

Qualifier Flag	Description
J	The reported result is an estimate. The value is less than the minimum calibration level but greater than the estimated detection limit (EDL)
U	The analyte was not detected in the sample at the estimated detection limit (EDL)
E	Exceeds calibration range.
D	Dilution data. Result was obtained from the analysis of a dilution
B	Analyte found in sample and associated blank
C	Co-eluting congener
Cxx	Co-elutes with the indicated congener, data is reported under the lowest IUPAC congener. ‘Xx’ denotes the IUPAC number with the lowest numerical designated congener.
NR	Analyte not reported because of problems in sample preparation or analysis
V	Surrogate recovery is not within method control limits
X	Results from reinjection/repeat/recolumn data
EMPC	Estimated maximum possible concentration. Indicates that a peak is detected but did not meet all met the method required criteria.

DRBC – Electronic Data Deliverables

Electronic Data Deliverables

Location_Table_EDD.xls (revised 1/13/09)
Chain_Custody_EDD.xls (revised 1/13/09)
Analytical_Results_EDD.xls (revised 6/20/05; no revisions were made 1/13/09)

Three Electronic Data Deliverable (EDD) files are required as part of the project-specific requirements for DRBC Stage 2 PCB TMDL development. The EDDs address aqueous sample collection and analysis associated with point source discharge samples. These requirements must be met to ensure consistency among participants in the project and cannot be modified.

The three files address the following data deliverables: location, chain of custody, and analytical information. Each file consists of an Excel spreadsheet and includes data fields with embedded formatting rules and tabs providing formatting definitions for each field.

1. Data for the Location_Table and Chain_Custody EDDs must be submitted in the Excel format specified in the file.
2. Laboratories must deliver analytical results which adhere to the formatting guidelines specified in the Analytical_Results EDD file as Quote Comma Separated Value (*.csv) files.

The Location_Table Excel file must be completed and returned to the DRBC before sampling begins. The Chain_Custody file must be completed and submitted to the DRBC within 14 days of a sampling event.

The properly formatted submissions should be mailed to:

Modeling, Monitoring, and Assessment Branch
Delaware River Basin Commission
25 State Police Drive, P.O. Box 7360
West Trenton, NJ 08628-0360

Questions or concerns about the EDD requirements should be discussed with Greg Cavallo at (609) 883-9500 ext. 270 or gregory.cavallo@drbc.state.nj.us.

Electronic Data Deliverables for Locational Information

DATA FIELDS	FORMAT	DEFINITIONS
Location ID	Alpha-Numeric	State abbreviation NPDES # and outfall (example PA0123456-001)
Sampling Location Name	Text	Location name, Trib name, Air location, Ambient (Estuary) by RM, sediment. Ex: General Motors Repauno facility outfall 001
Owner	Text	Legal entity responsible for facility, or State and Agency
Street Address	Text	Street address (no P.O. Box addresses)
City	Text	City name
State	Text	(LOOKUP TABLE) DE, PA, NJ
Zip Code	Special Zip Code	5 digit zip code
Municipality	Text	Municipality in which sampling location exists
Point of Contact	Text	Facility contact name and title
Contact Phone Number	Numeric	Phone number (example (609) 883-9500 ext 270)
Alternate Point of Contact	Text	Facility contact name and title
Alternate Contact Phone Number	Text	Phone number (example (609) 883-9500 ext 270)
Type of sampling location	Text	(LOOKUP TABLE) Type of sampling location; Industrial, Municipal, Tributary, Estuary (tidal mainstem), River (non-tidal mainstem), Air-Urban, Air-Rural
Latitude	Numeric	Decimal degrees located to an accuracy of +- 1 meter at 95% confidence level relative to NAD83. Please provide 6 digits after the decimal (example 40.123456). A conversion equation from degrees/minutes/seconds to decimal degrees is on the "Lat&Long Conversion" tab of this Excel Worksheet.
Longitude	Numeric	Decimal degrees located to an accuracy of +- 1 meter at 95% confidence level relative to NAD83. Please provide 6 digits after the decimal (example -75.123456). A conversion equation from degrees/minutes/seconds to decimal degrees is on the "Lat&Long Conversion" tab of this Excel Worksheet.
Zone	Numeric	(LOOKUP TABLE) DRBC Water Quality Zones 1,2,3,4,5,6,7,8 A map depicting DRBC Water Quality zones is available on the DRBC web site at http://www.state.nj.us/drbc/maps/InterstateZones-MainStem.pdf. Zone "7" refers to the non-tidal portion of the Schuylkill River. Zone "8" refers to NJ discharge locations outside of the Delaware River Basin
River Mile	Numeric	DRBC River mile location if known. Please provide two digits after the decimal point (example 75.25) If discharge locations are on the Mainstem Delaware you please refer to the river mileage system provided the DRBC web page at http://www.state.nj.us/drbc/mileage.htm
Drainage Area	Numeric	To discharge location if applicable in square miles. Please provide two digits after the decimal point (example 2.22)
Sample Depth	Numeric	Sample depth of sample collection from water surface, if applicable in feet. Please provide two digits after the decimal point (example 6.8)
Permit Number	Numeric	NPDES # only (example PA0123456)
Discharge ID	Numeric	outfall number (example 001, or 001a)
Status	Text	(LOOKUP TABLE) Active, Inactive

Electronic Data Deliverables for Chain of Custody Information

DATA FIELDS	FORMAT	DEFINITIONS
Location ID	Alpha Numeric	State abbreviation NPDES # and outfall (example PA0123456-001)
Location ID (second outfall)	Alpha Numeric	State abbreviation NPDES # and outfall (example PA0123456-002) if necessary
Sample ID	Alpha Numeric	Consists of 9 digit NPDES #, sample type, outfall, and collection date. Example: PA0123456-DW-001-12312004
Sample ID (second Outfall)	Alpha Numeric	Consists of 9 digit NPDES #, sample type, outfall, and collection date. Example: PA0123456-DW-002-12312004 if necessary
Sample Date	MM/DD/YY	Date format example 12/31/04 (End Date for composite samples)
Sample Time	HH:MM	Time Format example 2:30 PM (End Time for composite samples)
Sample Collection Type	Lookup	(LOOK UP) Grab, 24-hr Comp
Weather category	Lookup	(LOOKUP TABLE) WW= wet weather, DW= Dry Weather, NA=Not Applicable
Source Category	Lookup	(LOOKUP TABLE) Categories to be defined a latter date. A "NULL" entry is acceptable until categories are defined.
Sample Type	Lookup	(LOOKUP TABLE) Samples types: SA= Sample TB= Trip Bank, RB Rinsate Blank, INF=Influent Sample, OTH= Other
Sample Matrix	Lookup	(LOOKUP TABLE) Water (whole) = whole water sample including suspended particulate fraction, Water (dissolved) = water which passes through a filter media, Water (XAD) = water which is passed through an XAD media, Water (Particulate) = particulate fraction of water sample which remains on filter media, Sediment = solids which are collected sub-aqueaously, Soil = soils which are collected sub-arially, Tissue (whole body) = entire fish or organism, Tissue (fillet) = fish fillet, Air (particulate) = particulate fraction of air sample which remains on filter media, Air (dissolved) =dissolved fraction of air sample which passes through filter media
Sample Size	Numeric	Volume in Liters or milliliters, Mass in kilograms or grams
Sample Units	Text	Units (Liters, milliliters, Kilograms, grams)
Sample Depth (ft)	Numeric	Depth sample collected from water surface if Applicable in feet (example 6.8) Provide 1 digit after the decimal
Precipitation (total amount on sampling date)	Numeric	Amount of precipitation to (0.01")
Precipitation station	text	Precipitation station or gage used to determine amount of precipitation
Flow	Numeric	Total discharge from outfall during 24-hour event for continuous discharges or total flow during non-continuous event up to 24-hours (in MGD)
Notes	Text	Sampler's notes regarding conditions, description of collection methodology, identify previous rainfall event in excess of 0.1", any anomalies encountered
Analysis Requested	Lookup	(LOOKUP TABLE) 1668A, Other
Laboratory Undertaking Analysis	Text	Laboratory name
Laboratory Address	Text	Street address (no P.O. Box addresses)
Laboratory Contact	Text	Laboratory contact name and title
Laboratory Contact Phone Number	Text	Phone number (example (609) 883-9500 ext 270)
Date sent to lab	MM/DD/YY	Date sample sent to lab Date format example 12/31/2004
Sampler	Text	Name of sampler
sampler affiliation	Text	Employer of sampler

Electronic Data Deliverables for Analytical Information

DATA FIELDS	FORMAT	DEFINITION
Client_Sample_ID	TEXT	Client sample ID from Chain of Custody. Consists of 9 digit NPDES #, sample type, outfall, and collection date. Example: PA0123456-DW-
Lab_Sample_Id	TEXT	Laboratory Identification to be provided by Lab
Laboratory Name	TEXT	Name of Laboratory to be provided by Lab
Sample_Matrix	TEXT	Look up table Sample Matrix must match sample matrix from Chain of Custody. Water (whole) = whole water sample including suspended particulate fraction, Water (dissolved) = water which passes through a filter media, Water (XAD) = water which is passed through an XAD media, Water (Particulate) = particulate fraction of water sample which remains on filter media, Sediment = solids which are collected sub-aqueaously, Soil = soils which are collected sub-arially, Tissue (whole body) = entire fish or organism, Tissue (fillet) = fish fillet, Air (particulate) = particulate fraction of air sample which remains on filter media, Air (dissolved) =dissolved fraction of air sample which passes through filter media.
Percent_Moisture	NUMERIC	Percentage of moisture content of sample
Percent_Lipid	NUMERIC	Percentage of lipid content of sample
Qc_Code	TEXT	Look up table SA = sample, QADU = duplicate, MB = method blank, OPR = spike, MS = matrix spike, MSD = matrix spike duplicate, CCV = cal ver
Sample_Date	DATE (MM/DD/YY)	Sample Date from Chain of Custody
Sample_Time	TIME (HH:MM)	Sample Time from Chain of Custody
Analysis_Perfomed	TEXT	Analysis performed on sample (to be provided by Lab)
Extraction Date	DATE (MM/DD/YY)	Date sample was extracted
Analysis_Date	DATE (MM/DD/YY)	Sample analysis Date (to be provided by Lab)
Analysis_Time	TIME (HH:MM)	Sample analysis Time (to be provided by Lab)
Sample_Size	NUMERIC	Weight/volume of the sample (To two decimal places)
Size_Units	TEXT	Sample size units (g, L, mL)
Initial_Cal_Date	DATE (MM/DD/YY)	Date the initial calibration was run (to be provided by Lab)
Instrument Id	TEXT	Instrument identification (to be provided by Laboratory)
GC Column Id	TEXT	Gas Chromatograph Column identification (to be provided by the Laboratory)
Test Type	TEXT	Type of test values may include "initial, reextraction and reanalysis"
Test Batch Type	TEXT	Lab Batch Type values may include "prepratory, analysis and leach"
Batch_ID	TEXT	Batch Id (to be provided by the lab)
Cal_Ver_Lab_Sample_ID	TEXT	Calibration Verification Lab Sample ID associated with the sample (instrument run id to be provided by Laboratory)
Method_blank_lab _sample_ID	TEXT	Method Blank Lab Sample ID associated with the sample (instrument run id to be provided by Laboratory)
Compound	TEXT	Enter Full chemical compound name
IUPAC_PCB_#	TEXT	IUPAC PCB number from Table 1 EPA Method 1668 Revision A. 1999, for labeled analogs identify with "L" Example "3L","77L"
CAS_#	TEXT	CAS number from Table 1 EPA Method 1668 Revision A. 1999
Concentration Found	NUMERIC	Analyte concentration found
Dilution Factor	NUMERIC	Numeric Dilution Factor applied to extract
UNITS	TEXT	Reporting units (PG/L, PCT_REC, PG/G_DRYWT, etc - use CARP conventions)
Data_Qualifiers	TEXT	Look up table See Data Qualifier Tab on Spreadsheet
EDL	NUMERIC	Estimated Detection Limit See EDL Definition Tab on Spreadsheet
Minimum_Level	NUMERIC	Minimum Level as calculated by Lab
Conc_Lower_Limit	NUMERIC	Used for spikes and cal vers to show limits values are in percent recovery
Conc_Upper_Limit	NUMERIC	Used for spikes and cal vers to show limits values are in percent recovery
Ion_Abundance_Ratio	NUMERIC	Ion Abundance Ratio of the analyte (if present)
Ion_Abundance_Ratio Lower Limit	NUMERIC	Lower limit of Ion Abundance Ratio of the analyte in the Cal Ver associated with the sample
Ion_Abundance_Ratio Upper Limit	NUMERIC	Upper limit of Ion Abundance Ratio of the analyte in the Cal Ver associated with the sample
RRT	NUMERIC	Relative Retention Time of the analyte (if present)
RRT_Lower_Limit	NUMERIC	Lower limit of Relative Retention Time of the analyte in the Cal Ver associated with the sample
RRT_Upper_Limit	NUMERIC	Upper limit of Relative Retention Time of the analyte in the Cal Ver associated with the sample

DRBC – Estimated Detection Limits

Estimated Detection Limit

For analyte ‘x’, the EDL is calculated by the following formula:

$$EDL_x = 2.5 \bullet \frac{(Na \bullet Qis \bullet Rah)}{(Ais \bullet RRF \bullet wv)}$$

Where:

Na= Analyte peak to peak noise height.
Qis= Concentration of the internal standard
Rah= Area Height Ratio.
Ais= Area of internal standard
RRF= initial calibration average relative response factor for the congener of interest.
wv= Sample weight/volume.
2.5= Minimum signal to noise ratio.

Noise calculations are to be taken from the discrete sections of the chromatogram rather than the entire chromatograph for a mass descriptor.
No peak smoothing of the chromatograph is to be undertaken. Peak identification to be conducted on the raw chromatograph.

DRBC – Hard Copy Data Package Deliverables

OBJECTIVE

This Standard Operating Procedure (SOP) describes the requirements for the analytical data packages that will be generated in association with the Stage 2 Delaware River Estuary PCB Total Maximum Daily Loads (TMDL) Project. This SOP applies to the contractor(s) involved in analytical data generation and reporting. Two hard copy data packages as well as an Adobe Acrobat (.PDF) file format must be generated for the Stage 2 Delaware River Estuary PCB TMDL. The laboratory is required to maintain copies of the .pdf and hard copy data packages for 5 years from the time of submission.

DATA PACKAGE DELIVERABLES

The following sections describe in detail the types of data packages designed for the Stage 2 Delaware River Estuary PCB TMDL project. These details are provided to allow several participating laboratories to produce data packages that are similar in format, order of presentation, and content. The data package deliverables are divided into two sections; Section 1.1 and 1.2. These Sections have been developed based on deliverables specified in the US EPA Contract Laboratory Program Statement of Work (CLP SOW). Section 1.1 specifies the data package contents and order of presentation. Section 1.2 provides details concerning specific contents of the data deliverables described in Section 1.1.

The fully documented data package described in this SOP resembles the information required by the CLP SOW. This type of package includes a cover letter, SDG narrative, field Chain-of-Custody Records, analytical results summaries, a glossary of qualifier codes, summary forms for quality control procedures and all sample and quality control raw data to support the results reported.

1.1 Data Package Contents and Order of Presentation

The laboratory must submit supporting documentation for the reported analytical results. Furthermore, the data package deliverables must be submitted in the order in which the deliverables appear in the text.

The Sample Data Package shall include data for analyses of all samples in one SDG, including field samples, second column analyses, re-extractions, reanalyses, secondary dilutions, blanks, ongoing precision and recovery (OPR) standards, matrix spikes, matrix spike duplicates, and/or laboratory duplicates. The complete Sample Data Package is divided into the units as described below. The Sample Data Package must be complete before submission and must be paginated. The Sample Data Package will be arranged in the following order:

A) Cover Letter/Letter of Transmittal signed by the laboratory manager.

B) Title Page

C) Table of Contents (or indexing system such as tabs or bookmarks and links as in a .pdf file)

D) Sample Delivery Group (SDG) Narrative

This document shall be clearly labeled "SDG Narrative" and shall contain: laboratory name; SDG number; field sample identifications; laboratory sample numbers; and detailed documentation of any quality control, sample, shipment, and/or analytical problems encountered in processing (preparing and analyzing) the samples reported in the data package. (A glossary of qualifier codes used in the SDG must also be provided.)

The laboratory must also include any technical and administrative problems encountered, corrective actions taken and method of resolution, and an explanation of all flagged edits (*i.e.*, exhibit edits) on quantitation reports.

The SDG Narrative must be signed and dated by the laboratory manager, project manager or the chemist that reviewed and approved the release of data.

E) Field Chain-of-Custody Records and Sample Receipt Documentation Log

Copies of the field Chain-of-Custody Records for all samples within the SDG must be included in the deliverables. A description of the condition and temperature of the samples upon laboratory receipt (*i.e.*, custody seal condition, container status) must be provided for each Chain-of-Custody Record/sample cooler.

F) GC/MS Polychlorinated Biphenyl (PCB) Data

1. Quality Control (QC) Summary

   a. Duplicate Precision Summary (if requested).

   b. Ongoing Precision and Recovery (OPR) Summary.

   c. Method Blank Analysis Summary.

2. Sample Data

   Sample data shall be arranged in packets consisting of the Analytical Results Summaries followed by the raw data for PCB samples. These sample

packets should then be placed in increasing alphanumeric order by sample identification. The order of each sample packet is as follows:

a. Analytical Results Summary.

For each sample, including compound/peak relative retention times (RRTs), peak co-elution information, ion abundance ratios, reported concentrations, laboratory qualifiers, Estimated Detection Limits (EDLs), internal standard (both extraction and injection) recoveries, and clean-up standard recoveries.

b. Quantitation Report-must be submitted and include all information required to reproduce reported positive results and EDL results.

c. Selected Ion Current Profile (SICP) Chromatograms. (SICP are to be provided in pdf format only. They are not required to be submitted as hardcopies.)

d. Second Column Data (if necessary; will include 1.1.1.F, Section 2, items a, b, and c).

e. Work sheets which include example calculations showing how sample results were calculated using initial calibration and sample responses for at least one sample per data package. The calculations should cover both positive results and EDLs

3. Standards Data

a. Mass spectrometer resolution data for each calibration associated with the SDG, in chronological order by GC column, by instrument.

b. Multi-point Initial Calibration by Isotope Dilution Data for the toxics/LOC CBs (Initial Calibration Summary Form, quantitation report, and SICP Chromatograms) for each initial calibration associated with the SDG, in chronological order by GC column, by instrument. Note, for the labeled compounds, internal standard calibration is performed using the multi-point initial calibration data. If a curve equation is utilized, the laboratory must provide the curve equation and coefficient of determination.

c. Single-Point Initial Calibration by Internal Standard Data for the native CBs for which a labeled compound is not available (Initial Calibration Summary Form, quantitation report, and SICP Chromatograms) for each initial calibration associated with the SDG, in chronological order by GC column, by instrument.

d. Calibration Verification Data (Calibration Verification Summary Form, quantitation report, and SICP Chromatograms) for each calibration verification associated with the SDG, in chronological order, by GC column, by instrument.

4. Raw QC Data

For the blank, OPR standard, and Laboratory Duplicate provide in chronological order, by instrument:

i. Analytical Results Summary.

Compound/peak RRTs, peak coelution information, ion abundance ratios, reported concentrations, laboratory qualifiers, EDLs, internal standard (both extraction and injection) recoveries, and clean-up standard recoveries. Also provide any imbedded and/or associated calculations or comments imbedded on the chromatograms.

ii. Quantitation Report.

iii. SICP Chromatograms.

5. GC/MS Instrument Run Logs.

6. Extract Clean-up Data (if available)

a. UV traces from GPC cleanup (if performed).

i. UV traces for the initial calibration standards and blanks. Compound names shall be written or printed over the peaks, or retention times shall be written over the peaks, and a separate table listing compounds and retention times shall be provided.

ii. SICP Chromatograms and quantitation reports for the GPC calibration check solution and all standards used to quantify compounds in the GPC calibration check solution.

G) Preparation Logs

1. PCB Extraction and Clean-up Logs.

1.2 Deliverables Reporting Requirements for PCB Analyses

The laboratory will be required to submit the following information as support documentation for the reported analytical results. All documentation shall be provided in hard copy format and as an Adobe Acrobat ..pdf file with the exception of the chromatograms, which shall be provided only in the .pdf format. The quality control summary forms must include the acceptance criteria (*i.e.*, recovery ranges, relative percent difference limits, *etc.*) and spike-added amounts (where applicable). Additionally, the quality control summary forms must indicate any recoveries that are outside of the acceptance criteria. The raw data associated with the samples, blanks, and standards must clearly identify the laboratory sample number, the instrument, the laboratory file number for the analysis, and the peak areas/heights and retention times that correspond to the compounds of interest observed in all analyses reported. The raw data must provide all information necessary to reproduce all reported positive and EDL results. If the requirement of a summary form is not applicable to a particular sample, standard, or blank, the requirement should still appear on the form; however, no entry will be necessary on the form for that requirement.

A) 1. An analysis summary of the results for all target compounds for all sample analyses, second column analyses, re-extractions/reanalyses, secondary dilutions, matrix spike analyses, matrix spike duplicate analyses, laboratory duplicate analyses, OPR standard analyses, and method blank analyses must be supplied. The summary must include an entry for each congener concentration, date(s) and time(s) of analysis, sample identification, laboratory sample number, date of sample collection, date of sample preparation, sample matrix, sample weight, sample percent solids, column

type(s), column internal diameter(s) dilution factor, concentrated extract volume, concentration units, peak relative retention times, co-elution information, isotope ratios, and sample results. If positive results below the lowest calibration standard are reported, they must be flagged as estimated ("J") on the analysis summary. "Not-detected" results will be represented by the EDL and a "U" flag. If a compound was detected in a sample as well as in the method blank associated with the sample, the result must be flagged with a "B" on the summary form. Additionally, if a dilution is performed on a sample because a target compound is above the calibration range, then the positive result for the particular compound should be flagged with a "D". If the compound is still above the calibration rage after a dilution is performed on the sample, the positive result for the compound should be flagged with an "E". A description of qualifier flags for this DRBC project is provided in the description of Data Qualifiers.

2. The raw data for the field sample, second column, re-extraction/reanalysis, secondary dilution, blank, OPR standard, matrix spike, matrix spike duplicate, and/or laboratory duplicate analyses by GC/MS methodologies, consisting of the SICP, quantitation reports for the target compounds, the associated areas or height for each peak within the established retention time window, and all other information required to reproduce all reported positive and EDL results.

B) A OPR and percent recovery summary for each OPR analyzed is required. The OPR summary form will indicate the concentrations of the compounds present in the spiked sample. The summary form should also include the OPR recovery criteria. The laboratory should mark the compounds that do not meet the specified criteria.

C) A relative percent difference summary for each laboratory duplicate analyzed is required. The laboratory duplicate summary form will indicate the field identification of the parent sample, the sample, the matrix, and the concentrations of the compounds present in the parent and duplicate sample. The summary form should also include the RPD criteria. The laboratory should mark the compounds that do not meet the specified criteria.

D) A method blank summary form for each method blank that identifies the samples associated with each method blank. The date of extraction, date of analysis, time of analysis, lab file number, sample weight, and matrix of the method blank must also be reported on the summary form.

E) Mass spectrometer resolution data for the reference standard (PFK or other substance) analyzed to demonstrate mass resolution. Output for each descriptor should identify the lab file identification, date and time of analysis, instrument identification, and exact mass ions monitored.

F) A summary of the analytical sequence for each column and instrument used for the analysis of the project samples. The summary must contain the GC column number, the internal diameter of the column, initial calibration dates associated with the sequence, the instrument identification, a listing of the laboratory sample numbers, and dates and times of analysis. The summary must contain all of the analyses for the samples, blanks, initial calibration standards, and the continuing calibration standards associated with the sequence.

G) 1. An initial calibration summary for each multi-point initial calibration performed, summarizing all of the relative response factors for each calibration standard, the average relative response factor, and the relative

standard deviation among the relative response factors. If calibration curve equations are utilized, the laboratory must supply the curve equation and coefficient of determination. Additionally, the summary should indicate maximum relative standard deviation criteria as well as the compounds that did not meet the acceptance criteria. The summary should indicate the instrument identification, the dates and times of calibration commencement and completion, column type, and diameter of the column.

2. An initial calibration summary for each single-point initial calibration performed, summarizing all of the relative response factors for each compound. The summary should indicate the instrument identification, the dates and times of calibration commencement and completion, column type, and diameter of the column.

3. The raw data for the initial calibration, consisting of the SICPs and the raw quantitation report for each calibration standard.

H) 1. A calibration verification summary for each calibration verification standard analyzed, summarizing the true and found concentrations, and the percent recoveries and the relative response factors of the calibration verification, and the isotope ratios and retention times. Additionally, the summary must indicate the compounds that are subject to recovery criteria, and the compounds that did not meet the acceptance criteria. The summary should indicate the instrument identification, the date of the initial calibration, the date and time of analysis, column type, and diameter of the column.

2. The raw data for the calibration verification, consisting of the SICPs and the raw quantitation report for each calibration standard.

DRBC – List of Laboratories that Perform Low-Level PCB Congener Analyses

Listing of Laboratories Known to Perform Low Level PCB Congener Analyses

Axys Analytical Services Ltd.
P.O. Box 2219
2045 Mills Road
Sidney, British Columbia
CANADA V8L 5X2
Contact: Georgina Brooks
Phone - direct: (250) 655-5801
Phone - general: (250) 655-5800
Fax: (250) 655-5811
Email: gbrooks@axys.com

Battelle Laboratories
505 King Avenue
Columbus, OH 43201
Contact: Mary E. Schrock
Phone: (614) 424-4976
Fax: (614) 424-3638
Email: schrock@battelle.org

Battelle Ocean Sciences
397 Washington Street
Duxbury, MA 02332
Contact: John Thorn
Phone: (781) 952-5200
Fax: (781) 952-5221
Email: thornj@battelle.org

Cape Fear Analytical, LLC.
3306 Kitty Hawk Rd., Suite 120
Wilmington, NC 28405
Contact: Christopher Cornwell
Phone: (910) 795-0421
Email: chris.cornwell@cfanalytical.com

Midwest Research Institute
425 Volker Boulevard
Kansas City, MO 64110
Contact: Anne Reid
Phone: (816) 753-7600 ext. 1134
Fax: (816) 753-8420

Pace Analytical Services
1700 Elm Street, Suite 200
Minneapolis, MN 54414
Contact: Matt Burns
Phone: (732) 343-3558
Email: matt.burns@pacelabs.com

SGS North America, Inc.
5500 Business Drive
Wilmington, NC 28405
Contact: Amy Boehm
Phone: (910) 350-1903
Fax: (910) 350-1557
Email: amy.boehm@SGS.com

TDI-Brooks International, Inc.
1902 Pinon
College Station, TX, 77845
Contact: James M. Brooks
Phone: (979) 693-3446
Fax: (979) 693-6389
Email: Drjmbrooks@aol.com

TestAmerica - Knoxville
5815 Middlebrook Pike
Knoxville, TN 37921
Contact: John Reynolds
Phone: (865) 291-3000
Fax: (865) 584-4315
Email: info@testamericainc.com

TestAmerica - West Sacramento
880 Riverside Parkway
West Sacramento, CA 95605
Contact: Nilo Ligi
Phone: (916) 373-5600
Fax: (916) 372-1059
Email: nilo.ligi@testamericainc.com

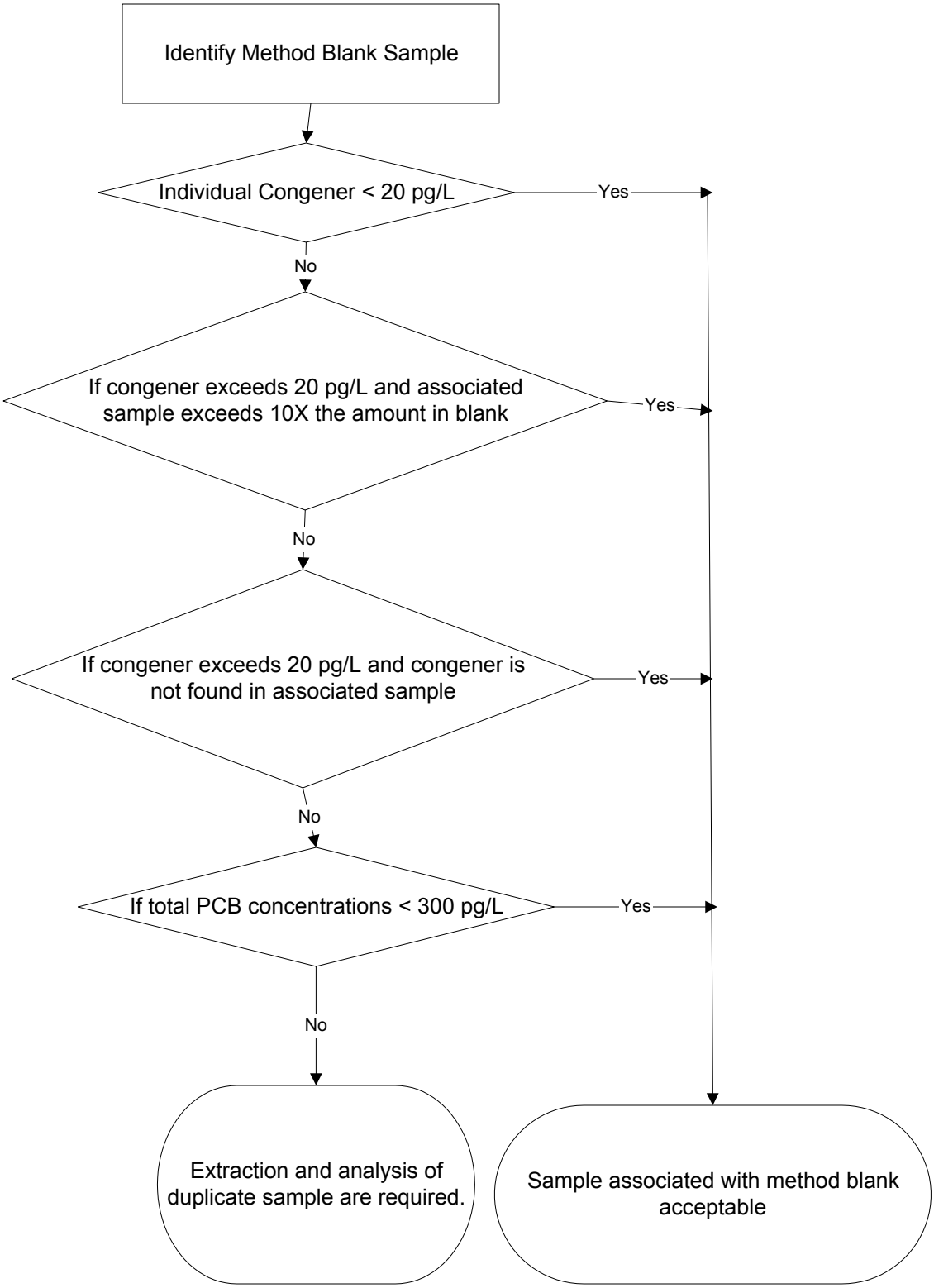
Texas A&M Research Foundation
Geochemical & Environmental Research Group
833 Graham Road
College Station, TX 77845
Contact: Terry Wade
Phone: (979) 862-2323
Fax: (979) 862-2361
Email: terry@gerg.tamu.edu

Vista Analytical Laboratory, Inc.
1104 Windfield Way
El Dorado Hills, CA 95762
Contact: William Luksemburg
Phone: (916) 673-1520
Fax: (916) 673-0106
Email: billux@altalab.com

Updated: 2/25/15

DRBC – Method Blank Contamination Decision Rules

Method Blank Contamination Decision Rules



DRBC – EPA Method 1668A, Project Quality Control Requirements

DELAWARE RIVER ESTUARY STAGE 2 PCB TMDL
Polychlorinated Biphenyls - EPA Method 1668A
Project Quality Control Requirements

INTRODUCTION:

This document provides a summary of the analysis quality control requirements for the Polychlorinated Biphenyls (PCBs) analysis by EPA Method 1668A for the Delaware River Estuary Stage 2 PCB TMDL. Additional information on sampling and analytical requirements can be found on DRBC's website (http://www.state.nj.us/drbc/PCB_info.htm). This summary of quality control requirements is based on EPA Method 1668A, (Method 1668, Revision A: Chlorinated Biphenyl Congeners in Water, Soil, Sediment and Tissue by HRGC/HRMS, EPA-821-R-00-002, December 1999) with project specific modifications to meet the Data Quality Objectives (DQOs) for the Stage 2 PCB TMDL. This QC summary primarily addresses aqueous analysis associated with Point Source Discharge samples. Revision may be necessary in the future to address additional matrices and DQOs. EPA Method 1668A is a Performance Based method. Project specific requirements stated in this QC summary must be met to ensure consistency amongst participants in the project and cannot be modified. Questions or concerns with the QC requirements should be discussed with the Delaware River Basin Commission prior to implementing any changes.

Quality Control Item	Frequency	Acceptance Criteria	Corrective Action
Sample Collection, Preservation, Storage and Holding Times	Samples must be collected in amber glass (or aluminum foil for tissue samples) following conventional sampling practices. • A two (2) liter (L) aqueous sample size will be collected for this project. Two, 2-L amber glass containers for aqueous sample collection should originate from the analysis laboratory and the container quality be verified for cleanliness. Documentation showing traceability and cleanliness of the containers must be maintained at the laboratory. A 2-L sample and 2-L replicate sample will be collected per location. The replicate is available in the event reextraction and reanalysis due to failing QC is necessary. • The laboratory will supply reagent grade water for use in collection of field blanks. A sufficient quantity of water should be provided to collect a 2-L field blank.	• Samples should be stored at <6°C until delivery to the laboratory. • If stored in the dark at <6°C aqueous samples may be stored for up to one year. (If residual chlorine is present, 80 mg of thiosulfate per liter of water should be added.) • If stored in the dark at <-10°C, solid, semi-solid, multi-phase, and tissue samples may be stored for up to one year. • If stored in the dark at <-10°C, extracts may be stored for up to one year.	• There are no demonstrated holding times for CBs. • Resample if able or deemed necessary for project DQOs. Otherwise, qualify results based on professional judgment if the potential for a low bias exists due to excessive holding times.

Quality Control Item	Frequency	Acceptance Criteria	Corrective Action
Aqueous Sample Extraction and Cleanup	Extraction and cleanup of the sample must use one of the techniques described in EPA Method 1668A. • The entire contents of the 2-L sample volume must be extracted (do not separate solids if greater than 1% as described in Section 11.5 of the Method 1668A- extract the sample as is). The exact volume extracted must be documented and used in calculation of the sample concentration. All spike additions must be added to the 2-L bottle containing the sample prior to extraction. • Laboratories may purchase glassware/extraction equipment to accommodate the larger 2-L sample volume (Note: the method typically extracts 1-L of aqueous sample). Alternatively, the laboratory may serially extract the 2-L volume in the same device one-liter at a time or extract two 1-L portions simultaneously in two different apparatus set-ups. Combine the solvent if serial or sequential extractions were performed prior to extract concentration and cleanup. • Sample extracts will be concentrated to a final volume of 20 ul.	• Method blanks, OPR samples, field blanks or other QC samples must be processed identically to the samples including the same extract cleanups.	• Contact the client for guidance if the sample size or matrix does not allow these conditions to be met.
Retention Time Calibration	• This project requires the use of the SPB-octyl column. • Each diluted individual congener solution (Section 7.10.2.1.2 of the method) is injected to establish the beginning and ending retention times for the scan descriptions in Table 7. • The diluted combined 209-congener solution is injected (Section 7.10.2.2 of the method).	• The absolute retention time (RT) of CB 209 must exceed 55 minutes on the SPB-octyl column. • The RT and relative RT (RRT) for all congeners must be within the windows in Table 2 of the method and the column performance specifications in Sections 6.9.1-6.9.1.2 of the method must be met.	• If the absolute RT of CB 209 does not meet criterion, the GC temperature must be adjusted and the test repeated until the minimum RT criterion is met. • Adjust chromatographic conditions and scan descriptors until all criteria are met. NOTE: Laboratories with newer injection technology such as Electronic Pressure Control (EPC) may render the RT requirement for CB 209 obsolete. The RT and RRT for all congeners and coeluting congeners must be documented at the same frequency as stated in this section for systems using EPC.

Quality Control Item	Frequency	Acceptance Criteria	Corrective Action
Mass Spectrometer Resolution	• The instrument is tuned using perfluorokerosene (PFK, or other reference material). • Static resolving power checks must be performed at the beginning and at the end of each shift.	• A minimum resolving power of 10,000 for a significant PFK fragment in the range of m/z 300-350. The deviation between the exact m/z and the theoretical m/z (Table 7 of the method) for each exact m/z monitored must be less than 5 ppm.	• Any problems must be corrected before analyses can proceed. • Any samples in the previous shift that may be affected by poor resolution must be reanalyzed.
Ion abundance ratios and signal-to-noise (S/N) ratios	The low calibration standard concentration for this project must be 0.5 ng/ml. A 1 or 2 µL aliquot of the 0.5ng/ml calibration solution is injected.	• All CBs and labeled compounds in the 0.5 ng/ml standard must be within the QC limits in Table 8 of the method for their respective ion abundance ratio. • The peaks representing the CBs and the labeled compounds in the 0.5 ng/ml calibration standard must have S/N ≥ 10.	The mass spectrometer must be adjusted and this test repeated until the m/z ratios fall within the limits specified If the adjustment alters the resolution of the mass spectrometer, resolution must be verified prior to the repeat of the test.
Initial Calibration	• Established initially and when calibration verification fails criteria. • Calibration by isotope dilution is performed at a minimum of 5 (6 may be used) concentration levels for each of the toxic/level of chlorination (LOC) congeners (refer to Table 3 of the method). • The low calibration standard concentration for this project must be 0.5 ng/ml. • Calibration by internal standard is performed for each native congener for which a labeled congener is not available, the labeled toxics/LOC/window-defining congeners, and the labeled cleanup congeners. For the native congeners, calibration is performed at a single point using the CS-3 standard. For the labeled congeners, calibration is performed using the data from the 5 (or 6) points in the calibration of the toxics/LOC congeners.	%RSD≤20% among relative response (RR) for each native toxic/LOC congener in order to use the average RR (as calculated in Section 10.4.2 of the method). Otherwise, the complete calibration curve for that congener must be used over the calibration range.	• Reanalyze the initial calibration curve and/or evaluate/correct instrument malfunction to obtain initial calibration that meets criteria. • Sample results above highest standard concentration require dilution and reanalysis. In addition, the concentration of the labeled injection internal standard must be adjusted to 100 pg/µL.

Quality Control Item	Frequency	Acceptance Criteria	Corrective Action
Calibration Verification	• Performed at the beginning of each 12-hour shift during which analyses are performed. • The CS-3 calibration verification (VER) standard and the diluted combined 209 congener solution are analyzed.	• The m/z abundance ratios for all congeners must be within the limits in Table 8. • The GC peak representing each native CB and labeled compound in the VER solution must be present with a S/N ratio of at least 10. • The concentration of each compound must be within the limit in Table 6 of the method.	• Adjust system, if necessary, and recalibrate. Criteria must be met before sample, blank, IPR, and OPR analysis may begin. • If the adjustment alters the resolution of the mass spectrometer, resolution must be verified prior to the repeat of the verification test.
Retention Times	• This project requires the use of the SPB-octyl column. • Retention times are verified using the calibration verification analysis. • Coeluting congeners must be reported according to the scheme defined in the Qualifier Codes for the project (http://www.state.nj.us/drbc/PCB-DataQualFlags.pdf)	• The absolute RTs of the labeled toxics/LOC/window-defining standard congeners in the verification test must be within ±15 seconds of the respective RTs in the calibration. • The RRTs of the native CBs and labeled compounds in the verification test must be within their respective RRT limits in Table 2 of the method.	Adjust system or replace GC column and repeat the verification test or recalibrate. (See previous note concerning GC systems with EPC.)
GC Resolution and minimum analysis time	As part of calibration verification, the diluted combined 209-congener solution is analyzed.	The resolution and minimum analysis time specifications in Sections 6.9.1.1.2 and 6.9.1.1.1 of the method must be met for the SPB-octyl column.	Adjust GC analysis conditions until the specifications are met, or the column must be replaced and the calibration verification tests repeated or the system recalibrated. (See previous note concerning GC systems with EPC.)
Ongoing precision and recovery (OPR)	• Prepared with each batch of samples (samples started through the extraction process on a given 12-hour shift, to a maximum of 20 samples). • Analyzed prior the analysis of samples from the same batch.	The recoveries of the toxic/LOC CBs must be within the OPR limits given in Table 6 of the method.	If any individual concentration falls outside of the range, the extraction/concentration processes are not being performed properly. The problem must be corrected and the sample batch must be reprepared, extracted, and cleaned up and the OPR test repeated.

Quality Control Item	Frequency	Acceptance Criteria	Corrective Action
Method Blank	• Prepared with each batch of samples (samples started through the extraction process on a given 12-hour shift, to a maximum of 20 samples). • Analyzed prior the analysis of samples from the same batch immediately following the analysis of the OPR. • The reference matrix must simulate, as closely as possible, the sample matrix under test. • When a reference matrix that simulates the sample matrix under test is not available, reagent water can be used to simulate water samples; playground sand or white quartz sand can be used to simulate soils; filter paper can be used to simulate papers and similar materials; and corn oil can be used to simulate tissues.	• Method blanks must meet the decision rules specified on DRBC's web site: http://www.state.nj.us/drbc/PCB-MethodBlankRules.pdf	If the method blank acceptance criteria is exceeded, analysis of samples must be halted until the sample batch is re-extracted (using the replicate sample) and the extracts re-analyzed, and the blank associated with the sample batch shows no evidence of contamination above the acceptance criteria. All samples must be associated with an acceptable method blank before the results for those samples may be reported or the specific conditions preventing the ability to achieve the method blank acceptance criteria discussed with the client.
Labeled Toxics/LOC/window-defining standard spike	All samples must be spiked with labeled compounds to monitor method performance. The spiking of the extraction standards must occur prior to extracting the sample. The addition of the cleanup standards must occur before the fractionation, while the addition of the injection standards is conducted prior the GC/MS analysis.	The recovery of each labeled compound must be within the limits in Table 6.	If any labeled compound falls outside of limits, the method performance is unacceptable for that compound in that sample. Additional cleanup procedures must be employed to attempt to bring the recovery within the normal range. If the recovery cannot be brought within the normal range after all cleanup procedures have been employed, water samples are diluted and smaller amounts of soils, sludges, sediments, and other matrices are analyzed.

DELAWARE RIVER ESTUARY STAGE 2 PCB TMDL
Polychlorinated Biphenyls - EPA Method 1668A
Project Quality Control Requirements

Quality Control Item	Frequency	Acceptance Criteria	Corrective Action
Qualitative/Quantitative Issues	Identification of a CB or labeled compound in a standard, blank or sample occurs when must meet all criteria are meet. • Report results for all 209 PCB congeners. The Qualifier Codes provides a mechanism to report coeluting congeners (http://www.state.nj.us/drbc/PCB-DataQualFlags.pdf). • Report results according to the hardcopy data package deliverable and Electronic Data Deliverable (EDD) specifications posted on DRBC's web site (http://www.state.nj.us/drbc/PCB_info.htm).	• The signals for the two exact m/z's in Table 7 must be present and must maximize within the same two scans. • The S/N for the GC peak at each exact m/z must be ≥ 2.5 for each CB detected in a sample extract, and ≥ 10 for all CBs in the calibration and verification standards. • The ratio of the integrated areas of the two exact m/z's specified in Table 7 must be within the limit in Table 8, or within ±15% of the ratio in the midpoint (CS3) calibration or calibration verification, whichever is most recent. • The RRT of the peak for a CB must be within the RRT QC limits specified in Table 2, or if an alternate column or column type is employed, within its respective RRT QC limits for the alternate column or column system. • Because of congener overlap and the potential for interfering substances, it is possible that all of the identification criteria above may not be met. It is also possible that loss of one or more chlorines from a highly chlorinated congener may inflate or produce a less-chlorinated congener that elutes at the same retention time. If identification is ambiguous, an experienced spectrometrist must determine the presence or absence of the congener.	• Congeners that are not detected are to be reported to the sample specific Estimated Detection Limit (EDL). EDLs must be calculated as described on DRBC's web site (http://www.state.nj.us/drbc/PCB-EDL.pdf) • If a peak does not meet the qualitative identification criteria (most commonly the ion abundance ratio criteria), the quantitative result for that congener must be reported as an Estimated Maximum Possible Concentration (EMPC).

DRBC – Rinsate Blank Contamination Assessment

RINSATE BLANK CONTAMINATION ASSESSMENT

The rinsate blank associated with a given set of field samples should be identified and labeled utilizing the sample identification protocols defined in the DRBC letter requesting sampling for the Stage 2 PCB TMDL.

The criteria presented below are set as an interim goal for evaluating rinsate blank contamination and may be reevaluated after the initial Stage 2 data set has been collected. An overall assessment of the rinsate blank results with respect to the associated field sample and method blank results will be conducted to determine if excessive contamination occurred from poor sampling or handling procedures.

Rinsate blank contamination acceptance rules:

- An individual congener cannot exceed 40 pg/L
- If a congener exceeds 40 pg/L and the associated sample concentration exceeds 3× the amount in the blank, then no action is required
- If a congener exceeds 40 pg/L and the congener is not found in the associated field sample, then no action is required
- The total PCB concentration cannot exceed 600 pg/L

If rinsate blank levels violate the rinsate blank contamination rules the source of external contamination should be investigated and eliminated, if possible. Furthermore, resampling and reanalysis may be required at the discretion of the DRBC.

Sample, Replicate and Blank Collection Techniques

All samples shall be collected as 24-hour time-weighted composite samples or grab samples at a nominal volume of 2 liters. Two 2-liter samples shall be collected simultaneously; however a single 4-liter sample cannot be split into a sample and its replicate. In addition all required sampling events shall provide for the collection of field replicate and rinsate blank samples.

Sample and Replicate Collection Techniques for Continuous Discharges

Sample collection technique during dry weather conditions

Dry weather conditions are defined as when no rainfall (defined as less than 0.1 inches) has occurred within the previous 72 hours. Samples collected from continuous discharges during dry weather will be taken as 24-hour time-weighted composites samples at a frequency of not greater than one aliquot every hour for a nominal sample volume of 2 liters for both the sample and the field replicate.

Sample collection technique during wet weather conditions

Wet weather conditions are defined as following the onset of a precipitation event of 0.1 inches or greater **and** an increase in wastewater flow, provided that no rainfall (defined as less than 0.1 inches) has occurred within the previous 72 hours. Samples collected from continuous discharges during wet weather flows will be taken as 24-hour time-weighted composite samples at a frequency not greater than one aliquot every hour for a nominal sample volume of 2 liters for both the sample and the field replicate. Sampling should start no sooner than 2 hrs prior to the start of the rising hydrograph or no later than 30 minutes after the start of the rising hydrograph for the discharge

Sample and Replicate Collection Techniques for Non-Continuous Discharges

Non-continuous dischargers refer to either batch discharges which occur intermittently and are not precipitation-induced or to storm water discharges which occur during and/or after precipitation events but do not provide for continuous long-term discharge.

Sample collection technique for Batch Discharges

A two liter grab sample will be collected into a laboratory supplied bottle, sealed and stored at between 0-4 degrees C for shipment. A replicate sample will be collected and treated in the same manner as the sample.

Sample collection technique for Storm Water Discharge

A two liter grab sample will be collected into a laboratory supplied bottle within 30 minutes of the start of the discharge, sealed and stored at between 0-4 degrees C for shipment. A replicate sample will be collected and treated in the same manner as the sample.

Field Replicates and Rinsate Blanks Collection

Field Replicates

Field replicate samples are defined as "Independent samples that are collected as close as possible to the same point in space and time. They are two separate samples taken from the same source, stored in separate containers, and analyzed independently." They are required as a backup for the analytical laboratory and will be analyzed in the event of:

> Damage to the primary sample during shipment and handling (e.g., sample bottle broken).
>
> and/or
>
> If lab blanks associated with the analysis of the primary sample show clear evidence of contamination.

Therefore, replicate samples should be shipped to the laboratory and held for possible analysis. EPA method 1668A provides for the storage of aqueous samples for up to one (1) year.

Rinsate Blanks

Rinsate blanks are defined as "a blank consisting of analyte-free media which has been used to rinse the sampling equipment. It is collected after completion of equipment decontamination and prior to sampling." Water and sample bottles used in the collection of rinsate blanks shall be supplied by the laboratory which will be performing the analysis. The laboratories shall certify that the bottles and water are PCB free.

Trip blanks are not required, but may be collected at the samplers' discretion.

Rinsate Blanks will be collected and analyzed with the following frequency:

a. A rinsate blank per sampling event per piece of sampling equipment shall be collected.
b. One (1) rinsate blank per sampling event shall be analyzed. Concentrations from this rinsate blank contamination will be compared to the rinsate blank acceptability criteria. If rinsate blank exceeds acceptability criteria, then other samples shall be analyzed to confirm the level of contamination. Alternatively, resampling shall be conducted.

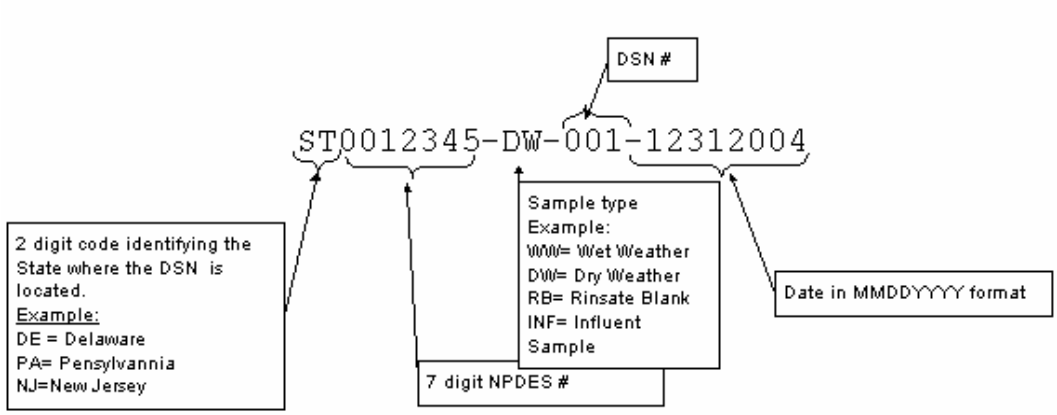
Rinsate Blank Collection Methods.

1. Rinsate Blank collection for 24-hour time-weighted composite samples:
    a. Two liters of laboratory supplied water is passed through all sample collection equipment that contacts the sample into a lab supplied 2 liter PCB free glass jar. Upon completion of rinsate blank collection, the bottle will be sealed and stored at between 0-4 degrees C for shipment. If contamination by the air surrounding the sampler or sample location is an issue, then an air blank sample may be collected by having a 2 liter bottle of lab water open to the air in a separate sampler or in an equivalent sampling environment for an equivalent amount of time that the composite sample is collected. Upon completion of the composite sample collection, seal the air blank and store at between 0-4 degrees C for shipment.
2. Rinsate Blank Collection for Grab Samples
    a. Two liters of laboratory supplied water is poured into a lab supplied 2 liter PCB free glass jar in the vicinity of the sampling location, sealed and stored between 0-4 degrees C for shipment.

DRBC – Sample Labeling and Identification

Sample Labeling and Identification

Each sample will be uniquely identified using the following labeling criteria:

1. NPDES# (9 -digit number including the state prefix (DE, NJ, PA))
2. Dry/Wet Weather Effluent Sample, Rinsate Blank, Influent Sample (DW, WW, RB, INF)
3. DSN# (three digit number)
4. Sample date (MMDDYYYY format)

An illustration is supplied below.