PBT Chemical Trends Determined from Age-Dated Lake Sediment Cores, 2014 Results



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2014 Highlighted Findings

- Lead and mercury concentrations increased in all three lakes until the late 20th century.
- Since the early 2000s, lead and mercury concentrations declined in Bead Lake, increased in Lake Goodwin, and remained stable in Mason Lake.
- Recent trends in lead and mercury fluxes were similar to concentration profiles, except in Mason Lake where fluxes consistently increased since the 1970s.
- 74% of samples contained one or more estimated detection of the chlorinated paraffins analyzed. No consistent temporal trends could be established for chlorinated paraffins.

Overview

The Washington State Department of Ecology's (Ecology's) Persistent, Bioaccumulative, and Toxic (PBT) Chemical Monitoring Program began collecting sediment cores from freshwater lakes in 2006 to help characterize the occurrence and temporal trends of PBTs in Washington State. A single sediment core is collected each year from three lakes and age-dated in order to reconstruct contaminant deposition profiles. The suite of chemicals to be tested is evaluated each year.

In 2014, Ecology analyzed lead, mercury, and chlorinated paraffins (CPs) in sediment cores collected from Bead Lake, Lake Goodwin, and Mason Lake (Figure 1). CPs are frequently used as flame retardants in plastics and sealants as well as additives in lubricants and cutting fluids (Bayen et al., 2006). They were the focus of testing because little information is available on their occurrence in Washington State.



Figure 1. Lake Sediment Core Locations, 2014.

Information provided by this sediment coring program supports development of agency Chemical Action Plans (CAPs) and is used to track progress on existing CAP recommendations and actions to reduce PBT levels in the environment.

For More Information

PBT Monitoring Program website: http://www.ecy.wa.gov/programs/eap/toxics/pbt.html

Chemical Action Plan website: www.ecy.wa.gov/programs/swfa/pbt/caps.html

Methods and Data Quality

This study followed a Quality Assurance Project Plan (QAPP) and QAPP addendum (Coots, 2006; Mathieu and McCall, 2014). Sediment cores measuring 35-45 cm deep were collected using a Wildco© box corer, according to Ecology standard operating procedures (Furl and Meredith, 2008). Surface sediments were collected with a standard ponar for grain size analysis. Manchester Environmental Laboratory (MEL) and contract laboratories analyzed samples for ²¹⁰Pb, total lead, mercury, total organic carbon (TOC), grain size, and CPs, using methods described in the QAPP and QAPP addendum. AXYS Analytical Services analyzed CPs by GC-MS, following method MLA-020. TOC and grain size data were used to help interpret contaminant concentrations and are not presented in this report. All data, including TOC and grain size, are available for download in Ecology's EIM database (<u>http://www.ecy.wa.gov/eim/</u>).

Data met measurement quality objectives (MQOs) with some exceptions. A small amount of activity was detected in one of the ²¹⁰Pb method blanks, below the reporting limit. Six samples were less than 5 times above the blank, but were already qualified as estimates, as they were below the reporting limit.

The contract laboratory conducting analysis of CPs changed their reporting procedure for CPs during the course of this study. At the time that the QAPP addendum for this analysis was written, the reporting procedure utilized a 3:1 signal to noise ratio for reporting, with estimated sample specific detection limits (SDL) of 1.5 - 4.5 ng/g. After receiving more data from this program and others, the lab identified a minimum concentration for positive identification (MCPI) for the confident identification of CPs. At the project manger's request, the lab reported results to SDLs, but results below the MCPI were qualified as "NJ", indicating the analyte has been tentatively identified and the result represents an approximate concentration. Several lab blanks contained "NJ" values above the SDL. Samples less than 10 times the blank levels were qualified as "U", not detected at or above the reported sample quantitation limit. Laboratory duplicate relative percent differences (RPDs) for short-chain and medium-chain CPs in one of the samples were above MQOs. Results were already qualified "NJ" and no further action was taken.

The CP results reported here represent estimated maximums and may include contributions from chlorinated compounds other than CPs. Due to the analytical difficulties inherent in CP analysis, all results and interpretations in this report should be considered estimations and comparability to other studies is limited.

Study Locations

Bead Lake is located in southeastern Pend Oreille County, north of Newport. The lake watershed consists of predominantly undeveloped forest land. Lodge Creek to the northeast and several other creeks provide inflow to Bead Lake, which has no outlet. The watershed

Table 2. Physical Descriptions of 2014 Study Lakes.

Waterbody	Max Depth (ft)	Mean Depth (ft)	Surface Area (ac)	Drainage Area (ac)	DA:SA	Elevation (ft)
Bead Lake	170	n/a	721	6,000	8.3	2,800
Lake Goodwin	50	23	560	3,315	5.9	324
Mason Lake	90	48	100	13,440	134	194

DA:SA = drainage area to surface area ratio

receives 38" of mean precipitation annually, and the basin is made up of gravelly sand soils (Schroeder, 1952).

Lake Goodwin lies 10 miles northwest of the city of Everett, in Snohomish County. The lake has a densely populated shoreline and the watershed consists of residential and forested land. Lake Goodwin is part of a series of lakes receiving inflow from Crabapple Lake and draining into Lake Shoecraft. The drainage area receives 32" mean annual precipitation, and basin geology consists of glacial drift with gravelly loam soils (Bortleson et al., 1976).

Located in Mason County, Mason Lake is 10 miles northeast of Shelton. Mason Lake has a large rural watershed, consisting mostly of commercial timberland with some residential development. The lake has year-round inflow from Shumocher Creek as well as numerous small intermittent streams. Sherwood Creek is the outflow for the lake. The watershed receives 68" mean annual precipitation and has predominantly gravelly, sandy loam soils (Bortleson et al., 1976).



Core Dating



Figure 2. ²¹⁰Pb Activity Plotted Against Sediment Core Depth. Dates assigned to sediment layer midpoint using the CRS model are included in graph.

Yearly unsupported ²¹⁰Pb fluxes

for Bead and Mason Lakes were 0.34 and 0.31 pCi/cm²/yr, respectively, while estimated unsupported ²¹⁰Pb flux in the Lake Goodwin core was low, at 0.05 pCi/cm²/yr. Focus factors were calculated to correct for the focusing of finegrained material to coring locations or the transport of sediments away from coring sites. The unsupported ²¹⁰Pb flux values were divided by estimated fluxes, calculated using lake-specific precipitation values and atmospheric ²¹⁰Pb deposition measured in Washington State (Nevissi, 1985; Lamborg et al., 2013). Focus factors for Bead, Goodwin, and Mason Lakes were 1.68, 0.21, and 0.88, and are applied to contaminant fluxes throughout this report.

Sediment Accumulation Rates

Sediment accumulation rates in all three lakes were low (Figure 3). Bead Lake ranged from 0.004 - 0.048 g/cm²/yr.

Rates increased in the early 1900s, peaking at approximately 1935, and have gradually declined to a modern rate of 0.008 g/cm²/yr.

Lake Goodwin sediment accumulation rates ranged from $0.004 - 0.013 \text{ g/cm}^2/\text{yr}$, similar to other Western Washington urban lakes with comparable drainage area to surface area ratios (e.g., Angle Lake and Lake Stevens; Mathieu and Friese, 2012; Mathieu, 2013). Sediment accumulation rates have remained near 0.01 g/cm²/yr throughout the 1900s, with a modest spike in the late 1960s.



Figure 3. Estimated Sedimentation Rates (g/cm²/yr).

Mason Lake sediment accumulation rates

ranged from 0.007 - 0.031 g/cm²/yr. Slight increases began in the early 1900s and rates rose more sharply from 1975 to the present peak rate of 0.031 g/cm²/yr.

Lead

Profiles

Lead concentrations in Bead Lake sediments remained near pre-industrial levels (13 ug/g) until the 1930s when concentrations began increasing. Concentrations rose over the 20th century until the peak of 78 ug/g in 2000. Modern sediments declined to 43 ug/g in 2008. Lead flux rates showed a similar profile to concentrations, with fluxes peaking in 2000 at 0.64 ug/ cm²/yr and decreasing to 0.23 ug/cm²/yr in the top-most layer.



Figure 4. Lead Concentration (ug/g) and Focus Factor-corrected Flux (ug/cm²/ yr) Profiles in Sediment Cores.

Lake Goodwin lead concentrations began at a baseline of 3 ug/g before the early 20th century. Concentrations began consistently increasing by mid-century to a peak concentration of 144 ug/g in the most recent sediment layer, 2008. Similar to Bead Lake, the lead flux profile closely matched the concentration profile. Maximum lead fluxes in Lake Goodwin were 5.43 ug/cm²/yr, occurring in the most recent layer.

Mason Lake lead concentrations were relatively low throughout the core. Lead concentrations remained near 2 ug/g until the early 1900s and then increased through the 1970s. Concentrations reached 54 ug/g in 1975 and peaked in 1991 at 55 ug/g, consistent with peak leaded gasoline use. Lead levels remained stable near 50 ug/g through modern sediments. Lead flux rates increased through the first half of the 20th century, along with concentrations. Fluxes stabilized between the 1950s and 1970s, then rose steadily through the top of the core to a peak rate of 1.78 ug/cm²/yr.

Enrichment

Enrichment factors (EFs) were calculated a individual lead concentrations divided by the average of stable pre-industrial lead concentrations at the bottom of the core (referred to as "baseline" in this report). "Modern" sediment refers to the top-most horizon of the sediment core (0 - 2 cm).

Table 3. Lead Concentrations and Enrichment Result

IS	Lake	Range (ug/g)	Baseline (ug/g)	Peak Enrichment	Modern Enrichment	
	Bead	4.94 - 77.7	12.9	6.0 (2000)	3.3 (2008)	
	Goodwin	1.47 - 114	2.6	44 (2007)	44 (2007)	
	Mason	1.37 - 54.8	1.6	34 (1991)	31 (2013)	

Table 3 displays lead enrichment factors for the three lakes.

Bead Lake sediments had the lowest enrichment, with peak concentrations 6 times greater than baseline in the year 2000 and modern sediments 3 times higher than baseline. Lake Goodwin sediments had the highest lead enrichment of the three lakes. Lead concentrations have increased each year since baseline levels in the late 1800s and were 44 times higher than baseline in 2007.

The Mason Lake core showed relatively low lead concentrations, but enrichment factors were 34 by 1991 and remained in the 30s through the top of the core.

Mercury

Profiles

Mercury concentrations in Bead Lake rose above pre-industrial levels by the 1930s, with a minor peak in 1950 measuring 57 ng/g. Concentrations showed a short decline between 1950 and 1970 and then began increasing until the maximum of 88 ng/g in 1994. Modern sediment concentrations have declined to 69 ng/g since the peak. Mercury flux rates followed a similar pattern to concentrations, although the peak flux (9.41 ug/m²/yr) occurred earlier, during

mid-century (1950). Mercury fluxes decreased in the most recent layer to $3.65 \text{ ug/m}^2/\text{yr}$.

Lake Goodwin's mercury concentrations remained fairly constant over the first half of the 1900s. Between 1950 and 2007, mercury concentrations showed a rapidly increasing pattern. The peak concentration (199 ng/g) occurred in the upper-most horizon, dated 2007. Mercury flux rates also rose throughout the Lake Goodwin core, with a minor peak in the late 1960s. The maximum mercury flux rate (94.8 $ug/m^2/$ yr) occurred in the top horizon, similar to peak concentrations.

The Mason Lake sediment core showed mercury concentrations



Figure 5. Mercury Concentration (ng/g) and Focus Factor-corrected Flux (ug/m²/yr) Profiles in Sediment Cores.

steadily increasing from the early 1900s to the 1970s. Concentrations have remained almost constant between the 1970s and most recent sediment layer, ranging from 135 - 142 ng/g. The maximum concentration was found in two sediment layers, dated 1991 and 2010. While concentrations remained stable over the last four decades, mercury fluxes increased consistently, with a maximum flux rate in the most recent layer (48.2 ug/m²/yr).

Enrichment

Mercury enrichment throughout the Bead Lake core was fairly low, with the peak EF of 3.3 occurring in the mid 1990s and a modern EF of 2.6 in 2008.

Peak and modern EFs were higher in the Goodwin and Mason cores.

 Table 4. Mercury Concentration and Enrichment Results.

Lake	Range (ng/g)	Baseline (ng/g)	Peak Enrichment	Modern Enrichment	
Bead	10.6 - 87.9	27	3.3 (1994)	2.6 (2008)	
Goodwin	22.8 - 199	31.9	6.2 (2007)	6.2 (2007)	
Mason	21.6 - 142	25.5	5.6 (1991 and 2010)	5.3 (2013)	

Mercury enrichment steadily increased from the 1950s through the top of the core in Goodwin Lake, with the peak of 6.2 occurring in the uppermost layer. Mason Lake sediment EFs increased until the mid 1970s, and then remained between 5.3 and 5.6 through modern sediments in 2013.

Chlorinated Paraffins

Chlorinated paraffins (CPs) are a group of chemicals that are used as industrial flame retardants, lubricants, and plasticizers, and as additives in adhesives, paints, rubber, and sealants (Muir et al., 2000). CPs were first produced in the 1930s and global production values increased considerably over the second half of the 20th century (Iozza et al., 2008). The term *chlorinated paraffins* refers to complex mixtures of polychlorinated alkanes with varying carbon chain lengths and chlorine contents. This study measured short-chain (SCCP), medium-chain (MCCP), and long-chain (LCCP) CPs, as well as total CPs (T-CPs). T-CP results are a separate chromatographic integration, rather than a sum of the mixtures.

SCCPs are listed on Ecology's PBT List because they are persistent, bioaccumulative, and toxic at low concentrations to aquatic organisms. SCCPs have been classified as "reasonably anticipated to be human carcinogens", based on animal

Waterbody	Year	Total SCCPs (ng/g)	Total MCCPs (ng/g)	Total LCCPs (ng/g)	Total CPs (ng/g)
	2008	146 NJ	135 NJ	6.78 UJ	283 NJ
	2000	156 NJ	153 NJ	10.6 UJ	307 NJ
	1994	66.7 NJ	72.4 NJ	10.9 UJ	1190 U
	1987	16.5 UJ	11 UJ	5.51 UJ	16.5 UJ
Bead Lake	1969	44.9 NJ	43.9 NJ	3.47 UJ	923 U
	1957	63.5 NJ	55.3 NJ	3.53 UJ	119 NJ
	1949	46.6 NJ	47.9 NJ	4.37 UJ	918 U
	1937	122 NJ	125 NJ	7.72 UJ	247 NJ
	< 1850	88.6 NJ	63.2 NJ	6.81 UJ	153 NJ
	2007	1580 U	1320 U	5.82 UJ	1320 U
	2000	1770 U	1480 U	6.32 UJ	1480 U
	1983	1470 U	1230 U	9.59 UJ	1230 U
	1968	1490 U	1240 U	23.9 NJ	1240 U
Lake Goodwin	1952	1620 U	1350 U	51.3 NJ	1350 U
	1922	1720 U	12.8 UJ	37.4 NJ	80.1 U
	1884	1600 U	1330 U	39.8 NJ	1330 U
	< 1850	112 NJ	112 NJ	93 NJ	320 NJ
	< 1850	119 NJ	118 NJ	99.2 NJ	337 NJ
	2013	1590 U	1320 U	12.2 UJ	1320 U
	2010	1510 U	1260 U	10.1 UJ	1260 U
	2006	1390 U	1160 U	15.1 NJ	1160 U
	1991	166 NJ	923 U	15.1 NJ	923 U
Mason Lake	1975	232 NJ	157 NJ	12.1 NJ	403 NJ
	1956	273 NJ	226 NJ	8.89 NJ	508 NJ
	1932	101 NJ	671 U	5.34 UJ	671 U
	1903	137 NJ	122 NJ	5.52 UJ	261 NJ
	< 1850	835 U	696 U	2.9 UJ	696 U

 Table 5. Chlorinated Paraffins Results in 2014 Sediment Cores.

studies (NTP, 2011). MCCPs and LCCPs are also persistent and bioaccumulative, but appear to have lower toxicity due to their lower solubility. However, their toxicity is not as well researched as SCCPs (EPA, 2009).

SCCPs have been found in many environmental media (Tomy et al., 1998 and Bayen et al., 2006), as well as in remote sediments attributed to long-range atmospheric transport (Tomy et al., 1999). The greatest mode of release to the environment is thought to be from manufacturing and lubricant applications, primarily via metal-working activities (EPA, 2009). Washington has no major CP manufacturing facilities. Releases in the state are likely from use of CP-containing products.

In this study, nine sediment horizons per core were analyzed for CPs. All detected results were qualified "NJ" to indicate that the result found was less than the concentration necessary for positive determination based on the chromatographic pattern.

Seventy-four percent of samples contained one or more of the CP mixtures. Bead Lake samples had the highest detection frequency of SCCPs and MCCPs among the three lakes. However, all Mason Lake samples and the upper seven samples of the Goodwin Lake core were analyzed in a laboratory batch affected by lab blank contamination of SCCPs, MCCPs, and T-CPs.

In samples not affected by method blank contamination, SCCPs and MCCPs were more frequently detected and present at higher levels than LCCPs.

NJ = Analyte was tentatively identified and the report result is an estimate. U = Analyte was not detected at or above the reported result. UJ = Analyte was not detected at or above the reported estimate.

Chlorinated Paraffins

In Bead Lake, estimated CP concentrations (and fluxes) ranged from <16.5 - 156 ng/g (<1.186 - 19.71 ug/m²/yr) for SCCPs and from <11 - 153 ng/g (<0.791 - 20.2 ug/m²/yr) for MCCPs. LCCPs were not detected in any of the Bead Lake samples. The maximum concentrations in Bead Lake were found in the sediment horizon corresponding to the year 2000. The lowest concentrations were measured in the sample dated 1987.

Interpretation of the Goodwin and Mason Lake profiles is hampered by lab blank contamination in the majority of the samples. All Goodwin Lake samples between the late 1800s and modern sediments had SCCP and MCCP concentrations that were less than ten times the lab blank level. The bottom two sediment samples, both dated older than 1850, were analyzed in a lab batch unaffected by contamination and contained SCCP and MCCPs concentrations between 112 - 119 ng/g. LCCPs were unaffected by lab contamination and were detected in samples dated 1968 and older, with the highest concentrations found in the pre-1850 sediments. Detections of CPs in sediments deposited before the 1930s–when production of the chemicals began–may be due to diffusion of the compounds through the sediment bed down to older sediments (Tomy et al., 1999), or difficulties encountered with laboratory analysis of the CPs.

In several Mason Lake samples, SCCPs and MCCPs were detected at greater than 10 times the lab blank concentration. Of these samples, SCCP concentrations ranged from 101 - 273 ng/g (flux range of $10.8 - 20.0 \text{ ug/m}^2/\text{yr}$), while three samples contained MCCPs in the range of $122 - 226 \text{ ng/g} (10.1 - 26.8 \text{ ug/m}^2/\text{yr})$. Peak concentrations of both SCCPs and MCCPs occurred in 1956. LCCPs were unaffected by lab blank contamination and detected in four samples at low levels (8.89 - 15.1 ng/g) between 1956 and 2006.

None of the study lakes displayed monotonic trends in CP concentrations. Lake sediment cores collected along a latitudinal gradient in Canada also displayed erratic profiles of CP concentrations but generally showed maximum levels in the 1980s and 1990s (Tomy et al., 1999). Surficial total CP concentrations found in the Canadian lakes ranged from 1.6 - 257 ng/g (fluxes of 0.45 - 147 ug/m²/yr), with lakes at the lower end of this range representing atmospheric inputs and the lake with maximum CP values reflecting urban/industrial sources. A sediment core collected in Switzerland contained a total CP concentration profile that closely matched trends in global CP production values, with increasing concentrations between the 1950s and 1990s and a peak concentration of 58 ng/g in 2000 (Iozza et al., 2008).



Figure 6. SCCP, MCCP, and LCCP Concentrations (ng/g) in 2014 Sediment Cores. Colored bars represent detected "NJ" values. White bars represent non-detected "UJ" values. Non-detects due to method blank contamination were excluded from the graph; samples containing one or more non-detects due to method blank contamination are indicated by an asterisk (*). All data can be found in Table 5.

□ 'UJ' values

■ SCCPs ■ MCCPs ■ LCCPs

Conclusions

Ecology collected sediment cores from Bead Lake, Lake Goodwin, and Mason Lake in 2014 for analysis of lead, mercury, and chlorinated paraffins (CPs). Contaminant deposition profiles showed the following trends:

- Profiles of sediment accumulation rates in the three lakes varied, with the maximum sediment accumulation rate occurring in 1934 for Bead Lake, 1968 in Lake Goodwin, and 2013 for Mason Lake.
- Lead concentrations consistently increased over the 20th century at all three lakes, with peak concentrations occurring in 2000, 2007, and 1991 at Bead, Goodwin, and Mason Lakes, respectively. Since the early 2000s, lead concentrations declined to a modern concentration of 43 ug/g in Bead Lake, increased to 114 ug/g in Lake Goodwin, and remained stable at 50 ug/g in Mason Lake.
- Mercury concentration profiles were similar to those of lead in the three lakes, with concentrations rising over the 20th century and peak years of 1994, 2007, and 1991/2010 for Bead, Goodwin, and Mason, respectively. In recent trends, Bead Lake concentrations declined to 69 ng/g, Lake Goodwin sediments continued to increase (to 199 ng/g), and Mason Lake remained stable at 135 ng/g.
- Lead and mercury fluxes showed generally similar patterns to concentrations in Bead and Goodwin Lakes, suggesting a true decline of inputs in Bead Lake and a true increase of the metals in Lake Goodwin, in recent sediments. While concentrations remained stable in recent sediments from Mason Lake, lead and mercury fluxes increased from the mid-1970s through the top of the core, suggesting lead and mercury deposition may continue to increase in Mason Lake.
- Laboratory analysis of CPs in sediment core samples encountered analytical difficulties which hampered interpretation of contaminant profiles. Seventy-four percent of samples contained one or more detections of the CPs analyzed (SCCP, MCCP, and LCCP). All detected samples were qualified as tentatively identified, because concentrations were lower than that necessary for confident determination of CPs.
- Of samples not affected by laboratory blank contamination, SCCPs and MCCPs were detected more frequently, and at higher levels, than LCCPs. Samples generally contained similar concentrations of SCCPs and MCCPs.
- None of the lake profiles displayed monotonic trends in CP concentrations. Maximum concentrations were found in the year 2000 at Bead Lake, in pre-industrial sediments in Lake Goodwin, and in 1956 for Mason Lake. Mason Lake samples contained the highest levels of CPs.

Recommendations

• The SCCP and MCCP concentrations reported by this study warrant further investigation into the levels and sources of chlorinated paraffins in Washington's aquatic systems. However, due to analytical difficulties encountered in this study, more research needs to be made to find a developed method with reporting limits appropriate for ambient environmental concentrations. The minimum concentration for positive identification used in the analysis for this study was higher than would be expected of non-point source sediment concentrations.

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This report is available on the Department of Ecology's website at https://fortress.wa.gov/ecy/publications/SummaryPages/1503027.html

Data for this project are available at Ecology's Environmental Information Management (EIM) website <u>www.ecy.wa.gov/eim/index.htm</u>. Search Study ID, SEDCORE14.

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