PBT Chemical Trends Determined from Age-Dated Lake Sediment Cores, 2013 Results

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2013 Highlighted Findings

- Lead and mercury showed decreasing trends since peak levels (1970s-90s) at all three lakes.
- HBCD increased at all three lakes from the 1960s-70s through recently deposited sedi-ments.
- HBCD levels in Sawyer and Kitsap Lakes were high compared to levels measured in other parts of the United States.

Why Does it Matter?

PBTs are a concern because they remain in the environment for a long time, increase in concentration up the food chain, and have toxic effects on fish, wildlife, and humans.

Overview

The Washington State Department of Ecology's (Ecology's) Persistent, Bioaccumulative, and Toxic chemical (PBT) 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 from the deepest area of three lakes per year and age-dated in order to reconstruct contaminant deposition profiles.

In 2013, Ecology analyzed lead, mercury, and hexabromocyclododecane (HBCD) in sediment cores collected from Lake Cavanaugh, Kitsap Lake, and Lake Sawyer (Figure 1). HBCD is a flame retardant primarily used in building material insulation.

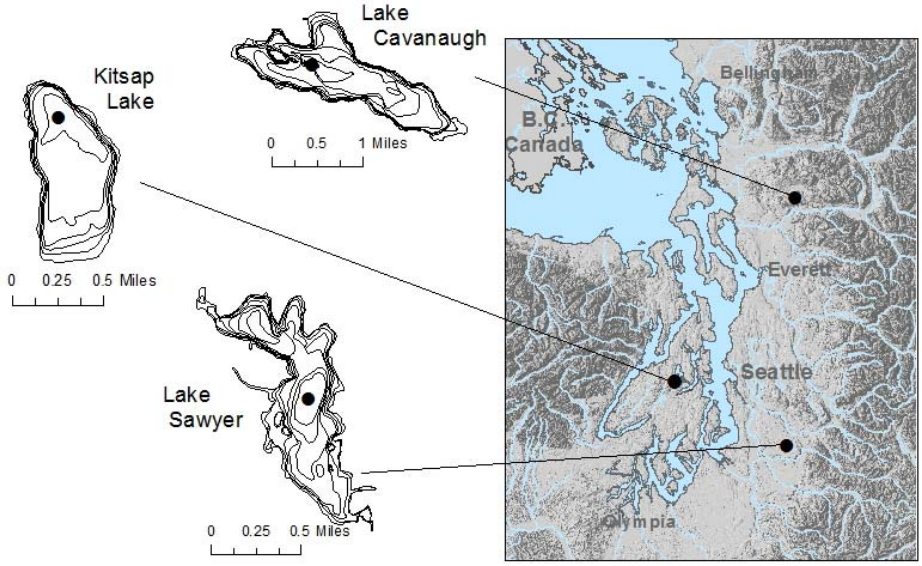

Figure 1. Lake Sediment Core Locations, 2013.

Ecology develops chemical action plans (CAPs) to evaluate uses and releases of PBTs in Washington State and make recommendations on actions to protect human health and the environment. Information from environmental monitoring efforts, such as this sediment coring program, help guide development of future CAPs and track progress on existing 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

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Methods and Data Quality

This study followed a Quality Assurance Project Plan (QAPP) and QAPP addendum (Coots, 2006; Mathieu and Bookter, 2013). Sediment cores measuring 35-45 cm deep were collected using a Wildco© box corer according to Ecology's standard operating procedures for sediment core collection (Furl and Meredith, 2008b). Surface sediments were collected with a standard ponar for analysis of grain size only.

Table 1 lists the parameters analyzed for this study. Sediment core horizons were selected for analysis using a weighted selection process described in the QAPP and QAPP addendum. A higher density of horizons near the top were selected for testing, while horizons were spaced farther apart moving down the core. Total organic carbon (TOC) and grain size data were used to help interpret metals and HBCD concentrations and are not presented in this report. All data, including TOC and grain size, are available for download in Ecology's EIM database (http://www.ecy.wa.gov/eim/).

Data generally met measurement quality objectives (MQOs) with a few exceptions. One surrogate recovery for HBCD was <40%. The laboratory duplicate for this sample had an acceptable surrogate recovery, and both were below detection limits. One laboratory duplicate for ^{210}Pb and one mercury matrix spike recovery were outside of MQOs; the associated source samples were qualified as estimates.

Gamma-HBCD was detected in three sediment horizons dated older than 1960 (Lakes Kitsap and Sawyer) at concentrations near the reporting limit (0.2 - 0.9 ng/g). Possible reasons for detections prior to commercial availability of HBCD include the potential for pore-water transfer down the core, polychaete mobilization, or background contamination. Eight other samples prior to 1960 were analyzed for HBCDs and did not contain target analytes.

Table 1. Sample Preparation and Analysis.

Analyte	No. of Samples	Method Description	Analytical Method
^{210}Pb	45	Alpha Spectroscopy	EML Po-2 Modified
T-Pb	45	ICP-MS	EPA 200.8
Hg	45	CVAA	EPA 245.5
HBCDs	27	LC-MS/MS	AXYS Method MLA-070 Rev 02
TOC	30	Acidification and CO_2 Measurement	PSEP-EPA, 1986
Grain Size	3	Sieve-Pipette	PSEP-EPA, 1986

ICP: inductively coupled plasma, MS = mass spectrometry, CVAA: cold vapor atomic absorption, LC = liquid chromatography, TOC: total organic carbon

Study Locations

Lake Cavanaugh lies in Skagit County. Located in a remote, rural setting with moderate-density residences along the shoreline, most of the watershed is forested with active logging operations. Surface water enters the lake through several ephemeral streams, and water exits through Lake Creek. Basin geology consists of consolidated sedimentary, metamorphic, and volcanic rocks with underlying basin hills and an alluvium valley bottom (Bortleson, 1974).

Located 3 miles west of Bremerton in Kitsap County, suburban Kitsap Lake is adjacent to military property and represents a mixed use watershed composed mainly of undeveloped forest, residential, and rock quarry land uses. With a densely populated shoreline, surface water flows in from several storm drain outfalls and through a small stream at the south end of the lake. The outflow channel is through Kitsap Creek. Basin geology consists of glacial recessional outwash deposits and volcanic rock (Collings, 1973).

Lake Sawyer, located 1.5 miles northwest of Black Diamond, is the fourth largest natural lake in King County. The lake's watershed consists largely of forested areas including an extensive wetland which historically received wastewater treatment plant effluent. Lake Sawyer is used extensively for several recreational activities and its shoreline is more than 85% developed by single family residential homes. Two inflow streams feed into the southeastern end of the lake and Covington Creek carries outflow. Basin geology consists of glacial drift deposited primarily as recessional outwash (McConnell, 1976).

Table 2. Physical Descriptions of 2013 Study Lakes.

Waterbody	Max Depth (ft)	Mean Depth (ft)	Surface Area (ac)	Drainage Area (ac)	DA:SA	Volume (ac ft)	Elevation (ft)
Lake Cavanaugh	80	44	804	4,710	5.9	35,800	1,010
Kitsap Lake	29	18	248	1,750	7	4,480	127
Lake Sawyer	58	25	310	8,320	26.8	7,700	512

DA:SA = drainage area to surface area ratio

Core Dating

Dates were calculated for the three cores using ^{210}Pb values and the constant rate of supply (CRS) model (Appleby and Oldfield, 1978). Percent solids were used to calculate dry mass. Supported ^{210}Pb was estimated as the average activity present at deep intervals where it appeared to no longer decline (i.e., background). Cavanaugh, Kitsap, and Sawyer Lakes average supported ^{210}Pb levels were estimated as 0.30, 0.18, and 0.25 pCi/g.

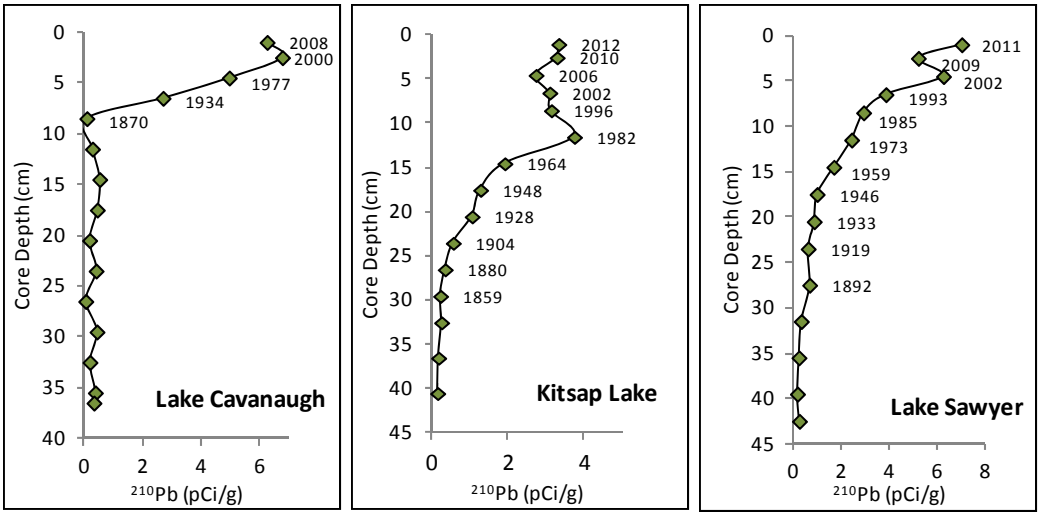

Figure 2. ^{210}Pb Activity Plotted Against Sediment Core Depth.
Dates assigned to sediment layer midpoint using the CRS model are included in graph.

Yearly unsupported ^{210}Pb fluxes for the lakes ranged from 0.16 - 0.19 pCi/cm^2/yr, which is within the range of values estimated for other sediment cores in Washington State (Mathieu, 2013; Mathieu and Friese, 2012; Furl and Roberts, 2011). These values are also close to estimated fluxes calculated using atmospheric ^{210}Pb deposition measured in Washington State (Nevissi, 1985; Lamborg et al., 2013).

Sediment Accumulation Rates

Lake Cavanaugh sedimentation rates were very low throughout the entire core, ranging from 0.006 - 0.023 g/cm^2/yr. Rates remained stable until the 1970s when modest increases started and continued through the top of the core to a present day rate of 0.023 g/cm^2/yr. Because of the low accumulation of sediments in this lake, the majority of the horizons analyzed for metals and HBCD were older than 1900. With the sample size of post-1900 sediment horizons so low (n = 4), care should be taken when interpreting the contaminant profile results of Lake Cavanaugh.

Sedimentation rates in Kitsap Lake ranged from 0.005 - 0.054 g/cm^2/yr. Slight increases in sedimentation began in the early 1900s, and rose more sharply between 1980 and 2006. Sedimentation remained steady in the three most recent layers at 0.05 g/cm^2/yr (2006 - 2012).

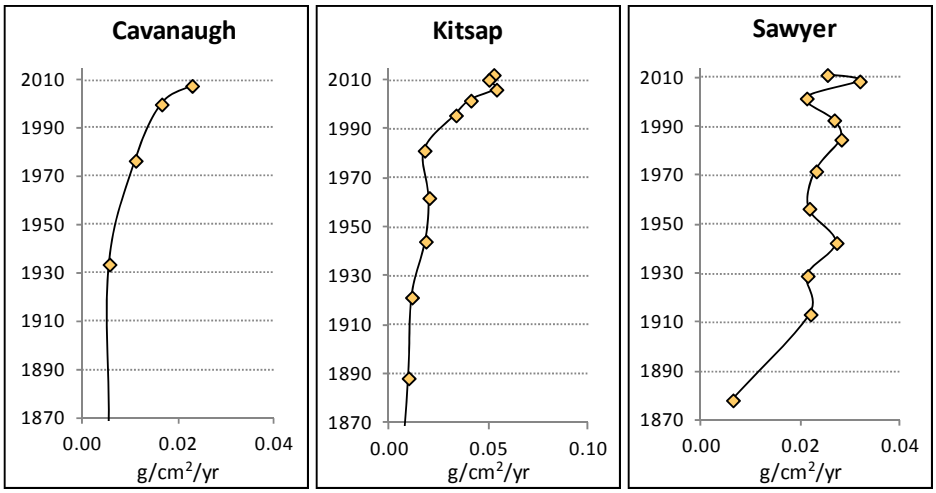

Figure 3. Estimated Sedimentation Rates (g/cm^2/yr).

Lake Sawyer sedimentation rates increased from 0.006 to 0.022 g/cm^2/yr between 1880 and 1910, during a period of increased coal mining activity in the watershed. Since the early 1910s, sedimentation rates varied but stayed within a constant range of 0.02 - 0.03 g/cm^2/yr.

Lead

Profiles

Lead concentrations in Lake Cavanaugh sediments remained near baseline levels (9 ug/g) until the late 19th century when concentrations increased to 31 ug/g. By the 1930s those levels were elevated to 139 ug/g. Peak lead concentrations occurred in 1977 at 290 ug/g, consistent with peak leaded gasoline use, and modestly declined into the late 2000s.

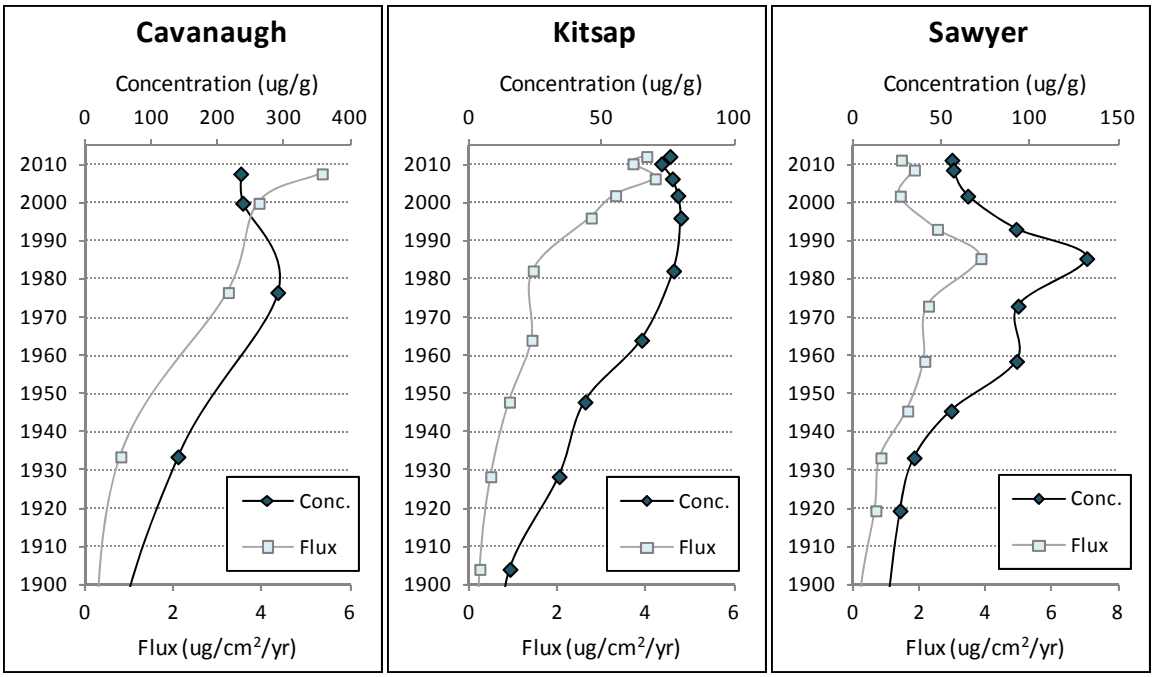

Figure 4. Lead Concentration (ug/g) and Flux (ug/cm^2/yr) Profiles in Sediment Cores.

Kitsap Lake lead levels were relatively low throughout the core. Lead increased steadily from baseline levels in the early 1900s (6 ug/g) to a maximum of 79.7 ug/g in 1996. Concentrations varied little from 1982 to 2012, remaining between 72.4 and 79.7 ug/g over that time period.

Lake Sawyer lead levels increased throughout the 1900s until a a peak in 1985 of 132 ug/g. Concentrations then decreased steadily through the 1990s and 2000s, reaching 55.5 ug/g in modern sediments.

Enrichment

Enrichment factors (EFs) were calculated as 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).

Lake Cavanaugh contained the highest lead concentrations of the three lakes (Table 3). At its peak, levels were 33 times higher than baseline conditions. EFs were still elevated at 27 in modern sediments. The very low sedimentation rates in the lake may partly explain the high concentrations found, due to a lack of dilution that occurs with high sedimentation.

Table 3. Lead Concentrations and Enrichment Results.

Lake	Range (ug/g)	Baseline (ug/g)	Peak Enrichment	Modern Enrichment
Cavanaugh	6.96 - 290	8.7	33 (1977)	27 (2008)
Kitsap	2.31 - 79.7	5.7	14 (1996)	13 (2012)
Sawyer	2.58 - 132	3.6	36 (1985)	15 (2011)

Kitsap Lake had the lowest concentrations, with EFs remaining between 13-14 throughout the second half of the 20th century.

Lake Sawyer sediments were highly enriched in the mid-1980s (EF = 36), but EF levels were lower throughout the rest of the core.

Mercury

Profiles

Because of the low sedimentation rates in Lake Cavanaugh, only four samples analyzed for mercury were deposited after 1900. However, the core appears to show a typical historical deposition pattern of increasing mercury levels through the first three quarters of the 20th century and declining concentrations over the last three decades. Lake Cavanaugh mercury concentrations were elevated above baseline by 1930 and reached peak levels in 1977 (882 ng/g). Concentrations declined to 519 ng/g in the late 2000s.

Mercury levels started rising above baseline in Kitsap Lake sediments in the late 1800s. Mercury consistently increased over the first half of the 20th century until the mid-1960s. Levels have remained fairly constant since the 1960s, with the exception of a modest decrease from the peak of 207 ng/g in 2002 through the most recent layer, which measured 182 ng/g (2012).

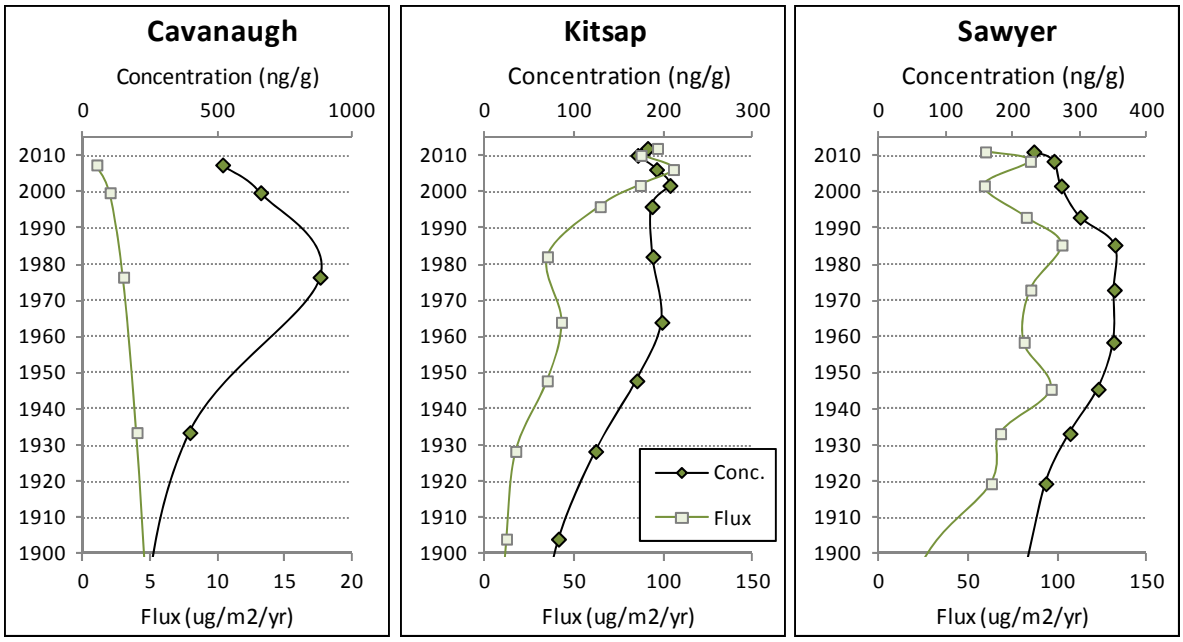

Figure 5. Mercury Concentration (ng/g) and Flux (ug/m^2/yr) Profiles in Sediment Cores.

The Lake Sawyer mercury concentration profile showed mercury levels starting to increase in the late 1880s, and then a continued rise until the 1960s. Levels remained near 350 ng/g throughout the '60s, '70s and '80s, followed by a decrease to a level of 231 n/g in modern sediments.

Enrichment

Mercury concentrations in the Lake Cavanaugh samples were surprisingly high, given the remote location of this lake. However, baseline levels were also fairly high making EFs moderate and similar to other cores. Comparable concentrations were recorded in a sediment core collected from Lake Stevens, located 21 miles south of Lake Cavanaugh (Mathieu, 2013). As with the elevated lead profile, mercury concentrations may be higher in Lake Cavanaugh as a result of non-dilution of sediments, given the very low sedimentation rates measured. Mercury concentrations in the Kitsap and Sawyer cores were much lower than Cavanaugh levels, but modern EFs were similar.

Table 4. Mercury Concentration and Enrichment Results.

Lake	Range (ng/g)	Baseline (ng/g)	Peak Enrichment	Modern Enrichment
Cavanaugh	144 - 882	169	5.2 (1977)	3.1 (2008)
Kitsap	41 - 207	46.2	4.5 (2002)	3.9 (2012)
Sawyer	89 - 352	94.8	3.7 (1985)	2.4 (2011)

Hexabromocyclododecane (HBCD)

Hexabromocyclododecane (HBCD) is a brominated flame retardant used primarily in extruded (XPS) and expanded (EPS) polystyrene for building insulation, as well as in furniture textiles, automotive upholstery, and other consumer products such as electronics. HBCD has been classified as a PBT by regulatory agencies (WAC 173-333-320; EPA, 2010). It exhibits high aquatic toxicity and is a human health concern for reproductive, developmental, and neurological effects, based on animal studies (EPA, 2010). HBCD can be transported long distances and has been found in many different environmental media throughout the world (Covaci et al., 2006).

HBCD was found in freshwater fish collected from four waterbodies in Washington State in 2011 (Johnson and Friese, 2012). Concentrations in fish tissue reported by this study were within the range of values recorded worldwide for ambient freshwater fish (Covaci et al., 2006 and references therein), with the exception of one higher concentration (1,120 ng/kg) in a largescale sucker composite from Lake Washington.

HBCD refers to a technical mixture of diastereomers. Commercial mixtures are primarily comprised of gamma-(75-89%), alpha-(10-13%), and beta-(1-12%) HBCD isomers (Covaci et al., 2006). All three isomers were analyzed in sediment core samples for this study.

Table 5. Statistical Summary of HBCDs in 2013 Sediment Cores.

Lake	Summary statistic	alpha-HBCD	beta-HBCD	gamma-HBCD	total HBCD
Cavanaugh	n	9	9	9	9
	#>RL	3	2	3	3
	%>RL	33%	22%	33%	33%
	min (ng/g)	0.1 U	0.1 U	0.1 U	0.1 U
	max (ng/g)	1.24	0.464	7.13	8.64
	median (ng/g)	0.1 U	0.1 U	0.1 U	0.1 U
	mean* (ng/g)	0.815	0.463	4.68	5.80
Kitsap	n	9	9	9	9
	#>RL	7	6	9	9
	%>RL	78%	67%	100%	100%
	min (ng/g)	0.1 U	0.1 U	0.159	0.159
	max (ng/g)	1.690	0.524	15.5	17.7
	median (ng/g)	0.659	0.301	6.60	7.56
	mean* (ng/g)	0.789	0.346	6.66	7.50
Sawyer	n	9	9	9	9
	#>RL	5	4	7	7
	%>RL	56%	44%	78%	78%
	min (ng/g)	0.1 U	0.1 U	0.2 U	0.2 U
	max (ng/g)	2.52	0.946	26.6	30.0
	median (ng/g)	0.272	0.163	2.55	2.82
	mean* (ng/g)	1.42	0.67	11.3	12.7

*mean of detected values

RL = Reporting Limit; U = not detected at reporting limit shown

HBCD Results

Nine sediment samples per core were analyzed for HBCDs. At least one HBCD isomer was detected in 70% of the 27 sediment samples analyzed. Summary statistics are presented in Table 5.

HBCDs were detected most frequently in the Kitsap Lake core, with all samples analyzed containing at least one HBCD isomer. HBCDs were detected in 78% of Lake Sawyer samples, and Cavanaugh had the fewest detections, at 33%.

Gamma-HBCD was detected in the highest amounts, ranging from 78% - 100% of the total HBCD concentration. The diasteromeric make-up of samples was generally consistent with commercial mixture percentages, with median percent contributions of 89%, 8%, and 3% for gamma, alpha, and beta, respectively. Previous studies have also found diastereoisomer profiles in sediments to be similar to commercial formulations (Harrad et al., 2009; Morris et al., 2004).

EFs were not calculated for HBCD because no known natural source of HBCD exists and ambient background levels have not been established.

HBCD

Temporal Trends

HBCDs were detected in all sediment samples dated later than 1960, when the chemical became commercially available (UNEP, 2010). Total HBCD (T-HBCD, sum of the three diastereomers) concentrations increased over the second half of the 20th century in all three sediment cores beginning in the 1960s and 1970s. Sharp increases in T-HBCD began in the 1980s, consistent with the beginning of the use of HBCD in insulation boards (UNEP, 2010).

HBCD first appeared in the late 1970s in the Lake Cavanaugh core, at low concentrations (0.95 ng/g). By 2000, T-HBCD levels had increased to a concentration of 7.8 ng/g. Between 2000 and 2008, concentrations appear to have leveled off, with levels just slightly higher in the most recent sediment layer, at 8.6 ng/g.

T-HBCD was present in the Kitsap Lake core during the mid-1960s at low concentrations (2.4 ng/g). Levels more than doubled between the 1960s and 1980s, and then remained stable between the 1980s and 2002 (range = 7.2 - 8.6 ng/g). Concentrations spiked between 2002 and 2006, and then again from 2010 to 2012. The peak concentration found in the top-most layer was fairly elevated at 17.7 ng/g.

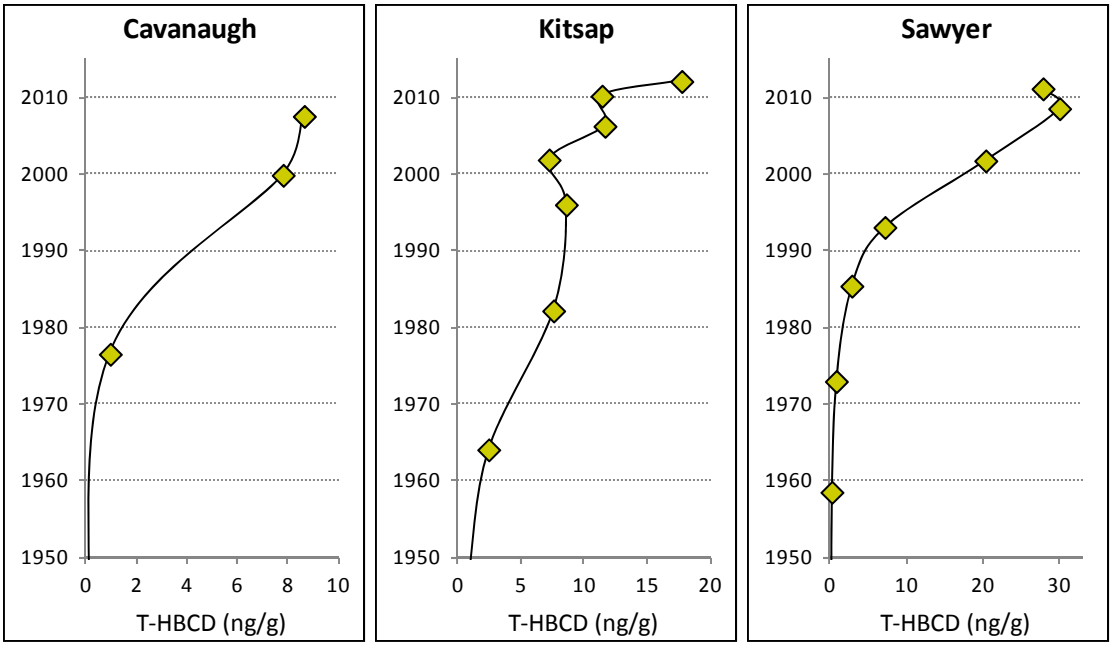

Figure 6. Concentration Profiles of Total HBCD (ng/g) in 2013 Sediment Cores.

T-HBCD concentrations were low through the mid-1980s at Lake Sawyer (< 3 ng/g), and then steadily increased until a peak in 2009 at 30 ng/g. A slightly lower concentration was measured in the most recent layer. Lake Sawyer historically received wastewater treatment plant effluent that discharged to a connected wetland between 1981 and 1992. HBCD concentrations increased during this period in the Lake Sawyer core; however, the sharpest increases were found between the 1990s and late 2000s.

T-HBCD levels in the upper sediments (0-2 cm) of Kitsap and Sawyer Lakes were relatively high compared to data from other freshwater sites lacking point sources. Previous research has found T-HBCD levels of less than 4 ng/g in North American waterbodies (Yang et al., 2012; Law et al., 2006) and less than 10 ng/g worldwide (Covaci et al., 2006 and references therein) in ambient freshwater surface sediments. In contrast, T-HBCD concentrations in the surface sediments of Kitsap and Sawyer Lakes were 17.7 and 27.8 ng/g. The reason for high HBCD levels in the cores studied here is unclear, as neither watershed is located near point sources. HBCD can enter the aquatic environment diffusely from releases to the soil during construction and demolition of XPS- or EPS-insulated buildings, with subsequent transfer to air or stormwater runoff (EC, 2011) and through wastewater treatment plant effluent and landfill emissions following the use or disposal of products containing HBCD (EPA, 2010). Kitsap and Sawyer Lakes had higher percentages of residential development in their watersheds than Cavanaugh, - 21% and 18%, respectively, compared to 8% for Cavanaugh. Atmospheric deposition may also play an important role in the lakes studied here, as other researchers have documented particularly high loading of HBCD compared to other brominated compounds via wet deposition in rural areas, possibly due to its higher vapor pressure (Robson et al., 2013; Tian et al., 2011).

Conclusions

Ecology analyzed lead, mercury, and HBCD in sediment cores collected from Lake Cavanaugh, Kitsap Lake, and Lake Sawyer in 2013. Contaminant deposition profiles showed the following trends:

- Profiles of sediment accumulation in Cavanaugh and Kitsap Lakes were similar, with increasing rates primarily in the last quarter of the 20th century, although Cavanaugh rates were very low. Lake Sawyer sediment accumulation increased rapidly in the late 1800s and reached the modern range of 0.02 - 0.03 g/cm^2/yr by the early 1900s.
- Lead concentrations increased from 1900 to 1980 in all three lake sediment cores, and then declined to present-day concentrations in Cavanaugh and Sawyer Lakes. Concentrations in Kitsap Lake remained fairly constant, between 72 and 80 ug/g, from 1980 to the most recent sediments.
- Mercury levels were highly elevated in Lake Cavanaugh, peaking in the mid-1970s and decreasing in recent sediments. Kitsap Lake had the lowest levels, with concentrations increasing until the 1960s and remaining stable until modest declines between 2000 and 2012. Lake Sawyer mercury levels peaked in 1985 and have declined since then.
- HBCD was detected in all sediment core samples dated later than 1960, when commercial production of HBCD began. Isomer patterns were similar to commercial mixtures, with gamma-HBCD being the dominant compound by concentration.
- Contaminant deposition profiles showed an increasing trend in HBCD concentrations beginning in the 1960s and 1970s through recently deposited sediments at all three lakes.
- HBCD concentrations in the upper sediments of Kitsap and Sawyer Lakes were higher than levels reported by other researchers for ambient freshwater sediments. The reason for the comparatively high levels is unclear, as Washington State has no known manufacturing sites or point sources. Kitsap and Sawyer Lake watersheds have a higher percentage of residential development, compared to the Lake Cavanaugh watershed.

Recommendations

- The increasing trends and high levels of HBCD in sediments reported here warrant further investigation into environmental levels of this contaminant in Washington. Only four waterbodies statewide have been tested for HBCD contamination of fish tissues. Additional spatial coverage of HBCD levels in sediments and fish tissue would support our understanding of HBCD in the aquatic environment of Washington and help prioritize Ecology efforts in reducing PBT chemicals.
- This sediment core monitoring program analyzes a different organic contaminant, in addition to lead and mercury, in sediment cores collected yearly. The rotating analyte list allows for monitoring of a broader suite of PBT chemicals. Because of the high levels of HBCD flame retardants found in this 2013 study, the authors recommend analyzing chlorinated paraffins - another class of flame retardants on the PBT List - in sediment cores in 2014. Chlorinated paraffins have been found in freshwater fish tissue in waterbodies of Washington (Johnson and Friese, 2012) and were reported in sediment cores in other parts of North America (Marvin et al., 2003). Trend data for flame retardants, such as chlorinated paraffins, are lacking in Washington State.

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This report is available on the Department of Ecology's website at https://fortress.wa.gov/ecy/publications/SummaryPages/1403036.html

Data for this project are available at Ecology's Environmental Information Management (EIM) website, www.ecy.wa.gov/eim/index.htm. Search Study ID, SEDCORE13.

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