PCB 11 and Possible Routes to its Environmental Presence in Relation to the Spokane River

Introduction

PCB 11 (3,3'-dichorobiphenyl) has been detected, amongst other PCB's in the Spokane River, Washington [1,2]. Chart 1 and 2 show a number of PCB congeners have been detected in the Spokane River [1] and in the Spokane County Regional Water Reclamation Facility [2].

Chart 1 – Selected PCB Congener Profile in the Spokane River

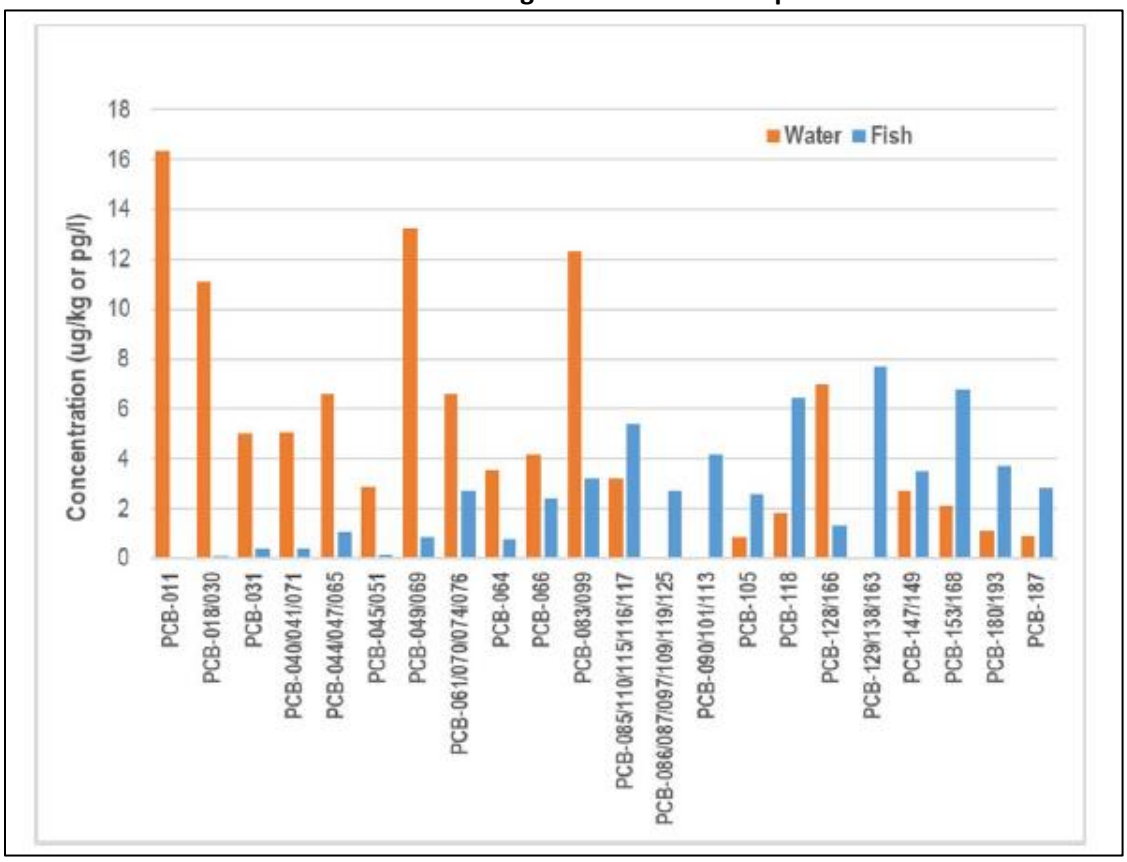
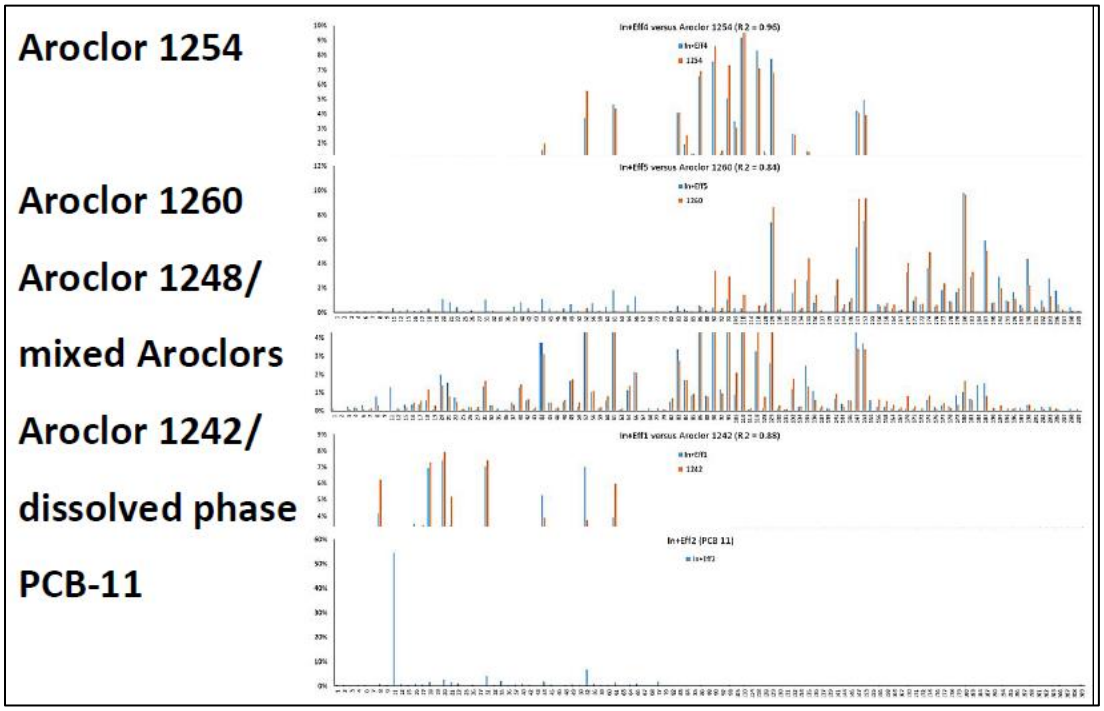


Chart 2 – Characterization of PCBs in the Spokane County Regional Water Reclamation Facility



It has been proposed that the sole source of PCB 11 in the Spokane River originates from printed matter containing diarylide pigments as these pigments may have inadvertent concentrations of PCB 11[3]. Specifically, the contention is that the PCB 11 Spokane River concentration originates from discarded/landfilled printed matter or from paper recycling operations discharging to the Spokane River. Rodenberg et al [3] considered all likely sources of PCB 11 in the environment and concluded that diarylide pigments were the most likely or only possible source of this PCB.

It is known that PCB 11 is an inadvertent (unintended) byproduct from diarylide pigment manufacturing One potential source of this substance in the Spokane River is from the effluent from Inland Empire Paper recycling facility located in Millwood, WA. However, the presence of PCB 11 in wastewater in the effluent from the sewer treatment facilities and stormwater in concentrations exceeding those found in the effluent from Inland Empire Paper provides a strong indication that sources of PCB 11 exists. The purpose of this paper is to examine the other possible sources of PCB 11.

A review of the literature (see references) has found that PCBs are degraded by anerobic biodegradation, photolysis and incineration. Most of these degradation routes follow a similar path, namely reductive dechlorination of higher level chlorobiphenyls to lower level chlorobiphenyls.

<u>Incineration</u>

Incineration of PCBs have been reported to generate high levels of monochloro and dichlorobiphenyls. Rodenberg et all [3] reported from the literature the following:

> "**Municipal solid waste incinerators (MSWIs) are another potential environmental source of non-Aroclor PCBs, including PCBs 11** and 209**, which may be released either by direct emission from PCB-containing combustion materials or through formation from carbon and chlorine precursors**. Traces of PCB 11 were measured in the stack gas from MSWIs in South Korea, although the contribution to total PCBs was minimal [4]. **Typical Swedish municipal waste was found to generate flue gases and ashes containing PCB 11 as high as 7%** of total PCBs during combustion between 200 and 450 °C in a laboratory-scale waste incinerator [5]. **The flue gases were dominated by mono- and dichlorinated PCBs**. **PCB 11 was among the most abundant PCBs, a feature associated with favoured formation** of meta and para substituted congeners in such situations. Kim et al. [6] investigated PCB emissions from Japanese waste incinerators with regard to the types of incinerator, combustion temperatures (from 740 to 920 °C), and the nature of waste materials. While **PCB 11 was detected in all flue gas samples, significantly higher concentrations**, as well as certain dioxin-like PCBs, were measured in two of the eight samples, in these cases considered to be due to lower combustion temperatures or to waste plastic in the combustion materials. Ogura et al. [7] estimated that incineration is roughly equal in importance to legacy sources as a source of dioxin-like PCBs in Japan, but they did not specifically consider PCB 11."

Whilst Rodenberg et al [3] considered incineration as a source of PCB 11 it was concluded that:

> "incineration is far more common in Japan than in the United States. Japan incinerates 78% of its garbage [8], versus just 12% in the United States [9]. Japan is home to 1500 incinerators which combust 38 million t of garbage annually, while 29 million t is incinerated in the much larger area of the United States. **Thus, while incineration may be a significant source of PCBs, including PCB 11**, in some countries, it is unlikely to be important in the United States, where high levels of PCB 11 have been measured."

The conclusion that incineration is not a likely source of PCB 11 within the USA has not been supported by any studies. It appears that no studies on the generation of PCBs from incinerators within Spokane area, Washington State, or the USA have been conducted.

Recommendation – it is highly recommended that the City and County of Spokane take measurements for PCB congeners within flue gases and ash of incinerators within the district. Understanding the presence and concentration of PCB-11 could explain why PCB-11 content increases in the Spokane County Regional Water Reclamation Facility during storm events being washed down from the atmosphere.

<u>Photochemical Dechlorination/Photolysis</u>

Photolysis is a known method for the degradation of PCBs. Whilst it has been reported that photochemical breakdown in water is lower than in solvents, there are numerous examples in the literature where it is shown that photochemical breakdown of higher level PCBs to PCB-11 is possible.

In 1972, Hutzinger et al [10] reported a time study of 2,2',5,5'-tetrachlorobiphenyl (PCB 52) decomposition by irradiating in hexane and under aqueous conditions. It was shown that PCB 52 decomposes over time to lower level PCBs. It should be noted that PCB 52 is a potential source of 3,3'-dichlorobiphenyl (PCB 11). Gas chromatograms were ran demonstrating the breakdown of PCB 52 to

the lower level PCBs, but no attempts were made to characterize and identify these PCBs. Aroclor 1254, a previously produced commercial product, containing a mixture of PCBs was also irradiated. It was again demonstrated that the higher level PCBs within Aroclor 1254 slowly degraded over time, with lower level PCB congeners increasing. It was concluded that the photolysis of PCBs reveals a number of degradative reactions which occur on irradiation in sunlight and a number of laboratory conditions. Irradiation of PCB 52 in aqueous media indicated complex degradation pathways, including dichlorination, formation of polymers, carboxylic products as well as hydroxylation.

In 1973, Herring et al [11] reported an irradiation study of Aroclor 1254 in hexane, water and benzene. Gas chromatograms were completed pre and post irradiation, however as with Hutzinger et al [10], no attempts were made to characterize the breakdown products. Herring et al reported "in general, the PCBs degraded fastest in hexane, then water, and slowest in benzene. The fact some peaks increased in size suggests that the more highly chlorinated PCBs were being dechlorinated to form PCBs with lower molecular weights and shorter retention times. It is also quite evident from the variations between each peak that the PCBs do not all degrade at the same rate". This conclusion is important when assessing the PCB content in the Spokane river and water treatment plant and the possible increase in PCB 11 content relative to other congeners. There is evidence in the literature (presented later on) that suggests PCB 11 is a relatively more stable PCB structure compared to other PCB congeners. This is due to the chlorine atoms being positioned in the meta positions within both rings. This study is also important in that it demonstrated that photochemical degradation to lower level PCBs occurs in aqueous conditions.

In 1974, Ruzo et al [12] studied a variety of tetrachlorobiphenyl congeners and their degradation products under UV irradiation in cyclohexane. Their study included 3,3',4,4'-tetrachlorobiphenyl (PCB 77), 2,2'4,4'-tetrachlorobiphenyl (PCB 47), 3,3',5,5'-tetrachlorobiphenyl (PCB 80), 2,2',3,3'-tetrachlorobiphenyl (PCB 40), 2,2',5,5'-tetrachlorobiphenyl (PCB 52) and 2,2',6,6'-tetrachlorobiphenyl (PCB 54). It was found that PCB 40 photodegraded to 2,3,3'-trichlorobiphenyl (PCB 20), 2,2',3-trichlorobiphenyl (PCB 16) and PCB 11 as the major products. PCB 52 also produced PCB 11, photodegrading to 2,5,3'-trichlorobiphenyl (PCB 26), PCB 11 and 3-chlorobiphenyl (PCB 2) as the major products. This was the first study that clearly identified (some) of the reductive dichlorination products from photolysis of higher level PCB congeners. Within this study both PCB 40 and PCB 52 were demonstrated to photodegrade to PCB 11. It should be noted that PCB 40 has been reported to be present at relatively high levels in the Spokane River watershed [1] and both PCB 40 and 52 have been reported to be present in the Spokane County Regional Water Reclamation Facility [2]. PCB 40 and 52 are also known congeners to be present in commercialized Aroclor products.

In 1978, Bunce et al [13] reported "in both aqueous and organic media, PCBs react mainly by stepwise dichlorination (replacement of Cl by H)". It also cites information from the literature suggesting that "highly chlorinated PCBs are the most photolabile, at least in organic solvents, while the less chlorinated ones are more reactive metabolically". The paper presents information to suggest that lower level PCBs retard the photolysis rates of higher level PCBs. 4-chlorobiphenyl (PCB 3) slowed the degradation of PCB 52 amongst others. The paper still demonstrated the photodegradation of PCB 52 was possible, although the final degradation products were not identified.

In 1992, Harari et al [14] investigated the sensitized photolysis of Aroclor 1254, using phenothiazine (PT) as the sensitizer. In this study, Aroclor 1254 was completely dechlorinated within 1 hour to biphenyl (BP), in the presence of PT in alkaline 2-propanol. What is interesting however, is that Harari et al also reported that "photolysis of Aroclor 1254 in an alkaline 2-propanol solution by solar radiation alone for 20 h gave less than 25% dichlorination. No biphenyl or monochlorobiphenyls were detected. Instead products such

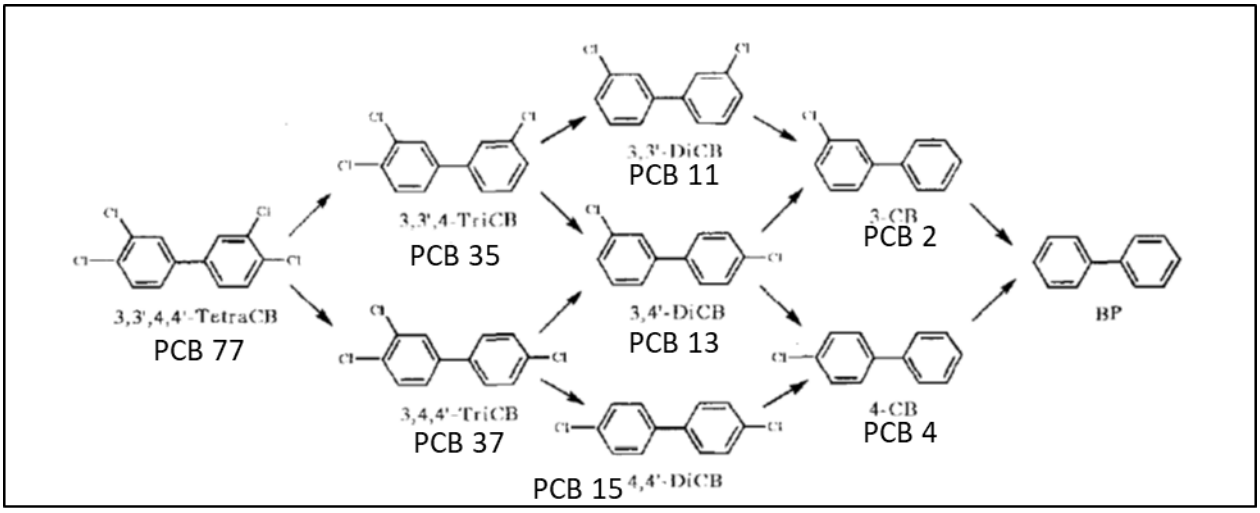
as 3,3'-dichlorobiphenyl (PCB 11), 2,3',5-trichlorobiphenyl (PCB 26), and PCB congeners with average chlorine content of 3-4 Cl atoms/PCB molecule were detected". The amount of PCB 11 etc detected was not reported, however it is evident that under non sensitized or catalyzed conditions Aroclor 1254 photodegrades to PCB 11. Aroclor 1254 has been reported to be a major PCB constituent in the Spokane County Regional Water Reclamation Facility [2], and the breakdown products PCB 11 and PCB 26 have been detected within the same facility.

In 1997, Yao et al [15] reported their research into the photochemical degradation of 3,3'4,4'-tetrachlorobiphenyl (PCB 77). In this paper Yao et al, clearly demonstrated the stepwise dichlorination of PCB 77 to its breakdown products as follows:

3,3'4,4'-tetrachlorobiphenyl (PCB 77) →
3,3'4-trichlorobiphenyl (PCB 35) + 3,4,4'-trichlorobiphenyl (PCB 37) →
3,3'-dichlorobiphenyl (PCB 11) + 3,4'-dichlorobiphenyl (PCB 13) +4,4'-dichlorobiphenyl (PCB 15) →
3-chlorobiphenyl (PCB 2) +4-chlorobiphenyl (PCB 3) → Biphenyl

This degradation is demonstration in Scheme 1

Scheme 1 – Photo-dechlorination of PCB 77

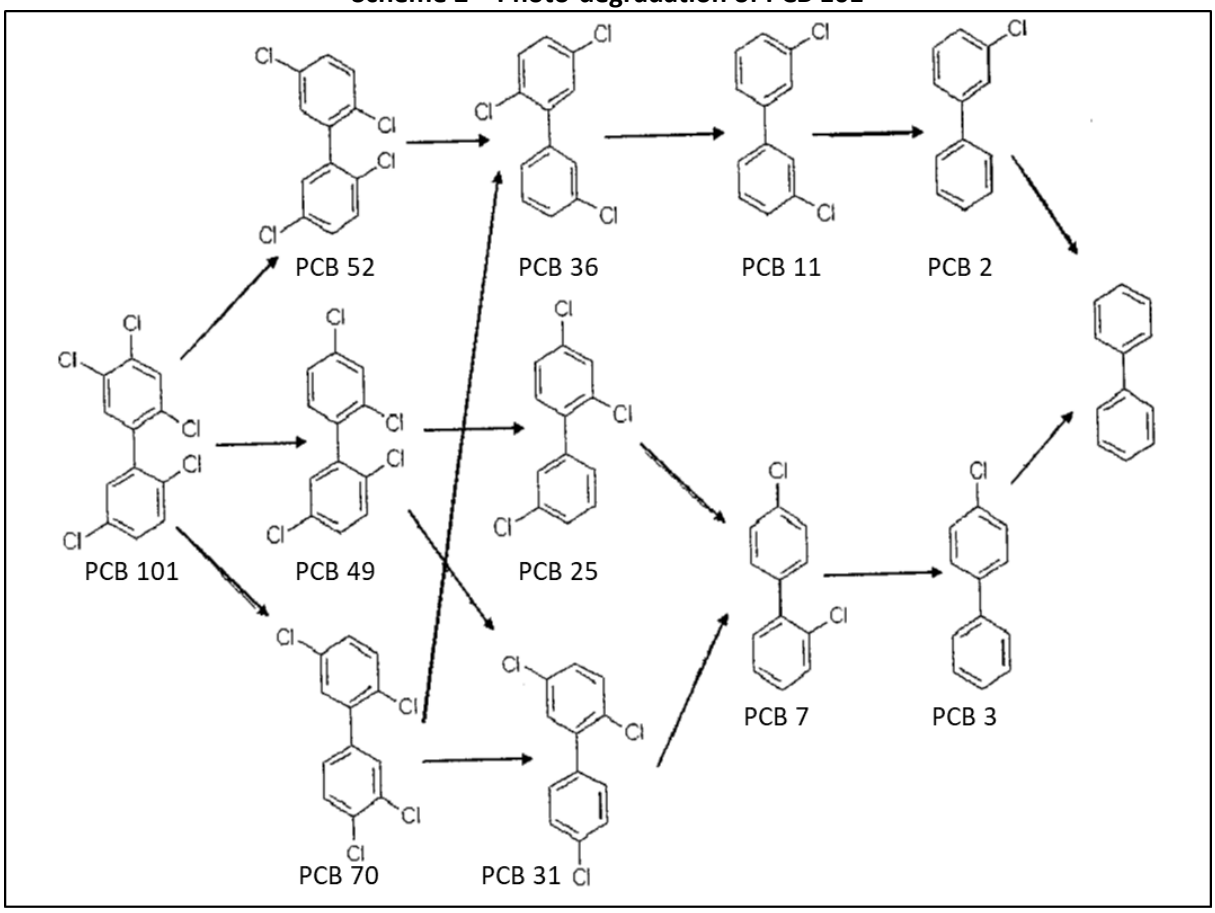


Yao et al's study showed that at a point in the irradiation process, PCB 11 was the highest concentration of all the congeners that formed, and did not completely disappear at the end of the experiment after 4 hours of irradiation time. 5% of PCB 11, 5% of PCB 2 and 13% of biphenyl remained. In addition to identifying the congeners formed, Yao et al also determined the rate of photolysis for each congener. Yao et al concluded that "it was found that the photolysis rates of congeners varied in the order of PCB 37=PCB 35>PCB 77>PCB 3>PCB 2>PCB 13>PCB 15>PCB 11. It was clear from their studies that PCB 11 was the slowest to photodechlorinate. This supports the hypothesis presented by Herring et al in 1973 that PCB 11 is a more stable PCB compared to other PCB congeners, at least to photodregadation.

Whilst PCB 77 is a minor contaminant in the Spokane River watershed [1] and the Spokane County Regional Water Reclamation Facility [2], the paper by Yao et al further supports that high level PCB congeners do photodegrade to lower level PCB congeners such as PCB 11 and that PCB 11 is a stable congener of the breakdown products.

In 2006, Wong et al [16] studied the photolysis of various PCB congeners, including 2,4,4'-trichlorobiphenyl (PCB 28), 2,2',5,5'-tetrachlorobiphenyl (PCB 52), 2,2',4,5,5'-pentachlorobiphenyl (PCB 101), 2,2',4,4',5,5'-hexachlorobiphenyl (PCB 153) and 2,2',3,4,4',5,5'-heptachlorobiphenyl (PCB 180). The authors identified that the intermediates produced were mainly less chlorinated congeners as reported in previous studies [15]. The congeners produced were further declorinated to lesser chlorinated congeners, and finally biphenyl. The degradation pathway for PCB 101 was provided where it can be seen that PCB 11 was an identified intermediate dechlorinated product (Scheme 2).

Scheme 2 – Photo-degradation of PCB 101



Reviewing the major PCBs present in the Spokane River Watershed and those identified in the Spokane County Regional Water Reclamation Facility a number of congeners have been identified with the potential to photochemically dechlorinate to PCB 11. These are presented in Table 1. The data for the PCBs present in the Spokane River Watershed are only the major ones identified [1]. The minor detections do not appear to be publicly available.

Table 1 – PCB Congeners identified in the Spokane River Watershed [1] and Spokane County Regional Water Reclamation Facility [2] with Potential to Photodechlorinate to PCB 11

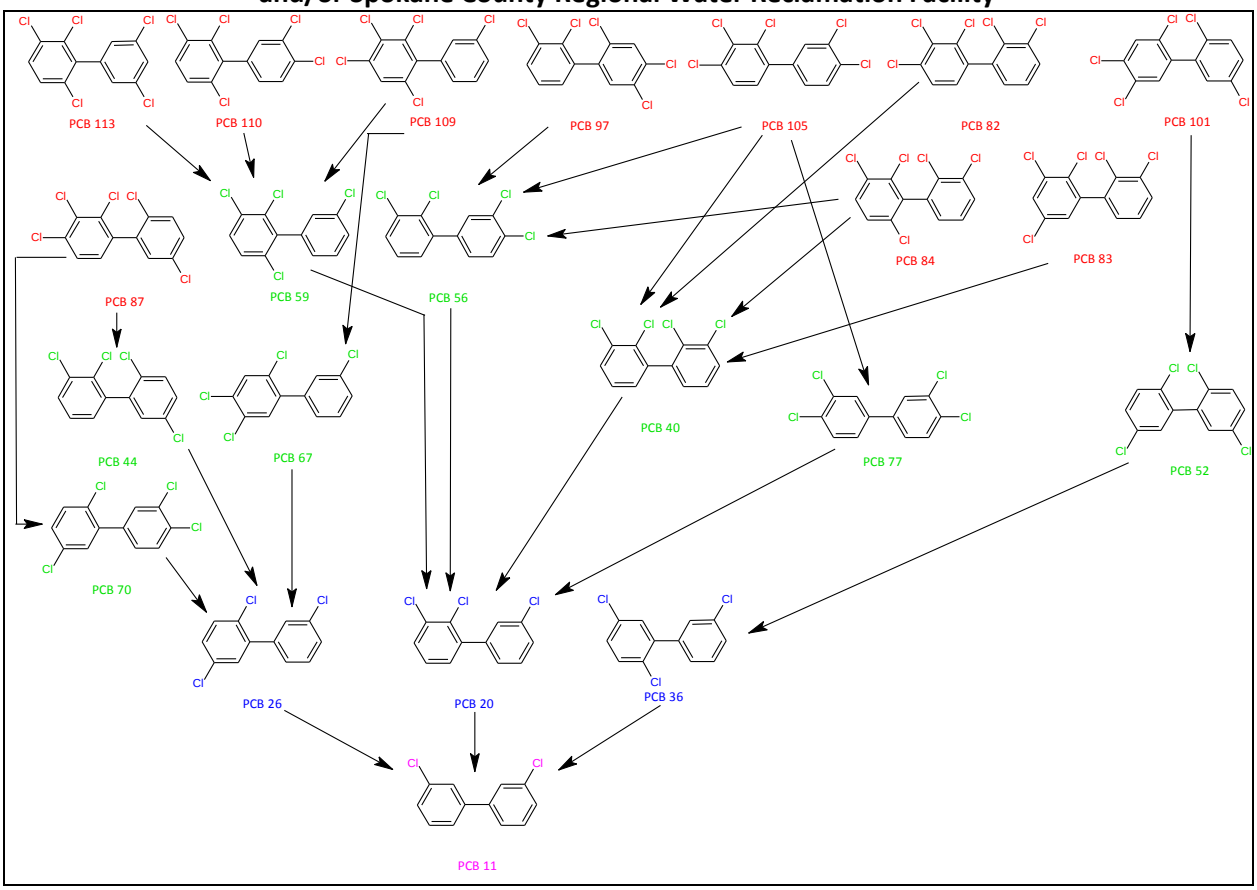
PCB Congener able to photodechlorinate to PCB 11	Type of PCB Congener	Identified in Spokane River Watershed [1]	Identified in Spokane County Regional Water Reclamation Facility [2]
PCB 11	Di	Yes	Yes

PCB 20	Tri	No	Yes
PCB 26	Tri	No	Yes
PCB 36	Tri	No	Yes
PCB 40	Tetra	Yes	Yes
PCB 44	Tetra	Yes	Yes
PCB 52	Tetra	No	Yes
PCB 56	Tetra	No	Yes
PCB 59	Tetra	No	Yes
PCB 67	Tetra	No	Yes
PCB 70	Tetra	Yes	No
PCB 77	Tetra	No	Yes
PCB 78	Penta	No	No
PCB 82	Penta	No	Yes
PCB 83	Penta	Yes	Yes
PCB 84	Penta	No	Yes
PCB 87	Penta	Yes	No
PCB 92	Penta	No	Yes
PCB 97	Penta	No	Yes
PCB 101	Penta	Yes	No
PCB 105	Penta	Yes	Yes
PCB 109	Penta	Yes	No
PCB 110	Penta	Yes	Yes
PCB 113	Penta	Yes	No
PCB 128	Hexa	Yes	Yes
PCB 129	Hexa	Yes	Yes
PCB 130	Hexa	No	Yes
PCB 132	Hexa	No	Yes
PCB 134	Hexa	No	Yes
PCB 135	Hexa	No	Yes
PCB 136	Hexa	No	Yes
PCB 138	Hexa	Yes	No
PCB 141	Hexa	No	Yes
PCB 144	Hexa	No	Yes
PCB 146	Hexa	No	Yes
PCB 153	Hexa	Yes	Yes
PCB 156	Hexa	No	Yes
PCB 158	Hexa	No	Yes
PCB 163	Hexa	Yes	No
PCB 164	Hexa	No	Yes
PCB 167	Hexa	No	Yes
PCB 170	Hepta	No	Yes
PCB 171	Hepta	No	Yes
PCB 172	Hepta	No	Yes
PCB 174	Hepta	No	Yes
PCB 176	Hepta	No	Yes
PCB 177	Hepta	No	Yes

PCB 178	Hepta	No	Yes
PCB 179	Hepta	No	Yes
PCB 180	Hepta	Yes	Yes
PCB 183	Hepta	No	Yes
PCB 185	Hepta	No	Yes
PCB 187	Hepta	No	Yes
PCB 190	Hepta	No	Yes
PCB 193	Hepta	Yes	No
PCB 194	Octa	No	Yes
PCB 195	Octa	No	Yes
PCB 196	Octa	No	Yes
PCB 197	Octa	No	Yes
PCB 198	Octa	No	Yes
PCB 201	Octa	No	Yes
PCB 202	Octa	No	Yes
PCB 203	Octa	No	Yes
PCB 206	Nona	No	Yes
PCB 208	Nona	No	Yes
PCB 209	Deca	No	Yes

In Table 1 it can be seen there are a total of 65 PCB congeners that have been identified in the Spokane River Watershed [1] or the Spokane County Regional Water Reclamation Facility [2] that have the potential to photochemically dechlorinate to PCB 11. Scheme 3 demonstrates how higher level PCBs congeners “funnel” on “concentrate” to PCB 11, which could explain why PCB 11 demonstrates a relatively higher level of concentration in the Spokane River Watershed [1] and the Spokane County Regional Water Reclamation Facility [2] than expected, in particular when PCB 11 is known to be more stable than other PCB congeners [15].

Scheme 3 – Pathway for the Photochemical Degradation of Pentachlorobiphenyls to Tetrachlorobiphenyls to Trichlorobiphenyls to PCB 11 as Detected in the Spokane River Watershed and/or Spokane County Regional Water Reclamation Facility



Scheme 3 only represents the photodegradation pathway of the pentachlorobiphenyls congeners. If the hexa, hepta, octa, nona and decachlorobiphenyls are considered (as in Table 1), the level of congeners potentially photodegrading in the Spokane region to PCB 11 increases significantly.

Within Scheme 3, there are 10 pentachlorobiphenyl congeners that potentially would photodegrade to 8 tetrachlorobiphenyls which sequentially photodegrade to 3 trichlorobiphenyls which ultimately degrade to 1 PCB 11 congener. Scheme 3 depicts the PCB congeners that have been detected and presented in the Spokane River Watershed [1] or the Spokane County Regional Water Reclamation Facility [2] test data. Of the 10 pentachlorobiphenyl congeners depicted in Scheme 3, nine congeners (PCB 82, 83, 84, 87, 97, 101, 105, 110, 113) have been detected at significant levels by the Spokane Task Force [1,2]. Of the 8 tetrachlorobiphenyl congeners, three congeners (PCB 40, 44, 70) and of the three trichlorobiphenyl congeners identified, two congeners (PCB 20, 26) have been detected at significant levels. The literature on the photochemical dichlorination and demonstrated in Scheme 1, provides strong evidence to suggest why PCB 11 is being detected at higher than expected levels in the Spokane River.

Rodenberg et al [3] reviewed the photochemical dichlorination of PCBs stating:

> "Another potential source of PCB 11 is abiotic photochemical dechlorination during atmospheric transport. Yao et al. [15] found that PCB 11 is produced during the photolysis of higher-molecular-weight PCBs in alkaline 2-propanol. Atmospheric deposition is an important source of PCBs to some water bodies, such as Lake Michigan. However, if PCB 11 were formed from photolysis of other PCBs, then the temporal trends in PCB 11 concentrations should follow those of the other congeners. They do not."

Later within the same paper Rodenberg et al stated:

> "The temporal trends of atmospheric PCB 11 levels have been investigated in the United States and Japan. According to Basu et al., the atmospheric PCB 11 concentration in the US Great Lakes Region did not follow the temporal trend of total PCBs, which were decreasing slowly over time. No significant pattern was observed for PCB 11 during the sampling period (2004–2007), except for one out of five sites where the atmospheric concentration showed a significant increase. This suggests that PCB 11 is not primarily formed via photochemical reduction of higher-molecular-weight PCBs, as this pathway would result in PCB 11 trends following those of the higher-molecular-weight precursors. In another study, monthly variations in PCB 11, 52, and 209 during 2005–2011 were reported for two urban/industrial cities in Japan. The ratio of PCB 11 to total dichlorobiphenyls (PCB 11/DiCB) in the atmosphere varied between seasons, with a higher contribution in summer, whereas the ratio of PCB 52 to total tetrachlorobiphenyls (PCB 52/TeCB) remained constant throughout the sampling period. As PCB 52 also originates from legacy PCBs, the atmospheric burden of PCB 52 is believed to largely result from those sources. In contrast, the large variations in PCB 11/DiCB over seasons confirmed that atmospheric PCB 11 was not associated with legacy PCBs. The ratio of PCB 209 to total PCBs also displayed a slight seasonal trend, suggesting non-Aroclor sources, such as pigments or combustion, for PCB 209."

The conclusion that PCB 11 does not follow temporal trends requires further investigation as the data is not definitive. The amount of PCB 11 increases in the atmosphere during the summer months due to several routes. Photochemical degradation of higher level PCB congeners (not just PCB 52) would occur at higher rates during the summer months increasing the amount of the more stable PCB 11 in the atmosphere. As PCB 11 has a higher vapour pressure it would be expected to be at higher levels during the summer months as higher level PCB congeners are broken down in soil, water, building products, atmosphere etc. As such it is not possible to complete a mass balance on atmospheric PCB content alone.

Other research on PCBs has indicated there are other mechanisms for the appearance of PCB 11 in the environment, besides diarlyde pigments. conducted by Garmash et al [17] in their paper "Deposition History of Polychlorinated Biphenyls to the Lomonosovfonna Glacier, Svalbard: a 209 Congener Analysis" found unexplained levels of PCB 11 within ice core samples dating from 1957-2009.

Whilst the presence of PCB 11 in diarylide pigments was discussed, other possible sources such as incineration were also considered. The final conclusion was that "more studies on PCBs originating from thermal processes (including incinerators) would be useful to determine their extent as a source of PCB 11 to the eastern Arctic. In general, the sources of PCB 11 to Lomonosovfonna are not well characterized".

The same can be concluded for the Spokane River. Indeed in a study carried out by the City of Spokane on PCBs in Municipal Products [18], PCB 11 was detected in synthetic and used motor oil (Chart 3), road dust suppressants such as EADA (Chart 4) and magnesium chloride (Chart 5), asphalt release agent (Chart 6), asphalt crack sealer (Chart 7, PCB 11 was detected at >4% of all PCB congeners in this sample), laundry soap (Chart 8) amongst others. These tests alone should allow for the conclusion that whilst it is not known the origin of PCB 11 in products and the environment, diarylide pigments are mostly likely not the significant contributor.

Chart 3 – PCBs Detected in Motor Oils

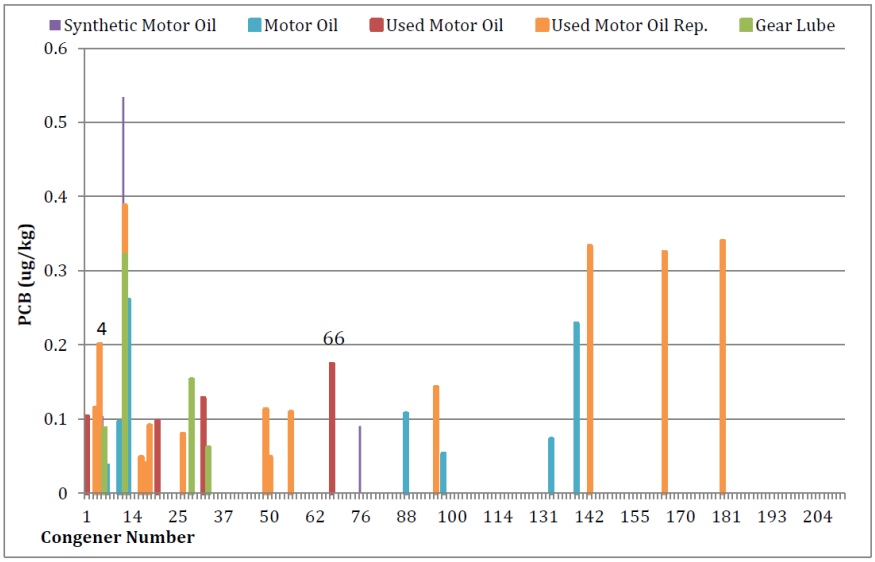


Chart 4 – PCBs detected in EADA Road Dust Suppressant

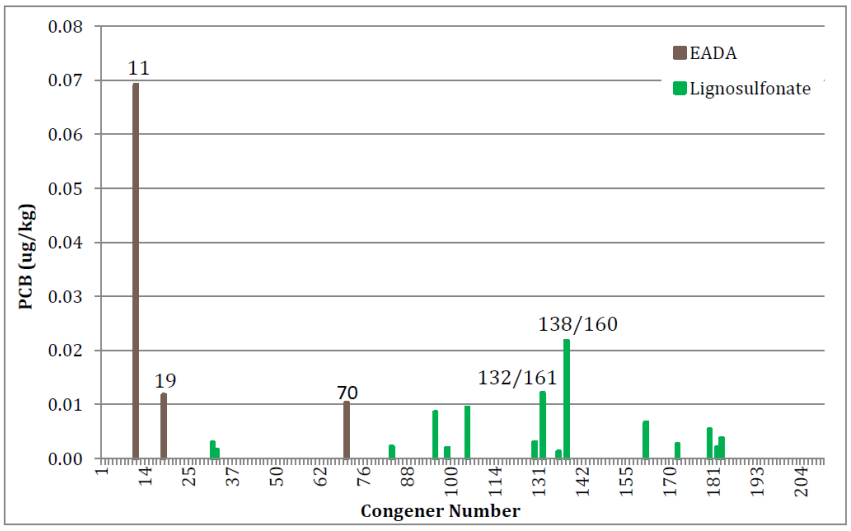


Chart 5 – PCBs Detected in Magnesium Chloride Road Dust Suppressant

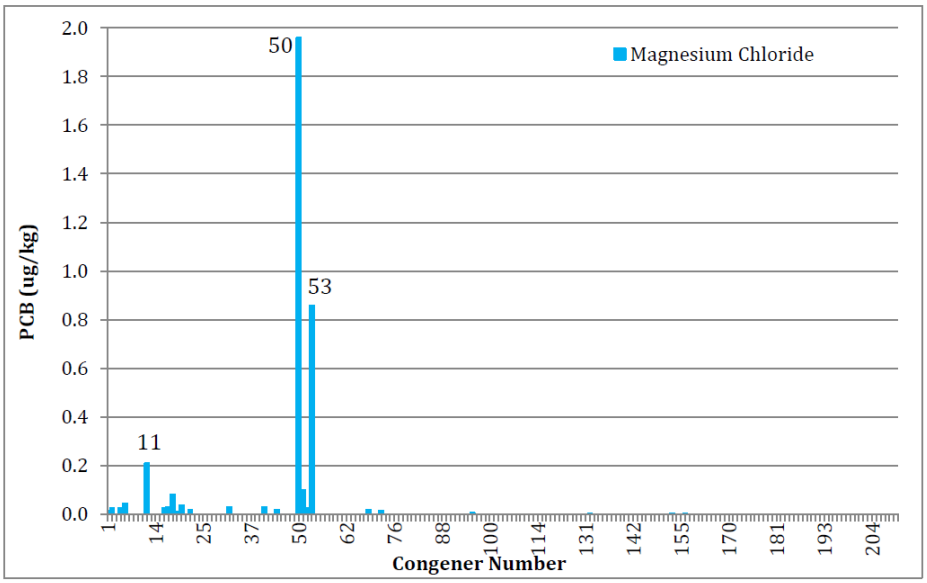


Chart 6 – PCBs Detected in Asphalt Release Agent

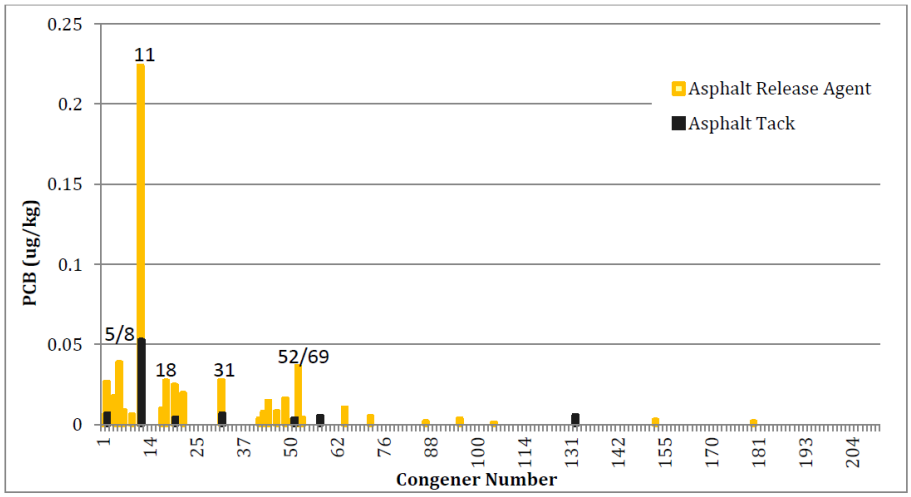


Chart 7 – PCBs Detected in Asphalt Crack Sealer

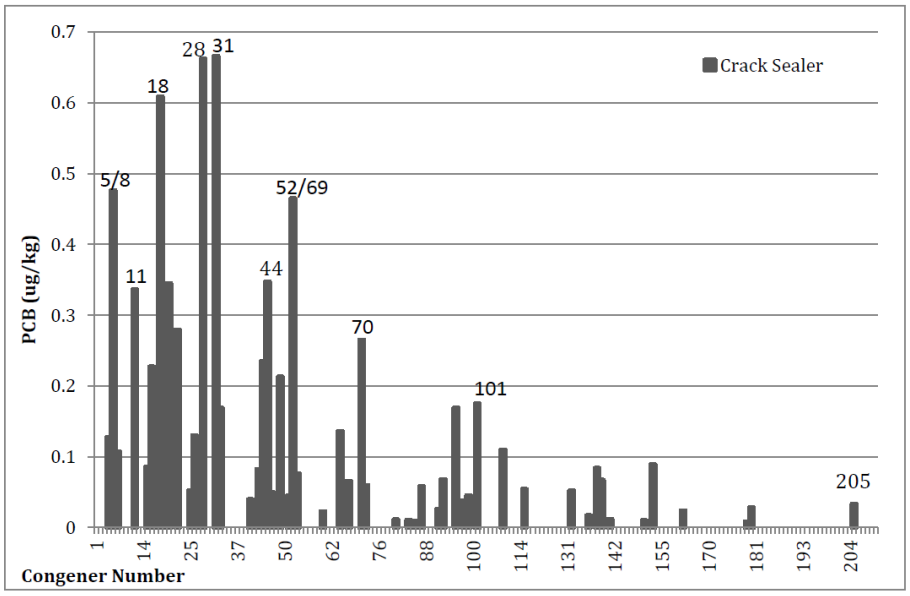
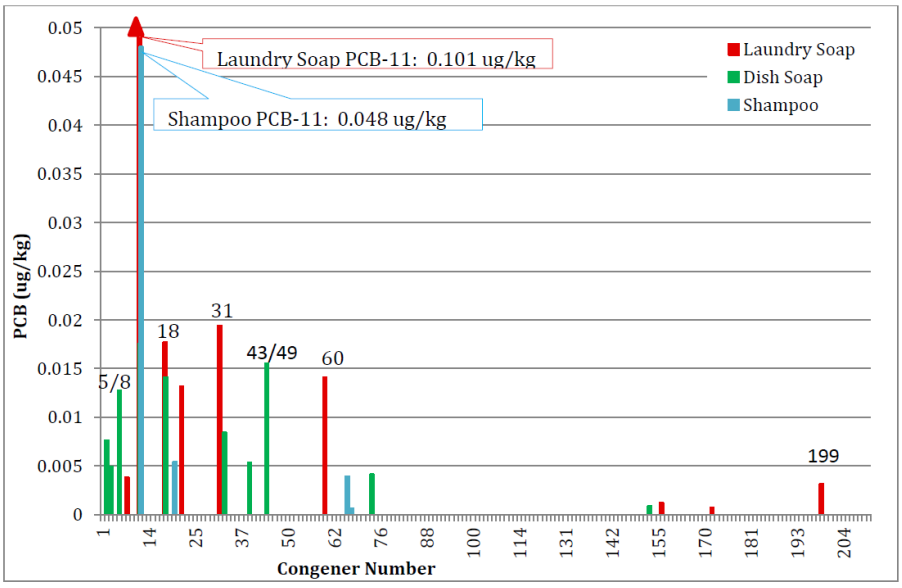


Chart 8 – PCBs Detected in Laundry Soap



Conclusions

At a minimum, the source of PCB 11 in the Spokane River requires further research and investigation. Without this any remedial actions implemented may be ineffective. Considering that levels increase during storm events [2] suggests that there are sources of PCB 11 that are currently unidentified. Whilst it is known that PCB 11 is present at low concentrations in diarylide pigments, the levels detected in the Spokane River cannot be attributed to pigments alone. There are at least two other possible sources of PCB-11 with known and well established routes and they are incineration and photochemical dichlorination.

Further study is needed related to generation of PCB 11 from incineration and photochemical photo-dechlorination and in products used by the City of Spokane not related to pigments (e.g. asphalt crack sealer).

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