Trends in the Distribution of PCBs Compared to PACs in Sediments and Mussels of Halifax Harbour

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Surficial sediments and blue mussels (Mytilus edulis) were collected around the perimeter of Halifax Harbour. Samples were analyzed for the presence of polychlorinated biphenyls (PCBs), ranging from mono- to decachloro congeners, and results compared to the presence of polycyclic aromatic compounds (PACs). The objective was to explore the relationship between contaminant profiles in sediments and in mussels, and the possibility of deducing their sources, patterns of deposition, circulation and bioavailability. IUPAC hexachlorobiphenyls 138/158/160/163 (unresolved) and 153/132/168 were the most abundant congeners in both sets of matrices, ranging in concentration between <0.5 to 46 and 4.5 to 25.5 ng/g (dry weight) in sediments and mussels, respectively. PCBs profiles point to a combination of commercial sources superimposed on some weathering. Biota-sediment accumulation factors (BSAF, concentration in mussels, dry weight/concentration in sediments, dry weight, and lipid/TOC normalized) varied throughout the harbour and per congener. Some similarities were observed between the most contaminated sites previously analyzed for PACs and presently analyzed for PCBs, while BSAF were highest at the entrance to the harbour, for PACs and PCBs.

Key words: transport, bioaccumulation, weathering, sewage, PAHs, PCBs

Introduction

Polychlorinated biphenyls (PCBs) were produced from the 1930s until the late 1970s and used as additives in a variety of products where their physical-chemical properties, including thermal stability and conductivity, were required (Niimi 1996). PCBs were added to dielectric fluids in capacitors and transformers, paints, pesticides, inks, hydraulic fluids, lubricating oils, adhesives and plastics (Safe 1986; deVoogt et al. 1990). They were synthesized by Fe-catalyzed electrophilic substitution of biphenyl with chlorine gas, and depending on reaction conditions, different mixtures were obtained. Some of the properties of PCBs, notably their thermal and chemical stability resulted in their persistence in the environment; which along with their lipophilic and hydrophobicity led to their biomagnification in the food chain. As well, due to atmospheric transport, PCBs have become ubiquitous around the world (Mackay and Wania 1995). These observations along with studies revealing a number of
toxic effects led to banning their industrial production in North America and Europe. Chronic exposure to PCBs has been associated with immune suppression, reproductive failure, growth and physiological anomalies, enzymatic induction, as well as changes in behaviour and effects on the nervous system (McFarland and Clarke 1989; Mousa et al. 1998).

Harbours receive discharges from a variety of contamination sources, where sediments represent a sink for lipophilic substances. In the present study, sediments of Halifax Harbour, Nova Scotia, Canada, were collected to determine the concentration and identity of PCB contaminants. Mussels, *Mytilus edulis*, used in many environmental studies, were also analyzed to compare the signature of congeners available for uptake by this filter feeder, to those deposited along the harbour. Results regarding the distribution of PCBs in these two compartments will be compared to those previously reported for PACs in the same samples (Hellou et al. 2002) and for PCBs in other studies. A comparison of the congener concentrations in sediments and mussels should provide insights into transport pathways for the congeners, and a test of complementary measurements of PCBs in these two phases as tools for assessing marine environmental quality.

**Materials and Methods**

Collection of surface sediments and inter-tidal blue mussels, *Mytilus edulis*, of 4 to 5 cm has been described (Hellou et al. 2000, 2002). Only dead animals were found at M 9 and 16 in April, and none at M17 in November (Fig. 1). Congeners numbering follows IUPAC convention.

Briefly, extraction of sediments and chromatography started with the addition of surrogate standards $^{13}$C-77, $^{13}$C-153, and $^{13}$C-194 to eight grams of dry sediment, which were extracted with a sonic probe in

![Fig. 1. Location of sampling sites in Halifax Harbour, numbers labelled with M represent mussel sites, while those with SS relate to sediment sites.](image-url)
dichloromethane. After concentration and solvent exchange into hexane, extracts were eluted through a multi-layer clean-up column as detailed by Raccanelli et al. (1994). The PCB fraction was concentrated and exchanged into isooctane, internal standard $^{13}$C-138 was added with adjustment of the final volume in isooctane. Samples were treated with 20 to 30 activated mesh copper pellets to remove sulphur from extracts. Sample SS12 did not display the presence of PCB surrogate standards and was re-analyzed later along with a blank, duplicate and reference material (RM). Because of higher detection limits (DL = 0.1 earlier and 1 ng/g, dry weight for SS12), results are not included in Fig. 2 and 3 for SS12.

Extracts were analyzed by electron impact mass spectrometry (EI-MS) in the selective ion monitoring mode. Congener identification, similar to King et al. (1995), was verified by retention time and by comparing the ratio of the quantifying ion with one confirmation ion, in the sample and commercial reference standard. Blanks, duplicates, and RM were run with each batch of 10 to 12 samples. Duplicate analyses of our samples yielded results with little variation (<10%), due to the use of internal standards. Surrogate recoveries were (average ± standard deviation) 72 ± 11% for $^{13}$C-77, 77 ± 12% for $^{13}$C-153 and 73 ± 12% for $^{13}$C-194, and results are corrected for recoveries. The first surrogate was used to correct for di-, tri- and tetrachlorobiphenyls. The second corrected for the penta-, hexa- and heptachlorobiphenyls, while the last one was used to correct the more chlorinated congeners. RM HS-2 was obtained from the National

![Comparison of relative proportions](image)

**Fig. 2.** Comparison of relative abundance of homologue series using 14 more abundant congeners in sediments. Sites are listed following the perimeter of the harbour, going from the most north-eastern site, around the harbour towards Bedford Basin and to the most south-eastern site.
Fig. 3. Concentration of the sum of PACs (µg/g, dry weight) and PCBs (ng/g, dry weight) observed in sediments, along with the total organic carbon content (TOC %).

Results and Discussion

Around 60 congeners were synthetically produced in commercial PCB mixtures, at a level above 1% of total, by weight (Frame et al. 1996; Schultz et al. 1989). However, the possible photo- and biodegradation of PCBs can lead to the identification of non-commercially produced congeners in samples. Our analytical approach targeted up to 159 congeners (King et al. In press).

Sediments

In sediments, fewer than 26 congener groups (some co-eluting; Table 1) made up 80 to 100% of the sum of PCB concentrations, with their sum varying by a factor of nearly 50. Only 14 of these were present in more than 50% of the samples, with IUPAC congeners 138/158/160/163 and 153/132/168 detected at levels between <0.5 to 46.2 ng/g and <0.5 to 28.6 ng/g, dry weight, respectively. The sum of pentachloro homologues
Table 1. Detectable PCBs in sediments

<table>
<thead>
<tr>
<th>Chlorin.</th>
<th>IUPAC congener</th>
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<th>IUPAC congener</th>
<th>Chlorin.</th>
<th>IUPAC congener</th>
</tr>
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<tbody>
<tr>
<td>Di</td>
<td>No. 9/7 (a)</td>
<td>Tetra</td>
<td>No. 49</td>
<td>Hexa</td>
<td>No. 154</td>
</tr>
<tr>
<td>Di</td>
<td>No. 6</td>
<td>Tetra</td>
<td>No. 47/48/62/65/75</td>
<td>Hexa</td>
<td>No. 144</td>
</tr>
<tr>
<td>Di</td>
<td>No. 8/5</td>
<td>Tetra</td>
<td>No. 42</td>
<td>Hexa</td>
<td>No. 153/132/168</td>
</tr>
<tr>
<td>Di</td>
<td>No. 12</td>
<td>Tetra</td>
<td>No. 72</td>
<td>Hexa</td>
<td>No. 138/158/160/163</td>
</tr>
<tr>
<td>Tri</td>
<td>No. 18</td>
<td>Tetra</td>
<td>No. 77</td>
<td>Hepta</td>
<td>No. 183</td>
</tr>
<tr>
<td>Tri</td>
<td>No. 25</td>
<td>Tetra</td>
<td></td>
<td>Hepta</td>
<td>No. 180</td>
</tr>
<tr>
<td>Tri</td>
<td>No. 28/31</td>
<td>Tetra</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Tri</td>
<td>No. 33/20</td>
<td>Penta</td>
<td>No. 95/121/88</td>
<td></td>
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<tr>
<td>Tri</td>
<td>No. 37</td>
<td>Penta</td>
<td>No. 101</td>
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<td>Penta</td>
<td>No. 110</td>
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<td>Penta</td>
<td>No. 114</td>
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\(a\)Bold numbers were present in more than 50% of samples, while italics were present in fewer.
was more abundant than the tetra- and hexachloro, except at sites SS 13 and 15 where tri- and tetrachloro were more abundant (Fig. 1 and 2). The mean relative concentration was determined by summing homologue series across the harbour, adding the results for total concentrations and representing PCBs in terms of relative proportions (“all” in Fig. 2). This result could be interpreted as having SS7, with the highest overall concentrations (Fig. 3), introducing the major amount of PCBs into the harbour, with further weathering and transport taking place towards Bedford Basin and the entrance to the harbour. This site is in downtown Halifax close to numerous raw sewage effluents (Hellou et al. 2002). However, it is very likely that there are several point sources introducing different PCB mixtures, or combinations of, into the harbour (Buckley and Winters 1992). PCBs are then transported a shorter or longer distance, depending on turbulence, currents and/or their physical-chemical properties. The water circulation in our harbour is estuarine, with a stronger deep-water inflow towards Bedford Basin and a stronger surficial-water outflow towards McNab Island (Fig. 1, Petrie and Yeats 1990). Tidal currents are important, moving discharges both inward and outward up to 1.5 km depending on location and tide (Petrie and Yeats 1990). Therefore, transport of contaminants is expected to occur.

To pinpoint the commercial PCB source, the homologue distribution is compared between sediments and that published for industrial Aroclor mixtures (Anderson 1991). Results (Fig. 2) suggest that the dominant Aroclors in the sediment of the central harbour area are 1254 and 1260. Because of weathering it is difficult to pinpoint the sources more precisely using congener ratios and/or principal component analyses. Interestingly, three di- and trichlorinated congeners 6, 8/5 and 18 detected in 2, 6 and 13 samples, respectively, have been reported as end products of dechlorination pathways (Bedard and May 1996). This could indicate that some biodegradation is taking place in the anaerobic sediments of the harbour. Elevated concentrations of polybrominated diphenyl ethers (PBDEs), identified in some animal extracts (ongoing work), therefore expected to be present in sediments, could be enhancing the activity of bacteria on PCBs at these low environmental levels (Rhee et al. 2001). This hypothesis would have to be verified, whether PCBs can be biodegraded when present in low concentration, but with elevated PBDEs.

Lowest PCB concentrations were observed at the entrance to the harbour, i.e., SS 6, 10, 13, 14, 15 and 19, similar to the pattern observed for PACs, in the same sediments (Fig. 3; Hellou et al. 2002). However, samples SS 11 and 12 from the Northwest Arm contained elevated concentrations of PACs assigned to the presence of used coal, which were similar in terms of sum of PACs to SS7. On the other hand, the concentrations of PCBs in SS11 (135 ng/g, with more ND at SS12 than in the rest of samples) are lower than at SS7 (718 ng/g). They are also within the range of samples collected in the main channel of the harbour, but higher than in samples collected from the entrance to the harbour. This difference between the sums of PACs and PCBs could be indicating that PACs derive from non-sewage sources in the Northwest Arm. The relatively lower levels of PCBs
than PACs, at SS6 compared to other sites are difficult to explain. A sewage treatment plant is located close to SS6 and SS9, while a petroleum refinery is near SS6. It was previously noted (Hellou et al. 2002) that PACs at SS9 were lower than at the two closest sites, SS 8 and 17, while they were lower at SS6 relative to SS20, but not relative to SS15, perhaps because located at the entrance to the harbour, with stronger currents. The partitioning of PCBs does not show a similar trend to PACs around these sewage treatment plants and this difference needs to be pursued with further work.

Pearson correlations (r) between summed PCBs, total organic carbon (TOC) and sulfur (S) content of sediments indicated positive correlations, above the 1% level of significance. Correlations between specific congeners and sediment characteristics were weaker for the lower chlorinated congeners compared to the hexachlorinated (>1% compared to >5% level). Stronger correlations, by a factor of 3 to 5 were observed between TOC than between S and the compounds. Higher correlations were observed between hexachloro congeners and the various sums of PACs (>0.01% level), than with di-, tri- and tetrachlorinated congeners (<0.1% level). This could reflect a greater mobility of the less compared to more chlorinated PCBs or than the PACs. Another explanation could be the different biodegradation behaviour of the two groups.

Lower chlorinated congeners would be expected to weather more readily than higher chlorinated ones (Dexter et al. 1978). A higher mobility of dichloro PCBs, followed by a decreasing trend up to the pentachloro, but then increasing up to the octachloro PCBs, has been reported (Axelman and Broman 1999). Lower chlorinated congeners have been reported as more susceptible to long-range transport and degradation than more chlorinated ones (Harrad et al. 1994). Differences between the distribution of homologue series are also observed here. Greater proportions of penta and hexa homologues are found at SS4, SS7, SS16, SS17, and SS18 near sewage sources. Higher proportions of trichloro homologues were found at sites SS 10, 13, 14, 15 and 20, farther from the primary depositional areas (Fig. 4).

Comparing Sediments Results to Other Studies

Generally speaking, PCB levels reported in the literature vary from <1 to >1000 ng/g (dry weight). Higher sediment concentrations are due to their proximity to industrial production sites (Bedard and May 1996; Lake et al. 1995). Our results for congeners 153 and 138 (different coelution in studies, 1–46 and <0.5–29 ng/g, respectively) are similar to those observed in Lake Ontario (15 ± 7 and 25 ± 11 ng/g, respectively; Oliver and Niimi 1988) and near other sewage effluents (7 and 8 ng/g, Hellou et al. 1998; 9 and 15 ng/g, Boese et al. 1997). Some of the variables affecting the observed concentrations relate to the grain size and TOC of sediments. Higher TOC content and smaller grain size are generally linked with higher levels of contaminants. These variables along with the organic matter circulating in the water play a major role in the transport, partitioning and deposition of hydrophobic organic contaminants.
Since the late 1980s, reviews have been published documenting the level of total PCBs in sediments associated with biological effects. MacDonald et al. (2000) presented a consensus-based sediment effect concentration (SEC), where the threshold SEC was 40; the mid-range was 400, while the extreme SEC was 1700 ng/g, dry. These authors discuss the difficulties in evaluating some of the toxic effects and limitations of the SECs. The Canadian Council for the Marine Environment (1999) has published an interim marine sediment quality guideline (SQG) and a probable effect level of 21.5 and 189 ng/g, dry, respectively. In Halifax Harbour, 14 of 18 surficial sediments are above the threshold SECs and only SS7 is above the consensus-based midrange SEC. Normalizing sediment concentrations using TOC was supported for polycyclic aromatic hydrocarbons (PAHs) and SQG, but not for PCBs (Swartz 1999). If normalization is applied, fewer sites would be above threshold SEC, i.e., SS 7, 8, 16, 19, 20 and 21, while SS9 and SS18 would be borderline (40–41 ng/g, dry). This comparison indicates that PCBs could cause biological effects in sediment-dwelling organisms located around the harbour area. This is similar to conclusions from our PAC study (Hellou et al. 2002).

Mussels

Mussels were sampled from 17 and 16 sites in November 1997 and April 1999, respectively. Animals are known to reproduce during May to July and therefore the two groups are non-spawners. At each season, 10 to 16 congeners (unresolved) were detected in more than 60% of the samples, but with differences in the relative profile of congeners (Fig. 5). The congeners consisting of IUPAC #11, 28/31, 47/48/62/65/75, 52, 61/74, 99, 118/106, 138/158/160/163 and 153/132/168 represented between 39 and 81% of the sum of PCBs detected in mussels. Congeners 11 and 28/31

Fig. 4. Comparison of the distribution of IUPAC congeners 28/31 and 153/132/168, representing tri- and hexachlorobiphenyls, with a log $K_{ow}$ of 5.67 and 6.92/6.58/7.11, respectively, in sediments.
were the only representatives of the di- and trichlorobiphenyls detected in many samples and at relatively high levels.

The lipid-normalized sums of PCBs indicated <40% reduction in concentration from November to April, in 9 of the 15 sets of mussels sampled on both occasions: M 1, 2, 3, 4, 5, 6, 7, 10, and 18 (Fig. 6). However, more than 3 times higher levels of summed PCBs were observed in M11 to M14 collected in November 1997 compared to April 1999. Three of these sites, i.e., M11 to M13, are located within the Northwest Arm, a relatively non-industrialized but urbanized area with a large amount of recreational boating activity during the summer. Effluents from a creek are discharged at the head of the Arm and a single sewage discharge enters the Arm. November animals from M 8, 15 and 16, within the main channel of the harbour had summed PCBs at least twice those observed in April. These results differ from those observed for PACs since in April mussels M 8, 14

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![Comparing sum PCBs (April)](image1.png)

![Comparing sum of PCBs (Nov)](image2.png)

**Fig. 5.** Comparison of the concentration of different classes of congeners detected in mussel samples. A is for November mussels, B is for April mussels.
The bioaccumulation of contaminants by mussels is described by quasi-steady-state, non-equilibrium steady-state, apparent steady-state or non-steady-state conditions (Bjork and Gilek 1999; Morrison et al. 1996; McDowell Capuzzo et al. 1989). Animals maintained in the laboratory and collected in the field do not always display similar responses (Gossiaux et al. 1998; Roe and McIsaac 1998). It has also been shown that contaminants in mussels can reach equilibrium within 2 to 3 months (Hummel et al. 1990). Under equilibrium conditions, concentrations would be expected similar for November and April mussels, although a dilution of PCB concentrations was previously observed during fat accumulation (Hummel et al. 1989). Therefore, the present results indicate a variable input of PCBs, sometimes different from that of PACs, in the harbour. It points to the importance of long-term monitoring, when there are numerous potential and unpredictable point sources of contamination.

Congener 11 found in water, phytoplankton, lobsters and mussels provides an interesting story to illustrate the current introduction of a PCB (Fig. 5; King et al. In press). This congener does not belong to an Aroclor signature, except for 0.15% in Aroclor 1221, nor could it be reasonably assigned to weathering processes. Congener 11 has been reported as a side product in the synthesis of diazo dyes used to produce red, orange or yellow pigments (Law 1995). Dyes are a likely source for this congener in our harbour.

Comparing Mussels Results to Other Studies

Concentrations of the more abundant and most bioaccumulative congeners, IUPAC #138 and 153 (different co-elution in studies, 4.5–20 and 6.5–25.5 ng/g, all dry weight) are within the lower range of levels and 15 had concentrations more than 50, 30 and 10 times above the rest of the samples, while only dead animals were found at M 9 and 16 (Hellou et al. 2000, 2002).

Fig. 6. Sum of PCBs (ng/g lipid, dry) detected in more than 60% of the samples, observed in November and April mussels.
observed in mussels from 10 locations covering the Atlantic and Pacific coasts of the U.S. (15–80 and 11.5–145 ng/g, Ylitalo et al. 1999). Concentrations are lower than those reported for another species of mussels in Lake Erie (2700 ± 250 and 2200 ± 100 ng/g, Roe and McIsaac 1998). Concentrations are low when compared to oysters analyzed in the National Status and Trends Mussel Watch Programs, for the Gulf of Mexico (Jackson et al. 1998) and other sites in the U.S.; to the Gulf of Maine Mussel Watch Program; but similar to those reported for other sites in Nova Scotia (Jones et al. 1998).

Comparing Mussels and Sediments

Water-soluble as well as particle-bound organic contaminants will bioaccumulate in mussels. Non-ionic organic compounds of log K\text{ow} below 3.5 to 4 are expected to be generally available from water, through respiration, while those of log K\text{ow} above 5.5 to 6, are more available from food (McKim 1994). In the present study, PCBs had a log K\text{ow} between 5.06 to 5.07 for congeners 6 to 9; and of 7.20 and 7.36 for congeners 183 and 180, respectively (Hawker and Connell 1988). Therefore, food could represent a substantial source of these contaminants for mussels. Sediments will collect PCBs from settling particles including the types of particles that constitute food for mussels. It is therefore of interest to determine the relative PCB concentrations in mussels and sediments, using biota-sediment accumulation factors (BSAF = concentration in animals/concentrations in sediments, both in dry weight; also lipid and TOC normalized).

Highest BSAF were observed at sites M1-SS15 and M14-SS10 located at the entrance to the harbour (Fig. 7). In a parallel study of a series of

![BSAF for April mussels](image)

Fig. 7. Biota sediment accumulation factors (BSAF) observed for some PCBs in April mussels. This ratio is normalized for the lipid content in animals and total organic carbon content in sediments.
PACs, M1-SS15 also displayed the highest BSAF, followed by M14-SS10 (Hellou et al. 2002). Since SS15 had concentrations <0.1 ng/g, dry, the BSAF of M1-SS15 would be expected higher than at the other two sites situated more to the west of the entrance to the harbour (Fig. 1). The higher BSAF means that mussels from these locations are relatively more exposed to contaminants circulating in the water column than would be assumed by comparison of concentrations in sediments. These locations are outside the primary depositional areas for suspended matter introduced into the central harbour (Petrie and Yeats 1990). There would appear to be a separation between dissolved PCBs and particles (planktonic) reaching mussels at these sites and the more quickly settling particles that reach the sediments within the central harbour and Bedford Basin. These variable BSAF results demonstrate the importance of using different tools in assessment studies interested in ecosystem health.

Bioaccumulation has been discussed relative to the structure of PCBs, where it was deduced that coplanar congeners accumulate more from water than through the food chain (Willman et al. 1999). Bioaccumulation has been described for PCBs and PAHs, in terms of log $K_{ow}$, relative to metabolism and gut assimilation efficiency, with examined species (Thomann and Komlos 1999; Gewurtz et al. 2000). Farrington (1991) pointed out that for PAHs, the physical form of introduced contaminants plays a major role in bioavailability. Aging and specific binding of hydrophobic contaminants to particles can also affect fate (Luthy et al. 1997a; Gong and Depinto 1998). For example, a study of the solubility of PCB congeners present in a commercial mixture with and without hydraulic oil pointed to increased water solubility in the latter case (Luthy et al. 1997b). The differing BSAFs of contaminants observed between sites in the present PCB and an earlier PAC study need further elucidation by examining the contaminants in water and particulate. A better understanding of exposure and partitioning processes would enhance our ability to protect the environment for future generations and will help with the management of aquatic resources.

An important observation deriving from this study of PCBs and PACs is the lack of detectable congener 11 in our sediments. The log $K_{ow}$ of this congener (5.19–5.28; Hawker and Connell 1988) is very similar to that of congener 18 (5.24–5.33) detected in sediments, and to fluoranthene (FLU) and pyrene (PY; 5.22 and 5.18, respectively, Mackay et al. 1992). These PAHs are found in various combustion and petroleum sources, and in our sediments (Hellou et al. 2002). The water solubility of the compounds is similar (#11, 18, FLU and PY: 0.354, 0.40, 0.26 and 0.132 g/m³, respectively, Mackay et al. 1992). The Henry’s Law constant is lower for the two PAHs than PCBs (0.92 and 1.04 compared to 17.26 and 92.21 m³/mol, Mackay et al. 1992), indicating higher partitioning into air compared to water, for the PCBs. The fugacity of these compounds in water, which reflects their partitioning, would be equal to the inverse of Henry’s Law constant (1.09, 0.99, 0.06 and 0.01 for FLU, PY, #11 and #18, respectively). This comparison of properties raises questions about the presence of PCBs in air. The
volatilization of less chlorinated PCBs has been questioned in at least another study and also needs further attention (Bushart et al. 1998).

A different aspect of the above study, that was discussed with the PAC results related to determining the health of blue mussels from a cumulative effects perspective, where condition indices and lipid content were investigated (Hellou et al. 2000, 2002). So far, these two effects appear to be perhaps more apparent in mussels from downtown Halifax. However, it should be pointed out that the chemical-biological link is being pursued with more sampling in time, at 3 sites (M 8, 11 and 14), to better understand the state of this complex environment. As well, additional biological effects are determined, as funding permits. However, numerous questions remain to be investigated including the presence of other anthropogenic contaminants discharged with raw sewage effluents, as well as reproductive and estrogenic effects in animals. Other estuarine species, which spend a portion or all of their lifetime in contaminated harbours need attention.

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References


