Caribbean Coastal Pollution Project (CCPP)

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Monitoring POPs in White Grunt from the Wider Caribbean Region

Final Report
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Summary of results:

- Preliminary dataset from limited samples of white grunt from 3 CARICOM countries (Jamaica, St Lucia and Trinidad and Tobago) and from Belize, but only regional data set for POPs in marine fish.
- PCB congeners and OC compounds were generally present in the muscle tissues of white grunt at concentrations <10 µg/kg wet weight (i.e. ppb).
- Mean concentrations of methoxychlor, ∑PCB and ∑BHC were significantly higher in white grunt collected from St. Lucia.
- Presence of DDE metabolite from all samples reflects transformation from DDT that was used at some time in the past, and shows that there has been no recent use of this insecticide.
- Methoxychlor (substitute for DDT) was observed in St. Lucia samples at concentrations significantly elevated relative to levels in fish from other two sites.
- Data from St Lucia indicate that the levels of methoxychlor, HCB and ∑heptachlor were relatively homogeneous across all stations but mean concentrations of ∑PCB and ∑ BHC were significantly elevated in fish collected at Vieux Fort Black Bay (LC006).
- Differences in OC contamination from these Caribbean countries may reflect:
  i) geographical differences in the use of pesticides
  ii) differences in overland or subsurface transport pathways from the source to the coastal zone
  iii) marine circulation patterns and currents, and/or
  iv) patterns of atmospheric deposition of POPs
- In general, the pattern of PCB congeners seen in white grunt indicates that the source of contamination is atmospheric deposition.
- The PCB congener pattern observed in white grunt collected from Vieux Fort Black Bay in St. Lucia indicates that there is a point source of PCBs at this single site.
- Belize samples analyzed for PBDEs revealed that several congeners were present at detectable concentrations in the muscle tissue.
- Data show that none of the concentrations of compounds detected in white grunt approached most stringent of fish consumption advisory levels of USA and Canada.
- Very low lipid content of the muscle tissues for this fish species contributed to the low concentrations of these lipophilic compounds.
- Data required to evaluate pesticide use in these countries to determine whether white grunt contamination reflects local use of OC compounds.
- More data are required to determine whether there are geographic and regional trends in the distribution of POPs in this region.
- Useful to determine distribution of POPs in biota from other levels in the marine food web in the Caribbean, including fish species that have a higher trophic status and/or have a high lipid content in their tissues.
- Need to monitor for 9 new SC POPs (e.g. PBDEs, PFOS, chlordecone), and persistent and toxic substances of emerging interest (e.g. mercury, synthetic musks, “new” brominated flame retardants).

1.0 Introduction:

Most Caribbean nations are signatories to the Stockholm Convention, which aims to reduce and mitigate contamination from selected persistent organic pollutants (POPs). However, there is little capacity within the Caribbean to analyze and monitor POPs in humans, fish and wildlife and the abiotic environment. Studies of POPs contamination in the Caribbean coastal environment have been limited to a few projects that have detected localized sources of contamination (Coat et al., 2006; Norena-Barroso et al., 2004). In addition to contamination from localized point sources of POPs, the Caribbean basin may be influenced by inputs of POPs at a regional scale from large continental rivers, such as the Orinoco River to the southeast and the three major rivers that enter the Gulf of Honduras to the southwest. Finally, atmospheric deposition of POPs to the Caribbean may be changing over time as a result of climate change (Semeena et al., 2006). This project was initiated to develop capacity within the Caribbean for monitoring POPs in the coastal environment and to gather data on the distribution of POPs in marine resources throughout the wider Caribbean region.

Through discussions at the first and second planning meetings for the Caribbean Coastal Pollution Project, the partner countries agreed to collect white grunt (*Haemulon plumieri*) for an initial survey to monitor the levels of POPs in fish tissues from the Wider Caribbean Region. It was agreed that each country would collect at least 3 fish (where available) at an average of 6 sites per country. Ideally at each site, the fish sampled would be 300 – 500 g in weight. If no white grunt were present, another benthic-feeding, non-pelagic fish could be substituted. The rationale for selecting the white grunt for the monitoring study was that this species: i) is widely distributed across the Caribbean, ii) is a reef fish that is relatively philopatric, and so reflects contamination at a discrete site and, iii) has been monitored previously for POPs in the western Caribbean through the MBRS program. It was decided that muscle tissue should be analyzed because of the relative ease in collecting dorsal muscle (as opposed to liver tissue), and because analysis of muscle can be related to risk analysis for the consumption of POPs in fish. It was acknowledged that there are drawbacks to using white grunt for a monitoring study of POPs. This species of fish is not high in trophic position, and therefore, is expected to show little effect of biomagnifications through food webs. It also has low lipid content in its tissues, and therefore, is not likely to accumulate lipophilic contaminants (i.e. POPs) to high concentrations.

Following an inter-laboratory comparison exercise in the summer of 2009, the two regional laboratories began to prepare white grunt tissues for analysis. This report provides an overview of the results generated from the analysis of white grunt that were collected from coastal waters.

Figure 1: Sites in the wider Caribbean region where white grunt were sampled for analysis by the regional laboratories at UWI Mona (Jamaica) and CINVESTAV (Mexico).

2.0 Methods:

White grunt were collected by the 8 partner countries from several coastal sites in the wider Caribbean region (Figure 1). White grunt from Jamaica, Trinidad and Tobago, and St. Lucia were shipped to the regional laboratory at UWI Mona in Jamaica, and white grunt collected from the Dominican Republic and the Caribbean coast of Mexico and Guatemala were sent to the regional laboratory at CINVESTAV in Merida, Mexico. Note that white grunt fish collected off the Caribbean coast of Honduras were destroyed and so no data were generated for this partner country.

Dorsal muscle tissues of white grunt (4-5 g) were spiked with an internal standard (PCB 30) and extracted using cold column extraction at UWI Mona, and by another solvent extraction method at CINVESTAV. Because of the low lipid content of the white grunt tissues (i.e. <0.5%), it was not necessary to remove lipid using gel permeation chromatography (GPC), so tissues were cleaned up directly using florisil column chromatography. Three fractions were generated by florisil chromatography: i) Fraction 1 containing primarily PCBs, and ii) Fractions II and III containing organochlorine compounds.

Table 1: List of target compounds analyzed in samples of white grunt dorsal muscle.

Organochlorine compounds

- Hexachlorobenzene (HCB)
- ∑chlordane: *cis*-chlordane, *trans*-chlordane, *oxy*-chlordane
- ∑DDT: o,p'-DDD, p,p'-DDD, o,p'-DDE, p,p'-DDE, o,p'-DDT, p,p'-DDT
- ∑BHC : α-BHC, ß-BHC, γ-BHC, δ-BHC
- ∑'drins: aldrin, dieldrin, endrin
- ∑heptachlor: heptachlor, *cis*-heptachlor epoxide, *trans*-heptachlor epoxide
- ∑endosulfan: endosulfan I, endosulfan II
- Mirex
- Methoxychlor

PCBs:

∑PCB: Congener numbers 18, 31/28, 33, 44, 49, 52, 66/95, 70/76, 74, 82/151, 87, 99, 101, 105/132, 110, 118, 128, 138, 149, 153, 156/171, 158, 170/190, 177, 180, 183, 187, 191, 194, 195/208, 201, 205, 206, 209

PBDEs:

∑PBDE : Congener numbers 3, 7, 15, 17, 28, 47, 49, 66, 71, 77, 85, 99, 100, 123, 138, 153, 154, 183, 184, 196, 197

These florisil fractions were analyzed for the PCB congeners and organochlorine compounds listed in Table 1 by gas chromatography using an Agilent 7890 gas chromatograph with an electron capture detector (i.e. GC-ECD). Laboratory blanks and a certified reference material (CRM) of Lake Michigan fish were extracted with each batch of 3-6 white grunt samples. Only the data for samples analyzed at the UWI Mona (Jamaica) laboratory are presented in this report, for reasons that are discussed below.

In addition, a limited number of samples of white grunt collected from Belize (n=5) were analyzed for concentrations of selected congeners of polybrominated diphenyl ethers (PBDEs). For PBDE analysis, Fraction II from the florisil cleanup step was analyzed by gas chromatography using an Agilent 7890 gas chromatograph with a mass selective detector (i.e. GC-MSD). Analysis of the PBDE congeners listed in Table 1 was conducted at the Great Lakes Institute for Environmental Research (GLIER) at the University of Windsor.

3.0 Results and Discussion:

3.1 Quality control:

Quality control checks of the laboratory blanks generated by the regional laboratory at CINVESTAV in Merida, Mexico indicated that there was considerable background contamination, which interfered with the analysis of extracts prepared from white grunt. Therefore, data generated by the CINVESTAV regional laboratory from white grunt collected off the coast of Mexico, Belize, Guatemala and the Dominican Republic are not included in this report.

The laboratory blanks generated by the regional laboratory at UWI Mona (i.e. Jamaica) had acceptable levels of background contamination, and so the results of the analyses of extracts from white grunt from Jamaica, St. Lucia, and Trinidad and Tobago are presented in this report. Typically, three white grunt were analyzed from each site, although in a few cases, 1, 2 or 4 white grunt were analyzed (Table 2). At some locations, no white grunt were collected, and so there are no contaminant data (Table 2).

Table 2: Sampling sites in Jamaica, St. Lucia and Trinidad and Tobago, site codes and numbers of white grunt analyzed from each site.

Jamaica	Cow River **JM001** 2	Cow Bay **JM002** 3	Discovery Bay **JM003** 2	Kingston Harbour **JM004** 0[1]	Negril **JM005** 3	Ocho Rios **JM006** 3	Portland Bight **JM007** 1	St. Marg Bay **JM008** 4
St. Lucia	Anse Le Raya Bay **LC001** 0[1]	Castries Harbour[2] **LC002** 0[1]	Ciceron Bay **LC003** 0[1]	Fond d"Or **LC004** 3	Roseau **LC005** 3	Vieux Fort BB **LC006** 3	Castries Harbour[2] **LC007** 3	Vieux Fort Airport **LC008** 3
Trinidad & Tobago	Charlotte-ville **TT001** 2	Matura **TT002** 3	Ortoire **TT003** 3	Mt. Irvine **TT004** 3	Moruga **TT005** 3	Chagar-amus **TT006** 3		

1) No white grunt were collected at these sites
2) Two sites at Castries Bay were sampled. No fish were collected at the first site.

At UWI Mona, the recoveries of the internal standard (i.e. PCB 30) varied between 72-113%, indicating that the analytes were extracted from muscle tissue with acceptable recoveries. Table 3 shows the mean concentrations of PCBs and organochlorine (OC) compounds in the 8 samples of the CRM that were analyzed at UWI Mona in comparison to the certified values for these selected compounds. These data indicate that the UWI Mona was reasonably accurate in analyzing the CRM samples, although, this regional laboratory tended to overestimate the concentrations of o,p-DDE and PCB congener 87, while underestimating the concentration of p,p-DDE, dieldrin and PCB congeners 52, 99, 153 and 180. Note that p.p-DDT was not detected in the CRM (Table 3), indicating that the UWI Mona laboratory experienced a problem with detecting this compound. The concentrations of PCBs and OCs were generally 2-3 orders of magnitude greater in the CRM (i.e. Lake Michigan fish) relative to the concentrations detected in white grunt. Therefore, the analysis of white grunt from the Caribbean presented a considerable technical challenge to the two regional laboratories. The compounds detected in white grunt were usually just above the analytical limits of detection.

Table 3: Mean concentrations of (n=8) of reference compounds in the Lake Michigan CRM compared to the certified values (µg/kg wet weight).

PCB congener	Concentration measured	Certified concentration	OC compound	Concentration measured	Certified concentration
31/28	16.2	10.4/14.1	HCB	5.2	7.5
44	9.6	20.4	αBHC	1.1	1.3
49	12.8	27.3	*trans*-chlordane	15.8	12.8
52	7.5	36.4	oxychlordane*	14.2	23.6/13.4
74	27.5	33.7	dieldrin	25.4	80.8
66/95	51.3	69.4	mirex	12.6	5.1
87	41.6	27.9	o,p-DDE	28.7	3.4
99	38.8	78	p,p-DDE	218.5	720
101	75.7	90.8	o,p-DDD	0.9	3.3
110	54.2	94.6	p,p-DDD	21.2	45.9
118	81.3	112	o,p-DDT	43.9	15.7
105/132	43.1	50.3/20.8	p,p-DDT	ND	59.5
128	26.1	31.6			
138	128.4	162			
149	40.1	67.1			
153	110.2	201			
156/171	15.4	13.3			
158	10.1	11.3			
180	50.9	80.8			
183	15.5	23.3			
187	36.2	54.8			
170/190	23.0	29.2			
194	13.9	13.2			
195/208	4.9	4.9			
206	5.1	6.2			

* plus heptachlor epoxide A

3.2 PCBs and organochlorines in white grunt:

The data generated from the regional laboratory at UWI Mona showed that PCB congeners and OC compounds were generally present in the muscle tissues of white grunt at concentrations <10 µg/kg wet weight (i.e. ppb). These low concentrations were expected, due to the low lipid content of the tissues and the low trophic position of white grunt within the marine food web. Marine fish with higher lipid contents, such as tuna or mackerel would likely accumulate higher concentrations of these lipophilic compounds (Uemo et al., 2004). However, these pelagic fish species are highly mobile and are not likely to provide an indication of regional or local patterns of POPs contamination.

Two pesticides, mirex and p,p-DDT were not detected in any of the tissues. However, it must be noted that the latter compound was also not detected in the CRM. Figure 2 shows the trends for mean levels of ∑PCB, ∑DDT, ∑BHC, ∑chlordane, ∑heptachlor, ∑endosulfan, HCB and methoxychlor in the white grunt collected from Jamaica, St. Lucia, and Trinidad and Tobago, respectively. These data indicate that the mean concentrations of these classes of compounds were relatively homogeneous across the sampling sites. However, the mean concentrations of methoxychlor, ∑PCB and ∑BHC were significantly higher in white grunt collected from St. Lucia.

For BHCs detected in white grunt from locations other than St. Lucia, ß-BHC was either the dominant congener, or the only one detected. This indicates that the BHCs originate from pesticide applications of “technical BHC”; a mixture of BHC isomers in which ßBHC is the dominant compound. ∑DDT was not present at high concentrations in any of the white grunt samples (Figure 2), and p,p’-DDE was the predominant compound detected from this class in white grunt samples. The presence of the DDE metabolite reflects transformation from DDT that was used at some time in the past, and shows that there has been no recent use of this insecticide. The structurally related compound, methoxychlor is used as a substitute for DDT because it is less persistent in the environment. Methoxychlor was observed in white grunt from St. Lucia at concentrations that were significantly elevated relative to levels in fish from the other two locations (Table 2).

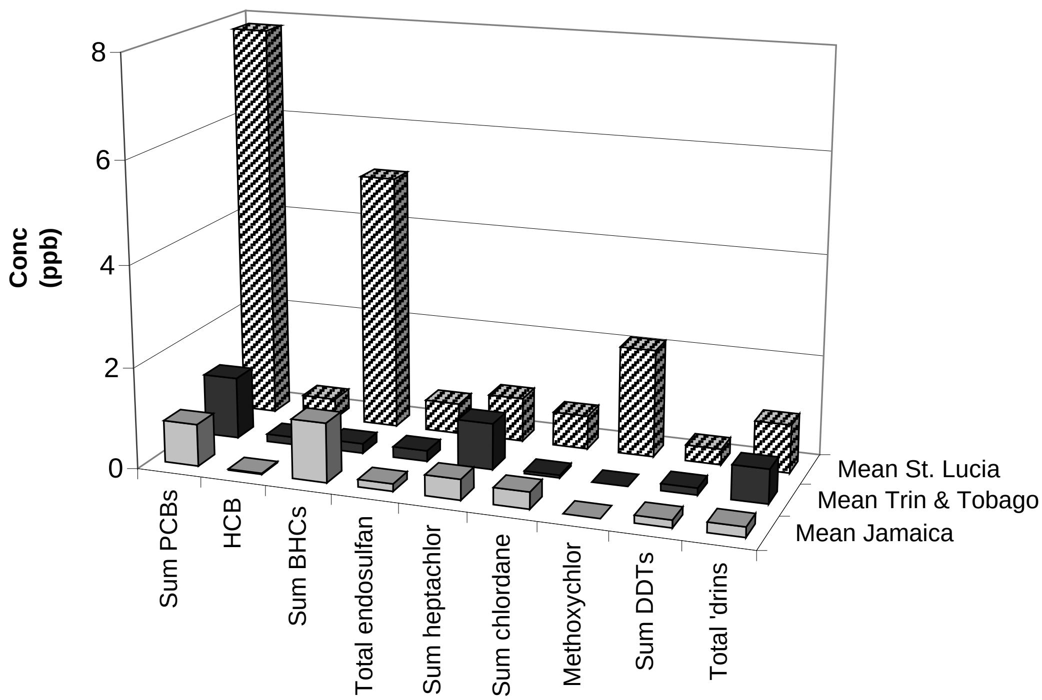

Figure 2: Mean concentrations (µg/kg wet weight) of classes of compounds analyzed in white grunt collected from all sites in Jamaica (n=19), St. Lucia (n=15), and Trinidad and Tobago (n=17), respectively.

Because of their recent or ongoing use for the control of insect pests, BHC and chlordane compounds have been detected in marine biota from tropical and subtropical countries in both the western and eastern hemispheres (Imo et al., 2008; Minh et al., 2006; Bayen et al., 2005; Norena-Barroso et al., 2004). Dieldrin has been detected in marine biota from other developing countries in the western hemisphere, such as Argentina (Menone et al., 2001), but the origin of this compound could be from the widespread use of the related insecticide, aldrin, which is rapidly transformed in the environment to dieldrin. Rainwater et al (2007) detected dieldrin, as well as DDE, DDT, endrin and methoxychlor in the caudal scutes of crocodiles sampled off the coast of Belize. However, these contaminants tend to occur at concentrations below the levels observed in fish from industrialized areas of the northern hemisphere. For instance, Table 3 shows the very high concentrations of selected certified OCs in the Lake Michigan fish CRM, including the very high concentration of p,p-DDE.

The differences in OC contamination observed in white grunt from the three Caribbean countries may reflect: i) geographical differences in the use of pesticides, ii) differences in overland or subsurface transport pathways from the source to the coastal zone, iii) marine circulation patterns and currents, and/or iv) patterns of atmospheric deposition of POPs in the Caribbean. However, a more complete data set is required to evaluate the factors that influence OC contamination in the Caribbean. Data are also required to evaluate pesticide use in these countries to determine whether white grunt contamination reflects local use of OC compounds.

To further investigate the trend of higher concentrations of PCBs and BHCs in white grunt from St. Lucia, the mean concentrations of several classes of compounds were determined in the white grunt (n=3 per site) collected from the five different sampling sites in St. Lucia (Figure 3). These data indicate that the levels of methoxychlor, HCB and ∑heptachlor were relatively homogeneous across all stations. However, the mean concentrations of ∑PCB and ∑BHC were significantly elevated in fish collected at Vieux Fort Black Bay (LC006). Although ∑endosulfan appeared to also be present at a higher mean concentration in white grunt collected at Vieux Fort Black Bay (Figure 3), this difference was not statistically significant. The significantly higher mean concentration of BHC in the samples from Vieux Fort Black Bay was primarily due to the very high ∑BHC concentration of 34.3 µg/kg wet weight observed in one of the 3 fish collected from this site (i.e. 26.2 µg/kg ßBHC, 6.9 µg/kg γBHC, 1.2 µg/kg δBHC). The ∑BHC concentrations in the other two fish from this location were 1.84 and 1.67 µg/kg, respectively. These data on BHC levels should be interpreted with caution until more fish are analyzed from this site.

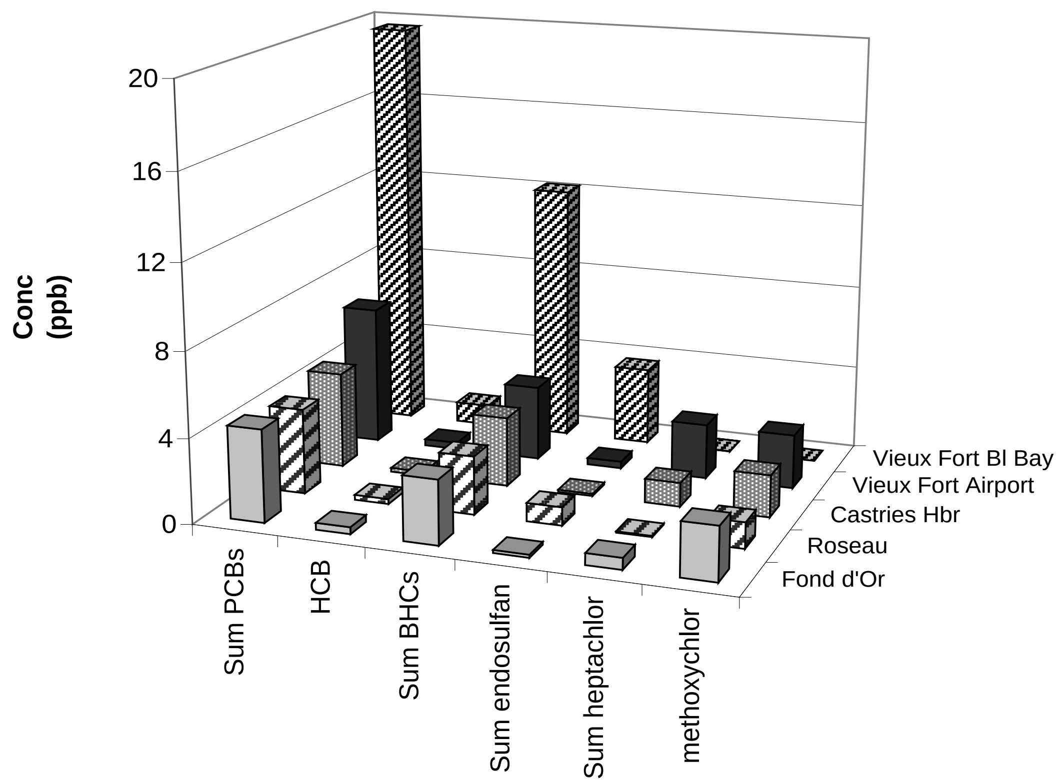

Figure 3: Mean concentrations (µg/kg wet weight) of classes of compounds analyzed in white grunt (n=3) collected from each of the 5 sites in St. Lucia.

Several PCB congeners were detected in extracts prepared from white grunt muscle tissue. Figure 4 shows the PCB congener pattern for 3 white grunt collected at the Vieux Fort Airport (LC008) site in St. Lucia, where the congener patterns were dominated by PCBs with a low degree of chlorination (i.e. tri-, tetra-, and penta-chlorobiphenyls). This pattern is typical of the PCBs detected in white grunt at other locations in the Greater Caribbean region, except for the 3 fish collected from Vieux Fort Black Bay in St. Lucia where more highly chlorinated PCB congeners were detected (Figure 4). The pattern of PCB congeners generally seen in white grunt from the greater Caribbean region (except for Vieux Fort Black Bay) indicates that the source of contamination is atmospheric deposition, since the less chlorinated PCB compounds are subject to transport in the atmosphere. However, the congener pattern observed in white grunt collected from Vieux Fort Black Bay in St. Lucia indicates that there is a point source of PCBs at this single site. A more extensive monitoring program may have identified other point sources of PCBs, but it was often difficult to collect white grunt at industrialized locations, such as Kingston Harbour (JM004) in Jamaica.

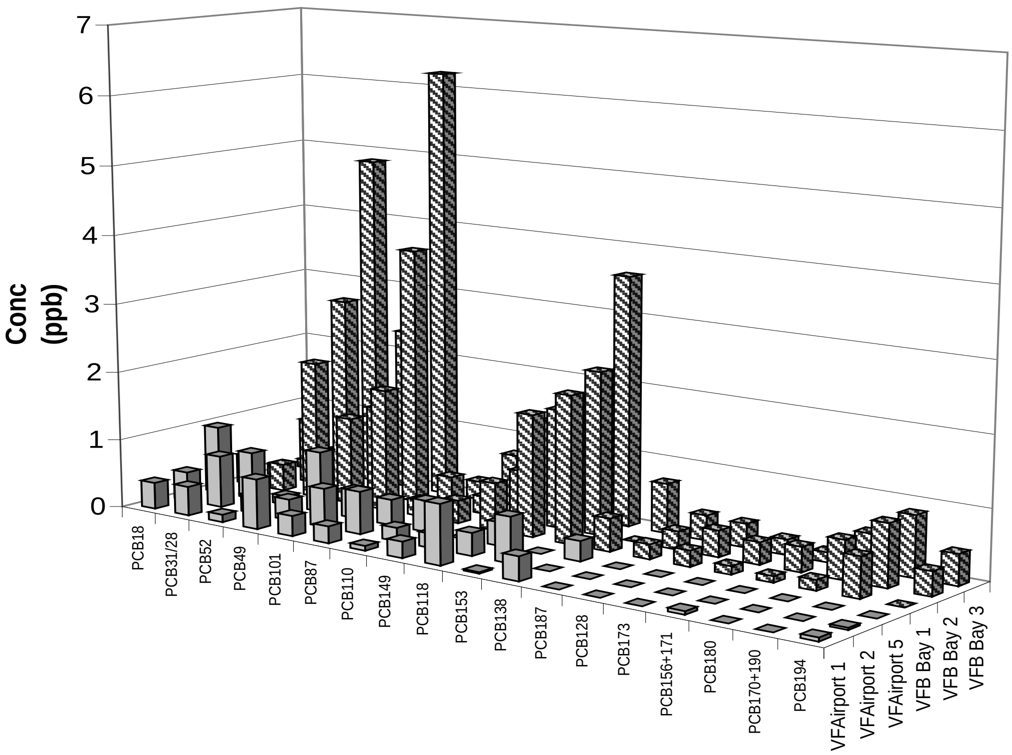

Figure 4: Concentrations (µg/kg wet weight) of major PCB congeners detected in the 3 white grunt collected from each of the Vieux Fort Airport (grey bars) and Vieux Fort Black Bay (black bars) sites in St. Lucia.

3.3 PBDEs in white grunt:

Analysis of samples prepared from five white grunt that were collected off the coast of Belize for PBDEs revealed that several congeners were present at detectable concentrations in the muscle tissue, including congeners 47, 77, 99, 85, 126, 153 and 184 (Figure 6). The mean and maximum total PBDE concentrations were 0.84 and 1.4 µg/kg wet weight, respectively, or 284 and 452 µg/g lipid weight, respectively. The concentrations on a lipid normalized basis are similar to fish from other regions of the world, and the congener pattern is the same as has been reported for marine fish from regions in the

Pacific, North America and Europe ((Minh et al., 2006; Brown et al., 2006; Ueno et al., 2004; Boon et al., 2002; Dodder et al., 2002).

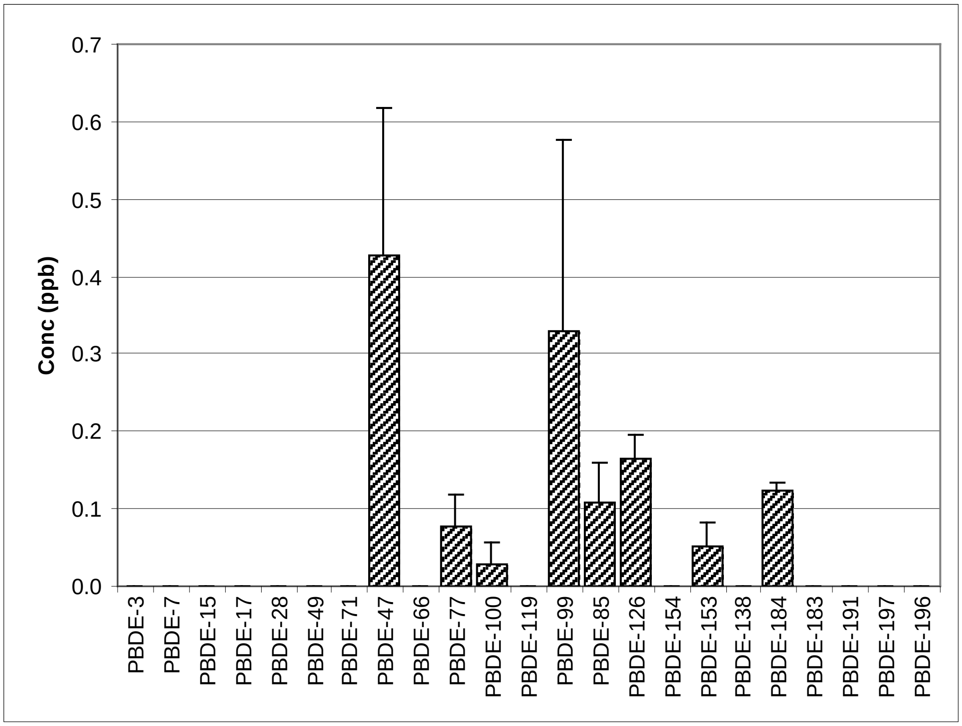

Figure 5: Mean concentrations (µg/kg wet weight) of PBDE congeners in white grunt (n=5) collected off the coast of Belize.

3.4 Fish consumption advisories:

The ∑PCB concentrations in the muscle of the 3 white grunt collected from Vieux Fort Black Bay were 12.8, 17.5 and 25.2 µg/kg wet weight, and ∑PCB concentrations in all other samples were less than 10 µg/kg wet weight. For comparison, the average measured ∑PCB concentration in the CRM of a fish from Lake Michigan fish was 1,079.5 µg/kg wet weight. Note that the concentrations of PCBs in the white grunt from Vieux Fort Black Bay are below the most restrictive fish consumption advisory for PCBs reported in the USA of 50 µg/kg wet weight (Table 4). The Health Canada fish consumption advisory for PCBs is 2,000 µg/kg wet weight. The summary data shown in Table 4 shows that none of the concentrations of compounds detected in white grunt approached even the most stringent of fish consumption advisory levels from the USA, or the higher advisories recommended by

Health Canada. Therefore, there are not likely to be any health impacts from the consumption of white grunt from these three regions of the Caribbean. The very low lipid content of the muscle tissues for this fish species contributed to the low concentrations of these lipophilic compounds.

Table 4: Mean and maximum concentrations and fish consumption advisory limits (µg/kg wet weight) for classes of organochlorine compounds and ∑PCB detected in white grunt muscle. The advisory levels reported are for the most stringent values from the USA, and where applicable, for higher values from Health Canada.

Chemical	**Jamaica**	**St. Lucia**	**Trinidad & Tobago**	**Advisory Limit**
Aldrin	0.07, 0.09	0.93, 0.84	0.45, 0.49	300
Endrin	0.21, 0.48	0.13, 0.14	0.42, 1.28	300
Dieldrin	0.14, 0.19	0.76, 1.28	0.05, 0.09	300
∑BHC	1.17, 4.91	5.07, 34.32	0.19, 0.84	100, 300
∑DDT	0.15, 0.51	0.32, 1.25	0.14, 0.32	5000
∑chlordane*	0.33, 0.98	0.67, 1.27	0.05, 0.15	300, 5620
HCB	0.02, 0.11	0.41, 1.25	0.15, 0.21	10, 100
∑PCB	0.83, 2.01	7.83, 25.24	1.22, 3.20	50, 2000

* plus heptachlor

Conclusions:

Overall, these data indicate that contamination by POPs in white grunt is not likely to be a health risk to fish consumers in the four Caribbean countries from which the samples were collected. However, it must be emphasized that these are only preliminary data from a relatively small number of fish collected from three of the eight partner countries. These preliminary data do indicate that atmospheric sources of contamination may be responsible for contamination by some compounds, but point sources may contribute to contamination at selected sites. More data are required to determine whether there are geographic and regional trends in the distribution of POPs in this region. In order to evaluate local trends in the distribution of POPs at some locations (e.g. Vieux Fort Black Bay) it may be appropriate to use other monitoring methods, such as passive sampling (O'Toole et al., 2006) or deployment of bivalves (Gewurtz et al., 2002).

It would be useful to determine the distribution of POPs in biota from the entire food web in the Caribbean, including fish species that have a higher trophic status and/or have a high lipid content in their tissues (Ueno et al., 2004). Other food web studies have shown that marine crustaceans can accumulate relatively high concentrations of POPs, including crabs (Bayen et al., 2005; Menone et al., 2001) and spiny lobster (Coat et al., 2006). Future work should also focus on determining whether there is contamination of white grunt and other marine biota from the wider Caribbean region by persistent organic pollutants that were recently added to the Stockholm Convention list (e.g. PBDEs, PFOS, chlordecone), and persistent and toxic substances of emerging interest (e.g. mercury, synthetic musks, "new" brominated flame retardants). However, it must be pointed out that this report contains the ONLY data that are currently available on POPs contamination in fish distributed across the wider Caribbean region, and therefore, are a valuable contribution to the literature on contamination of marine resources.

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