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Impacts of Cooking Technique on Polychlorinated Biphenyl and Polychlorinated Dioxins/Furan Concentrations in Fish and Fish Products with Intake Estimates

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ABSTRACT: Polychlorinated biphenyl (PCB) and polychlorinated dibenzo-*p*-dioxin (PCDD) and dibenzofuran (PCDF) concentrations were determined in composites of 18 different fish products and were prepared as raw, baked, boiled, and fried. ∑PCB concentrations were found to range from 0.12 ng·g^{-1} whole weight (ww) in raw octopus to 33 ng·g^{-1} ww in baked mackerel. Boiled monkfish was found to have the lowest ∑PCDD/F concentrations (0.41 pg·g^{-1} ww), while maximum concentrations were observed in fried catfish (59 pg·g^{-1} ww). PCB and PCDD/F concentrations in fish were generally reduced during cooking, although differences were small. The average PCB reduction in finfish was 7.9%, while an increase in PCB mass was observed in non-finfish (2.9%). PCDD/F losses, on average, were observed in both the finfish (3.6%) and non-finfish products (25%). Maximum ∑PCB, ∑PCDD/F, and $TEQ_{PCDD/F+DL-PCB}$ (toxic equivalency) intakes, based on 150 g serving size, were determined to be 3300 ng (mackerel), 6600 pg (catfish), and 270 pg (catfish), respectively. PCB and PCDD/F changes associated with cooking generally were small (<15%), although larger mean differences (~40%) were observed in some fish products (e.g., catfish).

KEYWORDS: *PCBs, PCDD/Fs, fish, shellfish, cooking*

INTRODUCTION

Persistent organic pollutants (POPs), including the lipophilic polychlorinated biphenyls (PCBs), and polychlorinated dibenzo-*p*-dioxins (PCDDs) and dibenzofurans (PCDFs), have been observed in fish worldwide.[1] Biomagnification of PCBs and PCDD/Fs results in concentrations observed in piscivorous fish exceeding those in fish lower in the food chain.[2,3] In contrast to the potential negative effects of PCB and PCDD/F exposure, including immunotoxicity and carcinogenicity,[4] fish consumption by humans is associated with many health benefits (e.g., improvement in vascular health and reduced heart-related problems).[5,6] The association between fish consumption and these positive health outcomes may contribute to the continued popularity of consuming fish and fish products.

Although numerous studies have been performed to determine PCB and PCDD/F levels in fish, they generally investigate levels in raw fish alone, which does not allow the impact of food preparation on dietary exposure to be examined.[7−11] The Canadian Total Diet Study (TDS) is performed annually to more realistically estimate dietary exposure because the contaminant concentrations are measured in food prepared as for consumption.[12,13] Within the TDS, composite samples are prepared to represent many food types (e.g., fish, meat, vegetables). Among the food composites prepared in the TDS program are three samples of finfish and one of shellfish. The finfish samples are prepared according to type: (i) marine, which is comprised of cod, flatfish, and haddock; (ii) freshwater (e.g., different species of trout); and (iii) canned fish (salmon and tuna), while the shellfish composite contains shrimp exclusively. These composites were developed to provide dietary exposure information, based on food consumption patterns observed in the 1970s. Fish consumption patterns in Canada, however, have been changing over the past few decades, likely corresponding to immigration from Asian and other high fish-consuming countries. North Americans of Asian origin are thought to consume greater amounts and different species of fish than the general population.[14] The samples included in the TDS, therefore, may not accurately reflect the exposure to PCBs and PCDD/Fs via fish and shellfish consumption for this subset of the Canadian population.

The impact of cooking fish on chemical contaminants (e.g., POPs, radionuclides, and mercury) has been investigated by

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some authors,[15−19] but these studies frequently focus on a single species of fish or fish from a specific catchment area and, therefore, result in directed information for a select group of individuals. Sherer and Price[20] developed a detailed review of the literature where the impacts of cooking fish on PCB concentrations were reported, and they described some of the challenges associated with relating data from one study to that of another. The impact of cooking on POP concentrations in food seems to be inconsistent: some report decreases in concentrations, due to concomitant loss of lipid, whereas other studies found that concentrations increase.[16,18,20,21]

Given the probability that a growing number of Canadians consume fish that are not included in the TDS, this study was established to determine contaminant concentrations in fish and fish products consumed by a subset of the population that are considered to be high consumers of fish. Fish types included in the study were those that have not been the focus of previous Health Canada investigations.[9,22] A sufficient amount of each fish product type was collected to ensure that samples could be subdivided and cooked using up to three different techniques (e.g., baking, boiling, frying), in addition to retaining raw portions of each fish type for analysis.

MATERIALS AND METHODS

Species Selection. The Canadian Food Inspection Agency (CFIA) identified the most frequently imported fish and fish products into Canada by use of annual import data between 2001 and 2004 as a proxy for consumption data. Import quantities were corrected for waste to estimate the relative amount consumed in relation to the amount purchased.[22] Fish products were then ranked on the basis of import quantity in decreasing order, and fish types routinely included in the TDS were removed from the list and not included in the present study. Fish products belonging to 18 species (catfish, cherrystone clams, conch, cuttlefish, grey mullet, grouper, mackerel, milkfish, monkfish, octopus, red snapper, scallops, sea squirt, silver pomfret, skate, squid, whiting, and yellow croaker) were selected for collection as part of this study.

Sample Collection and Preparation. Sample collection was performed in Asian-Canadian supermarkets and fish markets in Toronto, Mississauga, and Ottawa, Ontario, Canada, between February and April 2006. Three samples of each fish product type from three different markets were collected as a minimum (i.e., nine individual samples) from which to prepare composites. Upon collection, samples were packed on ice and shipped to Kemptville College, University of Guelph, Kemptville, Ontario.

Asian Canadians include the skin during preparation for some types of fish, although Canadians from other backgrounds may not.[23] For this reason, the skin was retained with the fish samples during preparation for analysis. All fish products (i.e., catfish, cuttlefish, grey mullet, grouper, mackerel, milkfish, monkfish, octopus, red snapper, scallops, sea squirt, silver pomfret, skate, squid, whiting, and yellow croaker) were prepared for cooking by removing the head, organs and bones, as appropriate. Some of these products were collected as fillets and, therefore, this step was not required in the sample preparation. Shellfish (cherrystone clams and conch) were prepared for cooking by removing the shell. The eyes, mouth, foot, snout, nail, feet and intestinal sacs also were removed from conch samples prior to cooking and homogenization.

An aliquot of each fish type was retained raw, as a control, to allow for comparison of PCB and PCDD/F concentrations relative to the equivalent cooked samples. Finfish samples were prepared by three cooking techniques (baking, boiling, and frying), while non-finfish (shellfish and other seafood) were baked and boiled only (Table 1). Baked finfish were prepared by marinating the fish in rice wine (ratio of 1 part wine to 40 parts of fish) and then baking at 375 °C for 15 min. Non-finfish were baked in dishes that had been greased with cooking oil at 325 °C for 20 min, or until tender. Finfish were cut up into 1-in. cubes and fried in sesame oil (ratio of 1 part oil to 16 parts of fish) for 10−15 min in a wok. Both finfish and non-finfish were boiled in water until firm, with water to fish ratios of 30:1 and 35:1 (v/v) for finfish and non-finfish, respectively. All samples were homogenized in a commercial blender with stainless steel blades and transferred to solvent-rinsed glass sample containers with polytetrafluoroethylene (PTFE) lined lids. Samples were then frozen at −20 °C until extraction and analysis commenced.

Sample Extraction. Thawed fish products (30 g) were spiked with 25 μL of surrogate standard solutions containing 2−4 pg·μL^{-1} $^{13}C_{12}$ PCDD/Fs and 25 μL of the standard solution containing 1000 pg·μL^{-1} 35 $^{13}C_{12}$ PCBs, prior to extraction. Surrogate standards were purchased from Wellington Laboratories (Guelph, ON, Canada). The method employed for the extraction and cleanup of samples was that reported by Rawn et al.[9] Briefly, samples were extracted by homogenization with acetone/hexane (2:1). The extracts were then partitioned with 60 mL of distilled deionized water. The hexane fraction was then evaporated to dryness to determine lipid content and taken up to ∼0.5 mg·mL^{-1} lipid in dichloromethane (DCM). Lipids were removed from the extracts via an Agilent high-performance preparative gel-permeation chromatography system (New Castle, DE), consisting of an 1100 Series quaternary pump, autosampler, and fraction collector. Two Waters Envirogel columns (150 mm × 19 mm and 300 mm × 19 mm) were used in series. The mobile phase was DCM with a flow rate of 5 mL·min^{-1}. Lipid content was determined gravimetrically.

Cleanup was completed by use of acidified treated silica, topped with anhydrous sodium sulfate and eluted with 70 mL of hexane. The eluent was concentrated, by rotary evaporation, to approximately 2 mL. These extracts were then passed through 1.5 g of activated Florisil, from which most PCBs were eluted with 40 mL of hexane and PCDD/Fs and coplanar PCBs were eluted with 60 mL of DCM.

The fraction containing the PCDD/Fs and non-ortho-PCBs, which required additional cleanup, was evaporated just to dryness and diluted to 1 mL with hexane. This fraction was cleaned up further with 0.4 g of 18% activated carbon/Celite. The column containing carbon was eluted first with 2 mL of hexane, followed by 2 × 1 mL volumes of DCM/cyclohexane (1:2), and finally 60 mL of toluene.

PCB and PCDD/F fractions were then prepared for analysis by concentrating just to dryness and diluting to final volumes of 25 μL (PCB fraction) and 10 μL (PCDD/F fraction) in toluene with performance standards containing $^{13}C_{12}$ analogues of PCBs and PCDD/Fs in the corresponding fractions.

Lipid Determination. Lipid content was determined gravimetrically, meaning that raw sample extracts were added to preweighed round-bottomed flasks and concentrated initially by rotary evaporation, followed by gentle blowing with a stream of nitrogen until dryness was achieved. Dryness was evidenced by the determination of constant weight following multiple measurements; the weight difference was taken to be the lipid weight of the sample and related back to the whole sample weight. From this, the lipid content was calculated as a percent of sample weight.

Analysis. Both fractions (PCB and PCDD/F) were analyzed on a Waters AutoSpec Premier high-resolution mass spectrometer (Milford, MA) linked to an Agilent 6890 Series II gas chromatograph (GC) (Palo Alto, CA) equipped with a splitless injection system. Injection volumes were 1.5 μL for all analyses. The column used for the GC separation was a fused silica DB-5 60 m × 0.25 mm × 0.25 μm (J&W Scientific, Folsom, CA) with a 1 m × 0.53 mm (J&W Scientific) retention gap. The injector temperature was set to 300 °C for all analyses, with a purge time of 1.5 min. Initially, the oven was set to 100 °C and held for 1.5 min, raised to 200 °C at 30 °C·min^{-1} and increased to 235 °C at 3 °C·min^{-1} and held for 10 min, with a final temperature increase of 6 °C·min^{-1} to 300 °C, which was held for 18 min for the analysis of the PCB fraction. The analysis of PCDD/Fs and non-ortho-PCBs was performed with the oven at 80 °C and held for 1.5 min, followed by an increase to 200 °C at a rate of 30 °C·min^{-1}, followed by a 5 °C·min^{-1} increase to 280 °C and held for 3 min, and the final increase to 300 °C was performed at a rate of 15 °C·min^{-1} and then held for 13 min.

The electron energy was set to 35 eV, with a multiplier voltage of 360 V for all analyses. The trap current was 640 μA, and both the source and capillary line temperatures were maintained at 260 °C. The re-entrant temperature was 280 °C, and perfluorokerosene-L (PFK) was used as the reference substance for tuning at m/z 293. The mass resolution was set to 10 000 for all analyses.

Quality Assurance. The analyses described in the present study were performed within a quality management framework following standard approaches to ensure that accurate measurements are developed. A reagent blank and a laboratory sample of butter, with known PCB and PCDD/F concentrations that has been tested many times within our laboratory, was included as an internal quality assurance sample with each set of six samples of unknown concentration analyzed. When detected, PCB and PCDD/F concentrations observed in reagent blanks were used to correct for laboratory background concentrations within each set individually. The quality control sample tested with each set of samples analyzed was found to consistently result in PCB and PCDD/F concentrations within 2 standard deviations of the measured mean concentration. The internal quality assurance system employed in the laboratory was confirmed through successful participation in the Norwegian Institute of Public Health international interlaboratory study, where several classes of POPs, including PCBs and PCDD/Fs in various foods, are examined.[24]

Within each set of samples, one sample was selected and prepared for analysis in duplicate, as a means of testing reproducibility and to provide an estimate of uncertainty between measurements. The percent difference in ∑PCB concentrations between duplicate samples was determined to range from below 1% (for octopus) to 23% (for red snapper). Higher percent differences in ∑PCDD/F concentrations (from 11% for octopus to 35% for mackerel) were established when the duplicate analyses were compared, consistent with the relatively lower concentrations of PCDD/Fs. The mean percent difference in concentrations obtained from duplicate analyses were 10% ± 6.7% for PCB and 21% ± 6.2% for PCDD/F.

The average limits of detection (LOD) in the fish samples for PCBs ranged from 0.06 pg·g^{-1} (PCB 205) to 4.5 pg·g^{-1} (PCB 40), while PCDD/F LODs ranged from 0.01 pg·g^{-1} (1,2,3,6,7,8-HxCDF) to 0.02 pg·g^{-1} (OCDF). Limits of detection were determined on the basis of a 3:1 signal to baseline noise ratio.

The average recoveries of PCBs from the fish samples analyzed ranged from 30% (PCB 1) to 87% (PCB 205). OCDD had the lowest average recovery in the finfish and non-finfish samples tested (44%), while 2,3,7,8-tetrachlorodibenzodioxin (TCDD), 1,2,3,7,8 pentachlorodibenzodioxin (PeCDD), 1,2,3,4,7,8-hexachlorodibenzodioxin (HxCDD), 2,3,4,6,7,8-hexachlorodibenzofuran (HxCDF), and 1,2,3,7,8,9-HxCDF all had maximum average recoveries (86%). PCB and PCDD/F concentrations in the unknown samples were recovery-corrected for reporting. Toxic equivalency ($TEQ_{PCDD/F}$ or $TEQ_{DL\text{-}PCB}$, where DL is dioxin-like) was calculated from toxic equivalency factors determined in 2005.[25]

RESULTS AND DISCUSSION

Concentrations and Trends. PCBs and PCDD/Fs were detected in all fish products analyzed in the present study. ∑PCB concentrations are reported as totals of a sum of 82 congeners, observed as 79 peaks, due to coelution of PCB 4 with 10, as well as PCB 84 coeluting with PCBs 90 and 101. Given that there are a limited number of congeners that contribute to the majority of the ∑PCB concentrations, such as the six indicator PCBs in the fish samples studied, the data were able to be compared with residue levels obtained in other similar studies. ∑PCB concentrations ranged from 0.12 ng·g^{-1} wet weight (ww) in raw octopus to 33 ng·g^{-1} ww in baked mackerel. Boiled monkfish was found to have the lowest ∑PCDD/F concentrations (∑2,3,7,8-substituted congeners) (0.41 pg·g^{-1} ww), and highest ∑PCDD/F concentrations were observed in fried catfish (59 pg·g^{-1} ww).

The hexachlorinated PCBs were the dominant homologue group contributing to ΣPCB concentrations in all fish sample types studied (from 25% for milkfish to 57% for silver pomfret). The penta- and heptachlorinated congeners (23% and 17%, respectively) were similar in their relative contribution to total PCB concentrations, followed by the tetrachlorinated PCB congeners (10%). PCBs 153 and 138 were the largest individual congener contributors to PCB concentrations in the fish products tested (mean contributions of 13.4% and 10.7%, respectively). In our previous work, PCBs 153 and 138 similarly were found to be the dominant contributor to ΣPCB concentrations in fish products (e.g., salmon, crab) purchased in Canada.[9] Minh et al.[8] observed a higher contribution to PCB concentrations from the hexachlorinated PCB congeners in catfish samples from Vietnam, followed by tetra- and pentachlorinated homologues, whereas fish from Italy were dominated by tetra- and pentachlorinated congeners.[3]

The ∑PCB concentration observed in raw catfish from the Canadian market in the present study (11 ng·g^{-1} ww [85 ng·g^{-1} lipid]) was higher than the concentrations reported in common catfish from Vietnam (0.91−27 ng·g^{-1} lipid).[8] Similarly, the levels measured in mackerel in the present study (22−33 ng·g^{-1} ww) (Table 1) were higher than reported in mackerel (3.9−4.1 ng·g^{-1} ww) collected as part of a market basket study in Belgium.[26]

OCDD was the dominant contributor (7.2−77%; mean 28%) to ΣPCDD/F concentrations in the fish analyzed in the present study. Other large contributions to ΣPCDD/F concentrations were from 1,2,3,4,6,7,8-HpCDD (14%), 2,3,7,8-TCDF and 2,3,4,6,7,8-HxCDF (10%). OCDD (19%) and 2,3,7,8 TCDF (13%) were similarly found to be dominant congeners in fatty fish samples from China.[27] Schecter et al.[16] reported that OCDD was the dominant contributor to ΣPCDD/F levels in raw catfish from the United States (61%). Total PCDD/F concentrations observed in raw mackerel collected as part of the present study (2.0 pg·g^{-1} ww) were slightly higher than reported for mackerel collected in China (0.58 pg·g^{-1} ww).[27,28]

The $TEQ_{PCDD/F}$ concentration in the mackerel from the present study (0.29 pg $TEQ_{PCDD/F}$·g^{-1} ww) was similar to concentrations reported for wild mackerel collected in the United Kingdom (U.K.) (0.42 pg $TEQ_{PCDD/F}$ ·g^{-1} ww).[29] The $TEQ_{PCDD/F}$ observed in raw whiting (0.06 pg $TEQ_{PCDD/F}$·g^{-1} ww) and scallops (0.08 pg $TEQ_{PCDD/F}$·g^{-1} ww) in the present study were similar to the reported concentrations in the United Kingdom (0.04 pg $TEQ_{PCDD/F}$·g^{-1} ww for whiting and 0.05 pg $TEQ_{PCDD/F}$·g^{-1} ww for scallops).[29]

PCDD/F and PCB concentrations in all samples analyzed in the present study (Table 1) were below European Commission maximum concentrations established for fish and fishery products [3.5 pg WHO-$TEQ_{PCDD/F}$·g^{-1} ww, 6.5 pg WHO-$TEQ_{PCDD/F+DL\text{-}PCB}$·g^{-1} ww, and 75 ng·g^{-1} ww ∑6 indicator PCB (28, 52, 101, 138, 153, 180) congeners].[30] The Canadian standard of 2 μg·g^{-1} ww PCBs in fish, which is currently under review,[31] was not exceeded in any fish samples tested. The 20 pg·g^{-1} ww tolerance for 2,3,7,8-TCDD in fish, identified in B.01.047(f) of the Canadian Food and Drug Regulations, was not exceeded in any of the fish products tested in the present study. Although this tolerance still is in place, it is considered to be outdated and no longer used as a health-based standard and is also currently under review by Health Canada.[31]

Table 1. PCB, PCDD/F, and TEQ Concentrations in Fish Samples, Sorted by Type and Cooking Treatment

	% lipid	∑indicator PCBs[a] (ng·g^{-1} ww)	∑PCB[b] (ng·g^{-1} ww)	∑PCDD/F[c] (pg·g^{-1} ww)	$TEQ_{DL\text{-}PCB}$[d] (pg·g^{-1} ww)	$TEQ_{PCDD/F}$[d] (pg·g^{-1} ww)
			Finfish			
catfish						
raw	12.5	3.7	11	44	0.70	1.1
baked	12.2	3.3	9.5	31	0.55	0.80
boiled	9.56	3.6	11	31	0.61	0.87
fried	22.9	4.2	13	59	0.51	1.5
croaker						
raw	7.00	4.4	13	0.9	0.26	0.07
baked	11.1	5.4	15	1.0	0.59	0.07
boiled	5.86	4.3	12	0.69	0.43	0.04
fried	16.8	5.5	16	*e*	0.32	*e*
grey mullet						
raw	5.60	5.1	15	6.7	0.54	0.84
baked	7.50	3.5	9.9	6.9	0.39	0.88
boiled	6.60	3.6	10	11	0.42	1.1
fried	13.2	5.2	15	5.5	0.54	0.71
grouper						
raw	2.29	5.2	12	4.8	0.48	0.29
baked	2.43	7.8	18	3.3	0.48	0.30
boiled	2.24	7.9	18	3.3	0.49	0.29
fried	7.92	9.1	21	6.6	0.59	0.46
mackerel						
raw	17.4	6.8	22	2.0	0.82	0.29
baked	21.4	12	33	6.0	1.2	0.61
boiled	23.0	9.3	31	2.8	0.96	0.39
fried	29.8	10	29	6.1	1.1	0.59
milkfish						
raw	9.31	0.28	0.82	1.7	0.07	0.15
baked	10.7	0.33	1.0	3.0	0.08	0.23
boiled	10.6	0.34	1.1	2.1	0.08	0.17
fried	16.7	0.33	1.0	1.5	0.08	0.17
monkfish						
raw	0.47	0.54	1.2	0.49	0.05	0.06
baked	0.90	1.0	2.5	0.60	0.10	0.08
boiled	0.86	0.69	1.6	0.41	0.07	0.05
fried	1.43	1.3	3.1	0.66	0.12	0.10
pomfret						
raw	4.83	1.6	6.1	1.2	0.24	0.18
baked	5.83	1.6	6.3	2.1	0.24	0.15
boiled	5.92	2.0	8.3	3.0	0.31	0.15
fried	12.9	2.0	8.4	2.9	0.34	0.15
red snapper						
raw	1.15	1.1	2.5	0.56	0.09	0.07
baked	1.76	1.5	3.5	0.52	0.12	0.08
boiled	1.50	1.3	3.0	0.43	0.10	0.06
fried	7.71	1.3	3.0	0.67	0.10	0.07
whiting						
raw	2.10	3.8	10	0.65	0.18	0.06
baked	2.02	3.9	9.8	0.65	0.18	0.06
boiled	2.20	4.2	11	0.71	0.20	0.07
fried	8.31	4.3	11	0.82	0.21	0.08
			Non-finfish			
cherrystone clam						
raw	0.43	5.0	17	5.3	0.43	0.62
baked	1.78	4.8	16	8.0	0.41	0.51
boiled	1.64	5.0	17	9.3	0.42	0.59
conch						
raw	0.43	0.43	0.91	1.2	0.05	0.18
baked	1.08	0.62	1.4	1.3	0.08	0.18
boiled	0.77	0.38	0.84	1.5	0.06	0.21

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Table 1. continued

	% lipid	∑indicator PCBs[a] (ng·g^{-1} ww)	∑PCB[b] (ng·g^{-1} ww)	∑PCDD/F[c] (pg·g^{-1} ww)	$TEQ_{DL\text{-}PCB}$[d] (pg·g^{-1} ww)	$TEQ_{PCDD/F}$[d] (pg·g^{-1} ww)
			Non-finfish			
cuttlefish						
raw	0.73	0.08	0.16	1.0	0.05	0.18
baked	1.59	0.14	0.28	1.3	0.05	0.22
boiled	1.48	0.17	0.32	1.5	0.05	0.22
octopus						
raw	0.59	0.05	0.12	0.83	0.10	0.09
baked	2.68	0.15	0.38	1.2	0.18	0.10
boiled	2.08	0.16	0.41	1.2	0.19	0.10
scallop						
raw	0.43	0.05	0.16	0.69	0.10	0.08
baked	1.10	0.13	0.38	0.73	0.13	0.08
boiled	0.83	0.13	0.42	0.72	0.20	0.09
sea squirt						
raw	2.20	0.18	0.50	6.3	0.03	0.32
baked	3.45	0.23	0.63	8.1	0.04	0.39
boiled	3.00	0.30	0.84	10	0.06	0.47
skate						
raw	0.50	1.0	2.5	1.2	0.01	0.16
baked	1.70	1.1	2.8	1.4	0.02	0.19
boiled	0.80	1.1	2.8	1.5	0.02	0.21
squid						
raw	1.56	0.13	0.35	0.52	0.61	0.07
baked	3.75	0.26	0.68	0.52	0.64	0.07
boiled	4.60	0.34	0.90	0.62	0.73	0.08

[a]Sum of PCBs 28, 52, 101 (coeluted with 84 and 90), 138, 153, and 180. [b]Sum of PCBs 1, 3, 4/10, 6, 8, 15, 16, 18, 19, 22, 28, 31, 33, 37, 40, 41, 44, 49, 52, 54, 60, 66, 70, 74, 77, 81, 84/90/101, 85, 87, 95, 97, 99, 104, 105, 110, 114, 118, 119, 123, 126, 128, 129, 135, 137, 138, 141, 149, 151, 153, 155, 156, 157, 158, 167, 168, 169, 170, 171, 174, 177, 178, 180, 183, 187, 188, 189, 191, 193, 194, 199, 200, 201, 202, 203, 205, 206, 207, 208, and 209. [c]Sum of seven 2,3,7,8-substituted PCDD congeners and 10 2,3,7,8-substituted PCDF congeners. [d]TEQ determined from 2005 TEFs.[25] [e]Sample lost.

Impact of Cooking on Concentrations. Highest ΣPCB concentrations were observed in the fried samples for six of the 10 finfish collected (catfish, croaker, grouper, monkfish, pomfret, and whiting), while two had highest concentrations in the baked fish (mackerel and red snapper), and concentrations in grey mullet were the same in the fried and raw composites. Although concentration differences between raw and cooked fish were observed, they were generally small (Table 1). In most cases, the difference in concentration associated with cooking type (e.g., boiling versus frying) was small (e.g., red snapper, whiting) although a larger difference was observed for the mackerel, which may be related to the higher lipid content in this fish (Table 1). Maximum ΣPCB concentrations observed in the boiled milkfish were similar to the baked and fried samples, with slightly lower concentrations in the raw sample (Figure 1A). Similar to the finfish tested, only small concentration differences were observed in the non-finfish products as a result of cooking. In contrast, maximum ΣPCB concentrations were found in the majority of the boiled non-finfish product composites tested (cuttlefish, octopus, scallops, sea squirt, and squid), while the baked composite had the highest ∑PCB concentrations in the conch (Figure 2A). Similar to the impact of cooking on the ∑PCB concentrations, maximum ΣPCDD/F concentrations were observed in the fried samples for six of the finfish tested (catfish, grouper, mackerel, monkfish, red snapper, and whiting) (Table 1). All of the non-finfish, with the exception of octopus and scallops, had the highest PCDD/F concentrations in the boiled composite. The ∑PCDD/F concentration in the baked scallop was very similar to the boiled composite (Figure 2B), and the baked and boiled octopus composites had the same concentration (1.2 pg·g^{-1} ww).

Numerous studies have been performed to look at the impacts of cooking on POP levels in foods, many of which have been focused on fish and shellfish tissue.[18,19,32−38] In the majority of these studies, a reduction in PCB, PCDD/F, and organochlorine pesticide concentrations has been observed; however, some studies have reported an increase or no change to the concentrations following cooking relative to levels observed in raw samples.[34,37]

The approach individual authors have used for reporting impacts of cooking to POP concentrations has been identified as a source of potential confusion. Changes in POP concentration have been reported in the literature on both a whole weight basis as well as based on the amount lost per gram of fat (i.e., lipid-adjusted values). Sherer and Price[20] proposed that the effect of cooking fish should consider not only the difference in PCB concentrations but also factor in the difference in fish mass as a result of cooking. This step would ensure that losses of lipids and water are included in the investigation. Following the proposal of Sherer and Price,[20] the effect of cooking on PCB and PCDD/F concentrations in the fish products in the present study was determined by use of the following equation:[35]

$$\text{PCX change}_{\sum \text{mass}} = \frac{[\text{PCX}_{\text{raw}}](\text{mass}_{\text{raw fillet}}) - [\text{PCX}_{\text{cooked}}](\text{mass}_{\text{cooked fillet}})}{[\text{PCX}_{\text{raw}}](\text{mass}_{\text{raw fillet}})}$$

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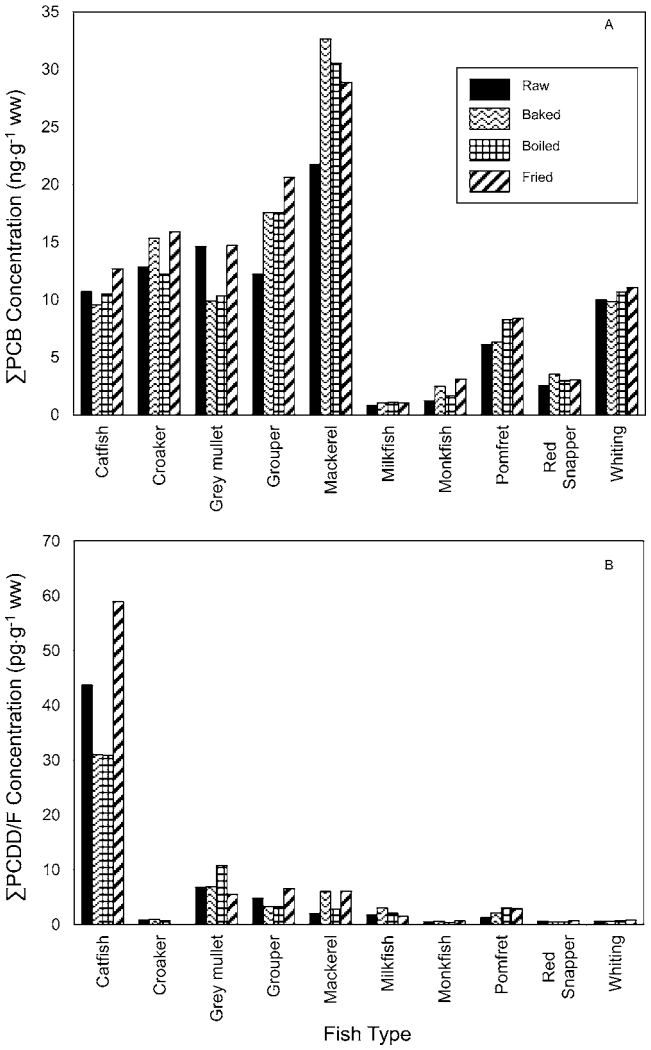

Figure 1. (A) ∑PCB (ng·g^{-1} ww) and (B) ∑PCDD/F (pg·g^{-1} ww) concentrations in raw and cooked (baked, boiled, fried) finfish samples.

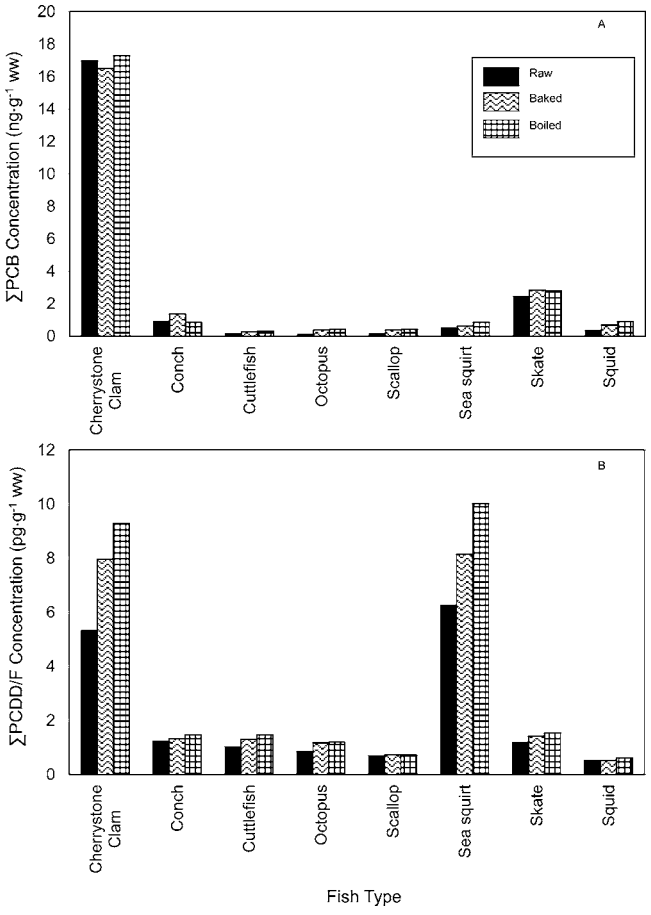

Figure 2. (A) ∑PCB (ng·g^{-1} ww) and (B) ∑PCDD/F (pg·g^{-1} ww) concentrations in raw and cooked (baked, boiled) non-finfish samples.

where PCX change $_{\sum mass}$ is the total PCB or PCDD/F mass change during cooking; [PCX$_{raw}$] and [PCX$_{cooked}$] represent the PCB or PCDD/F concentration in raw and cooked fish, respectively; and mass$_{raw\ fillet}$ and mass$_{cooked\ fillet}$ are the masses of the fish fillet before and after cooking, respectively. The whole weight concentrations were used for these calculations. The results were then multiplied by 100 to report differences as a percentage of the total PCB or PCDD/F mass change during cooking.[20]

In the majority of samples analyzed in the present study, small changes in concentration were observed following cooking relative to the concentrations in the raw fish (Table 1). By use of the relationship described above, the average PCB reduction in finfish was 7.9%, while an increase in PCB mass (average = 2.9%) was observed in non-finfish (shellfish and other seafood) (Table 2). Frying of finfish resulted in the highest average reduction to ∑PCBs (12%), while boiling resulted in maximum average reduction to ∑PCBs in non-finfish (0.8%). A PCB mass increase was observed in nine of the baked fish products (five finfish and four non-finfish) and 10 of the boiled fish products (five each of finfish and non-finfish), while a mass loss was observed in all of the fried fish, with the exception of fried monkfish (Table 2; negative values indicate an increase as determined by use of the equation listed above).

The average PCDD/F mass reductions from cooking were 3.6% in finfish and 25% in non-finfish (Table 2). Baking non-finfish resulted in the greatest average ∑PCDD/F reductions (29%), whereas boiling resulted in the maximum average ∑PCDD/F mass reductions (8.5%) in finfish (Table 2). In the present study, five of the 18 fish were observed to have a net PCDD/F mass increase resulting from baking (three finfish and two non-finfish), while a total of six boiled fish samples (four finfish and two non-finfish) had increased PCDD/F masses (Table 2). Schecter et al.[16] similarly observed both increases and decreases in PCDD/F and non-ortho-PCB concentrations in catfish, bacon, and hamburger.

Small PCB mass changes (<10%) were observed as a result of cooking for several fish product types, and frequently these differences were below the determined uncertainty associated with these analyses, based on the duplicate analyses of individual samples performed throughout the study (mean 10% ± 7%). The observed differences in PCDD/F mass resulting from cooking were similarly close to the analytical variability determined from duplicate analyses in many of the samples (21% ± 6%). Cooking did result in larger changes in PCB and PCDD/F masses in some fish products (e.g., catfish) (Table 2).

Concentrations of PCBs and PCDD/Fs are generally reported to decline as a result of cooking, but increases also have been reported in the literature.[21] Perelló et al.[19] reported enhancement of PCBs in cooked fish relative to the raw samples, indicating that variable effects of cooking can be

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Table 2. ∑PCB and ∑PCDD/F Mass Change during Cooking[a]

	mass change (%)	
	PCB	PCDD/F
	Finfish	
catfish		
baked	35	48
boiled	28	48
fried	43	35
croaker		
baked	8.4	15
boiled	22	34
fried	9.4	*b*
grey mullet		
baked	44	16
boiled	42	−31
fried	31	44
grouper		
baked	−11	48
boiled	−4.1	50
fried	10	27
mackerel		
baked	−28	−154
boiled	−13	−13
fried	16	−91
milkfish		
baked	−3.2	−43
boiled	−1.9	7.4
fried	15	42
monkfish		
baked	−31	21
boiled	11	44
fried	−60	16
pomfret		
baked	28	−19
boiled	−1.7	−85
fried	5.7	−59
red snapper		
baked	−21	18
boiled	4.3	36
fried	22	23
whiting		
baked	15	13
boiled	−3.5	−6.5
fried	31	20
	Non-finfish	
cherrystone clam		
baked	54	29
boiled	59	29
conch		
baked	62	73
boiled	48	31
cuttlefish		
baked	−15	19
boiled	−22	15
octopus		
baked	−46	35
boiled	−40	41
scallop		
baked	−33	42
boiled	−46	42
sea squirt		
baked	2.2	−0.13
boiled	−26	−18
skate		
baked	2.2	−1.7
boiled	4.3	−9.1
squid		
baked	−21	37
boiled	−30	39

[a]Negative value indicates a PCB or PCDD/F increase. [b]Sample lost.

observed for thermally stable compounds, such as PCBs and PCDD/Fs, which may be attributable to other processes during cooking. Unlike some pesticides, which can be transformed and degraded during cooking of food for consumption, PCBs and PCDD/Fs are generally resilient to these processes, and the observed losses of these compounds may result from changes to the levels of moisture and lipid content in the cooked samples relative to the raw tissue.[18,37] Although small concentration differences were observed in the cooked fish products relative to the raw samples in the present study, the overall change in PCB and PCDD/F mass may have occurred due to loss of contaminants with the fat as the sample was cooked and to changes in the water content.

The results obtained in the present study suggest that PCBs and PCDD/Fs are reduced during cooking for most but not all fish products. It is important to recognize that the concentrations observed in all fish were relatively low and the apparent changes in PCB and PCDD/F concentration observed in cooked fish may be associated with analytical error or measurement. Additionally, greater information might have been achieved if cooking oils and juices had been retained for separate analysis to allow for a complete mass balance.[39]

Intake Determination. Intake estimates were calculated from the ∑PCB and ∑PCDD/F concentrations observed in the raw samples, to allow for comparison with previous study results where cooking was not performed. Estimates were based on consumption of a single 150 g portion of fish for all fish product types. The maximum ∑PCB concentration in the raw fish samples was 22 ng·g^{-1} ww in mackerel, corresponding to a maximum ∑PCB intake of 3300 ng, while the minimum intake was estimated for octopus (18 ng), which had the lowest ∑PCB concentration (0.12 ng·g^{-1} ww) (Table 3). The median PCB intake established for all fish tested was 370 ng. In comparison, consumption of 150 g of farmed salmon would result in 2200 ng ∑PCB exposure, based on a PCB concentration of 15 ng·g^{-1} ww.[9] This comparison indicates that a higher PCB intake would occur from farmed salmon per serving, which is more frequently consumed by Canadians relative to intakes determined for most of the species considered in the present study, with the exception of mackerel and cherrystone clams (Table 3). PCDD/F intakes ranged from 74 pg in monkfish to 6600 pg in catfish (Table 3). The PCDD/F intake estimated for farmed salmon was 140 pg (based on ∑PCDD/F concentrations of 0.93 pg·g^{-1} ww),[9] which is closer to the median of all the PCDD/F intakes

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Table 3. ∑PCB, ∑PCDD/F, and $TEQ_{PCDD/F+DL-PCB}$ Intake Estimates[a]

fish type	PCB intake (ng)	PCDD/F intake (pg)	$TEQ_{PCDD/F+DL-PCB}$ (pg)
		Finfish	
catfish	1600	6600	270
croaker	1900	240	49
grey mullet	2200	1000	210
grouper	1800	720	120
mackerel	3300	300	170
milkfish	120	260	32
monkfish	190	74	16
pomfret	920	190	64
red snapper	380	83	23
whiting	1500	97	36
		Non-finfish	
cherrystone clam	2500	800	160
conch	140	180	34
cuttlefish	23	150	34
octopus	18	130	28
scallop	24	103	26
sea squirt	74	940	52
skate	370	180	26
squid	53	77	100

[a]Based on a single 150 g serving of raw fish products.

estimated in the present study (185 pg). When elevated ∑PCDD/F intakes were estimated in the present study (e.g., catfish) (Table 3) relative to the intake determined for farmed salmon, a higher contribution of OCDD to ∑PCDD/F was generally observed. This may be related to the bottom-dwelling nature of some of the fish included in the present study and exposure to elevated concentrations of POPs in sediment or to their position in the aquatic food chain.[40,41]

No one type of cooking method resulted in a consistent decrease in PCB or PCDD/F masses in all of the fish products studied, and in fact increases were observed in some of the cooked fish products. Cooking, regardless of treatment (boiling or baking), resulted in an increase in the relative PCB mass, where a decrease in PCDD/F was observed for a select group of fish products (cuttlefish, octopus, scallop and squid) while the opposite pattern was found in skate samples (Table 2). On the basis of the average PCB reduction associated with cooking catfish (35%) determined, PCB intakes in cooked catfish would be reduced from 1600 ng in raw fish to 1040 ng for a 150 g serving of cooked catfish. Similarly, a PCDD/F reduction from 6600 pg in raw catfish to 3700 pg would be anticipated on the basis of an average 44% reduction resulting from cooking.

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Notes

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ABBREVIATIONS USED

PCB, polychlorinated biphenyl; PCDD/F, polychlorinated dibenzodioxin/dibenzofuran; (WHO-)$TEQ_{PCDD/F}$, (World Health Organization) toxic equivalency factor for polychlorinated dibenzodioxin/dibenzofurans; (WHO-)$TEQ_{PCDD/F+DL-PCB}$, (World Health Organization) toxic equivalency factor for polychlorinated dibenzodioxin/dibenzofurans + dioxin-like polychlorinated biphenyls; TCDD, tetrachlorodibenzodioxin; PeCDD, pentachlorodibenzodioxin; HxCDD, hexachlorodibenzodioxin; HpCDD, heptachlorodibenzodioxin; OCDD, octachlorodibenzodioxin; TCDF, tetrachlorodibenzofuran; HxCDF, hexachlorodibenzofuran; HpCDF, heptachlorodibenzofuran; OCDF, octachlorodibenzofuran

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