

Chemical Residue Concentrations  
in Four Species of Fish and the American Lobster  
from Long Island Sound, Connecticut and New York: 2006 and 2007

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## ABSTRACT

Chemical residues, primarily polychlorinated biphenyls (PCBs), in fish from Long Island Sound have been assessed only sporadically and recent information is lacking. In 2006 and 2007, a bi-state (Connecticut and New York) effort, supported by the U. S. Environmental Protection Agency, was conducted to update chemical residues in important fisheries, and in fisheries with existing health advisories or having a significant potential for health advisories. Striped bass, bluefish, weakfish, American eels and American lobster (hepatopancreas only) were collected and analyzed for PCBs (as Aroclors) and mercury. In addition, lobster (hepatopancreas) were analyzed for cadmium and chlorinated dioxins and furans. Where possible, the influence of year and season of collection, length, sex, and spatial distribution on chemical residue concentrations were assessed.

With both sampling years combined, PCBs averaged 0.333, 0.110, 0.565, 0.512, 0.506 and 1.31  $\mu\text{g/g}$  in striped bass, bluefish 305 to 508 mm, bluefish greater than 508 mm, American eel, weakfish, and the hepatopancreas of American lobster, respectively. In striped bass, there were no length-PCB relationships, and no spatial or sexual differences in PCB levels but there were differences in PCBs between sampling years. In contrast, bluefish displayed differences in PCB between years and PCB concentrations were related to length of fish. PCB concentrations in both striped bass and bluefish have declined by 70 percent or more since the mid 1980s but the declines are primarily due to reduced levels of lipids. PCBs in hepatopancreas of lobster differed by sex.

Average concentrations of mercury were 0.365, 0.271, 0.353, 0.110, 0.141 and 0.073  $\mu\text{g/g}$  in striped bass, bluefish 305 to 508 mm, bluefish greater than 508 mm, American eel, weakfish, and the hepatopancreas of American lobster, respectively. Occasional striped bass and bluefish contained mercury in excess of 1.0  $\mu\text{g/g}$ . Length-mercury relationships were present for striped bass, bluefish and weakfish.

Cadmium concentrations in lobster hepatopancreas were elevated ( $4.37 \pm 3.14 \mu\text{g/g}$ ) and showed no significant change in concentration since 1979/1981. Males contained greater cadmium concentrations than females.

Chlorinated dioxins and furans concentrations in lobster hepatopancreas were generally consistent throughout the Sound. 2,3,7,8-TCDD toxic equivalents were  $14.6 \pm 6.95 \text{ pg/g}$  ( $n = 64$ ). Males had greater levels of dioxins and furans than females.

As a consequence of these data, the health advice for human consumption of some fish species as modified by the state health authorities.

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## INTRODUCTION

Health advisories have been issued to restrict consumption of striped bass and bluefish in both Connecticut and New York, and further, New York issued health advice for American eels (CTDPH 2008, NYDSOH 2008). In each case, the advisories are due to excessive concentrations of PCBs. Health advice to restrict consumption of the hepatopancreas of American lobster is based on excessive levels of PCBs plus cadmium and chlorinated dioxins and furans (NYSDOH 2008). The specific health advisories in 2008 are provided in Table 1.

New York has examined PCB concentrations in striped bass (*Morone saxatilis*) taken from marine waters of New York, including Long Island Sound, in 1984, 1985, 1987, 1990 and 1994 (Sloan and Horn 1985; Sloan *et al.* 1986, 1988, 1991, 1995). In addition, other studies of PCBs in striped bass associated with sources of PCBs in the Hudson River and New York Harbor provide an extensive additional historical data set (Sloan *et al.* 2002 and 2005; Skinner *et al.* 1996; Skinner 2001; McReynolds *et al.* 2004a). However, the lack of recent data for Long Island Sound precludes the ability to provide meaningful assessments of current conditions.

In contrast to striped bass, only one PCB data set - from 1985 - is available for bluefish (*Pomatomus saltatrix*) representing the entirety of Long Island Sound (NOAA/EPA/FDA 1986a, 1986b). Other bluefish collections from New York Harbor provide additional insights in areas associated with PCB sources (Skinner *et al.* 1996; Skinner 2001) but the data are not spatially appropriate. For other species from Long Island Sound, there is a general lack of information on PCB concentrations.

Mercury has been measured in striped bass, bluefish, American eels (*Anguilla rostrata*) and weakfish (*Cynoscion regalis*) from New York Harbor (Skinner *et al.* 1996, Skinner 2002, McReynolds *et al.* 2004b). However, assessments of mercury in the Sound are limited to 61 striped bass in 1985 (Sloan *et al.* 1991), the vicinity of Eatons Neck, NY for six striped bass in 1999 (McReynolds *et al.* 2004b), and four American lobster (*Homarus americanus*) in 1979 (Sloan and Karcher 1984). In view of the limited data set available for mercury, this assessment will provide a substantial additional data base for mercury in fish and lobster from Long Island Sound.

Cadmium concentrations were measured in the hepatopancreas of 10 lobster from Eatons Neck, NY in 1981. All samples exceeded 1000 ng/g cadmium (Sloan and Karcher 1984). In contrast, striped bass from Eatons Neck contained less than 2.0 ng/g cadmium and striped bass plus four other species of fish from New York Harbor contained less than 40 ng/g cadmium. In blue crab from New York Harbor, the hepatopancreas contained about 20 times as much cadmium as muscle tissues (McReynolds *et al.* 2004b). Due to the propensity for accumulation of cadmium in hepatopancreas rather than in muscle tissue, cadmium analysis of lobster hepatopancreas is appropriate.

The data set for chlorinated dioxins and furans in aquatic biota is primarily limited to New York Harbor and the New York Bight (Skinner *et al.* 1997, Skinner 2001, McReynolds *et al.* 2004c). The only known data for Long Island Sound is that of O'Keefe *et al.* (1984) for 2,3,7,8-TCDD and 2,3,7,8-TCDF in striped bass from Little Neck Bay. As the focus of this study is on examination of residues in species for which a health advisory exists or may be anticipated, chlorinated dioxins and furans will be addressed in the hepatopancreas of American lobster only.

## STUDY OBJECTIVES

There were four study objectives:

- Determine the current status of PCB and mercury concentrations in striped bass, bluefish, American eels and weakfish, and in the hepatopancreas of American lobster.
- Determine the concentrations of cadmium and chlorinated dioxins and furans in American lobster hepatopancreas.
- Assess spatial and temporal differences in PCB levels in striped bass and bluefish from Long Island Sound.
- Provide a data base for health advisory assessments to be conducted by the CT Department of Public Health and the NYS Department of Health.

## METHODS

### Field sampling

Striped bass, bluefish, American eel, weakfish and lobster were collected from four areas of Long Island Sound as defined below and depicted in Figure 1.

<u>Area</u>	<u>Area description</u>
1	Western Long Island Sound from the Throggs Neck Bridge easterly to an imaginary north-south line between the Housatonic River, CT and Mount Misery Point near Port Jefferson, NY.
2	Long Island Sound north of the Connecticut-New York boundary and between the imaginary line from the Housatonic River, CT to Mount Misery Point near Port Jefferson, NY and the imaginary line from the east shore of the Connecticut River, CT to Orient Point, NY.
3	Long Island Sound south of the Connecticut-New York boundary and between the imaginary line from the Housatonic River, CT to Mount Misery Point near Port Jefferson, NY and the imaginary line from the east shore of the Connecticut river, CT to Orient Point, NY.
4	Long Island Sound east of the imaginary line between the east shore of the Connecticut River, CT and Orient Point, NY and to an imaginary line between the Connecticut-Rhode Island border and Montauk Point, NY.

American eel could be collected from the lower reaches of major rivers of Connecticut or in the bays of New York waters. The approximate location of each sample collected was indicated by UTM coordinates.

Due to observed seasonal differences in PCB concentrations in striped bass in past studies, striped bass and bluefish were collected in two seasons, i.e., spring/early summer and late summer/fall. Other species could be collected at any time they were available. Sample availability was

tempered by environmental conditions, life history requirements and type of sampling gear being used. Targeted sizes of fish and lobster were:

Striped bass greater than 610 mm (>711 preferred),  
Bluefish between 305 mm and 508 mm,  
Bluefish greater than 508 mm (> 635 mm preferred),  
American eel greater than 457 mm,  
Weakfish greater than 406 mm, and  
American lobster greater than 83 mm carapace length.

Sampling for striped bass and bluefish was conducted in 2006 but due to lack of completion of sampling requirements, supplemental samples were taken in 2007 to fulfill sampling needs. Other species were sampled in 2007.

Samples were collected through use of trawls, angling, lobster pots, and, for eels by eel pots or purchase from commercial vendors. For the latter, the location of collection by each vendor is known. Upon collection, samples were placed on ice until sample processing could occur. Sample processing usually occurred on the day of collection but on occasion occurred the day following collection.

Each sample was assigned a unique identifying number. Fish total length was measured to the nearest millimeter as well as weight to the nearest gram. The carapace length and total weight of lobster was determined. On collection records, the date and location of collection, the assigned identification number, species, length and weight were recorded for each sample. Chain of custody was maintained. Each sample was separately packaged in a food grade plastic bag, labeled and placed in a freezer at -20 degrees C. Samples were held by the CT Department of Environmental Protection, Marine Resources Division at their Old Lyme facility until shipped to the designated contract laboratory for chemical analysis.

### Chemical analyses

All samples were analyzed for PCBs as Aroclors and for total mercury. A semi-random subset of 25 bluefish samples were analyzed for PCB congeners. The one criteria for selection was that the approximate full range of total PCB concentrations should be represented. Lobster hepatopancreas was analyzed for cadmium and chlorinated dioxins and furans.

All samples were delivered frozen to the Mississippi State Chemical Laboratory (MSCL) at Mississippi State University. MSCL prepared each sample for analysis according to specifications for the project and developed aliquots of samples for analysis by their laboratory for PCBs as Aroclors and lipids, or by other contract laboratories for mercury and cadmium (CEBAM Analytical, Inc., Seattle, WA), or chlorinated dioxins and furans and congener specific PCBs (Pace Analytical Services, Inc, Madison, WI).

A filet (skin on, scales removed) from striped bass, bluefish and weakfish was excised for analysis. For American eels, the head, viscera and skin were removed and the remaining tissues, considered the edible portion, were used for chemical analyses. The hepatopancreas of American lobster was removed for analysis; edible muscle of lobster was not analyzed due to the low levels of chemical residue typically found. Each portion for analysis was ground three times and homogenized to assure sample consistency. Sample aliquots were taken from this homogenized mixture.

Lipids were determined gravimetrically as part of the analytical procedures for organic compounds. PCB Aroclors were determined by a method developed by Mississippi State Chemical Laboratory (MSCL Method NY-4) which is included as Appendix A. Briefly, the method includes drying with anhydrous sodium sulfate, soxhlet extraction of lipids with hexane, concentration by rotary evaporation, chemical extraction with methylene chloride, Florisil column clean-up with a diethyl ether-petroleum ether mixtures to obtain three analytic fractions, additional clean-up of the PCB fraction on a silicic acid column for separation of PCBs, and analyte determinations by gas chromatography with 30 m megabore columns (DB-608 and DB-5 dual columns) and electron capture detector. Concentrations of PCB Aroclors are adjusted to discount double counting of certain PCB peaks found in more than one Aroclor mixture. No adjustment of data was made for recovery of spiked materials.

PCB congeners were determined by Method 1668A (USEPA 1999), a high resolution gas chromatography-high resolution mass spectrometry (HRGC/HRMS) method. The data were adjusted by the laboratory for recovery of internal standards.

Mercury was analyzed by Method 1631A (USEPA 2001), a flameless atomic absorption method. No adjustments of data were necessary.

Samples for cadmium analyses were first digested with HNO<sub>3</sub>/HCl/H<sub>2</sub>SO<sub>4</sub>, then chelated with APDC in a buffered solution, pre-concentrated to CCl<sub>4</sub>, back extracted to 0.5% HNO<sub>3</sub>, and then analyzed by Method 200.9 (USEPA 1994a). No adjustments of data were necessary.

Chlorinated dioxins and furans were analyzed by Method 1613B (USEPA 1994b), a HRGC/HRMS method. The data were adjusted by the laboratory for recovery of internal standards.

Quality control included analysis of blanks, duplicate samples, matrix spikes and their duplicates, and, as required by the analytical method the analysis of internal standards, laboratory control spikes, surrogate spikes, and other alternative chemical spikes. In addition, reference fish samples (Schantz *et al.* 2004; Sloan *et al.* 2007) were analyzed for PCBs. Assessment of quality control measures was conducted with reference to “Guidance for assessing chemical contaminant data for use in fish advisories” (USEPA 1995).

#### Data analysis

Some data qualifiers were present when the data were reported by the laboratories. Briefly, each qualifier was handled as follows.

J qualified data - the analyte concentration was between the method detection limit and the quantitation limit - were used as reported.

B qualified data indicate detectable concentrations of the analyte were present in blanks and may be present in the tissue sample analyzed. No corrections of sample data for blank values were made. As a consequence, the statistical summaries may overestimate concentrations of the specific analyte. For this paper, where a blank value represents 10 percent or more of a sample analyte concentration, the blank may represent a significant portion of the sample concentration, therefore, a B qualifier was applied, where necessary, in the data summaries.

K qualified data – the analyte peak did not meet peak retention time criteria – indicates there was a lack of positive identification of the analyte. An estimated maximum possible concentration was reported by the laboratory for the chromatographic peak. Due to the uncertainty of the analyte identity, the analyte detection limit was applied.

I qualified data indicates an interfering chemical may comprise a major portion of and perhaps the entire analyte peak. The absence or presence of the analyte and its concentration cannot be confirmed by the analytical methods employed. The analyte detection limit was assigned.

U qualified data – the analyte, if present, is below the detection limit – were used as reported.

Total PCBs were expressed as Aroclor concentrations are a modified sum of Aroclor concentrations. The Aroclors are categorized into classes of low and high chlorinated Aroclors, i.e., Aroclors 1016, 1221, 1232, 1242, and 1248 are low chlorinated PCBs, and Aroclors 1254, 1260 and 1262 are highly chlorinated PCBs. If both classes of PCB have detected concentrations, the sum of those detected concentrations is total PCB; the nondetects are ignored. If either class of PCBs contains all nondetect values, then one half the detection limit of one Aroclor is added to the detected concentrations in the other class of PCBs to produce total PCB. If both classes of PCBs are all nondetect values, then one half the detection limit of one Aroclor per class is summed for total PCB. Where PCBs were quantified by congener concentrations, total PCBs are the sum of detected congener concentrations in each sample; non-detects are ignored. Since PCB concentrations are often related to lipid content, PCB concentrations were also normalized to lipids for spatial, temporal, and other assessments.

Computations of 2,3,7,8-TCDD toxic equivalents for humans and mammals employ toxic equivalency factors of Van den Berg *et al.* (2006). Where an analyte concentration was less than a detection limit, two computations were conducted that use one of two assigned values for the detection limit, i.e., zero or one half the detection limit. Where other statistical summaries are conducted, nondetects were assigned one-half the detection limit, unless all values for a compound were non-detect, then the value of the largest detection limit is reported.

Where multiple data comparisons could be made, spatial and temporal changes were assessed using the Kruskal-Wallis test to reduce the influence of potential outliers on the data set. Where only two data sets could be compared, the Mann-Whitney test was used (Conover 1980). A test produced a statistically significant difference when  $P < 0.05$ .

## RESULTS

### Quality control assessments

#### *Polychlorinated biphenyls*

Quality control for PCBs quantified as Aroclors was generally excellent (Appendix B, Table A). There was no blank contamination, all matrix spike samples were within acceptance limits, the reference material total PCB concentrations were acceptable, and the PCB 209 surrogate spikes were within acceptance limits. Determinations for duplicates were acceptable in 136 of 138 cases (98.5 %). Two duplicate samples had relative percent difference (RPD) values (i.e., 34 and 108 percent) that were outside acceptance limits. These outliers were due to concentrations of Aroclor 1248 that approximated the detection limit – indeed, one set of duplicate samples had Aroclor 1248 concentrations reported above and below the detection limit – thus, analytical variability would be expected to be greater. Since Aroclor 1248 was seldom detected, and samples associated with these duplicate samples did not have reportable concentrations of Aroclor 1248, the two outliers had no practical impact on the Aroclor 1248 concentrations reported for other samples. Recovery of total PCBs reference materials averaged 112 percent.

Quality control data summaries for PCB congener analyses are found in Appendix B, Table B. The two blanks lacked any PCB congeners in reportable quantities, except for PCB 11 at concentrations up to 141 pg/g. The B qualifier was applied to all PCB 11 data as an indicator that concentrations are likely overestimated. Lab control spikes were within acceptance limits for all native PCB congeners and 19 of 26 radio-labeled PCB congeners. Exceptions included very low recovery of PCB 1, PCB 3 and PCB 4, and somewhat low recovery of PCB 19, PCB 54, PCB 104 and PCB 155. Radio-labeled internal standard recoveries were similar to the recoveries of lab control spikes but recoveries ranged from 5 to 290 percent, of which 61 spikes were outside the target recovery range. The contract laboratory adjusted data based on recovery of internal standards.

### *Metals*

Fourteen of the eighteen blank samples for mercury analyses had non-detectable mercury concentrations (Appendix B, Table C). Three of the four remaining blank samples had mercury at the method detection limit (i.e., 0.0005 µg/g) and the last blank sample had 0.0006 µg/g. These blank values were less than 1.0 percent of the associated sample mercury concentrations, therefore, there was no discernable impact of blanks on the mercury concentrations reported for any sample. All duplicate samples, matrix spikes and matrix spike duplicates were acceptable, as were recoveries of mercury in reference materials. Overall, quality control for mercury analyses was excellent.

For cadmium, there was no blank contamination and all other quality control data were within acceptance limits (Appendix B, Table C). The cadmium data are considered of high quality.

### *Chlorinated dioxins and furans*

There were frequent detections of chlorinated dioxin and furan analytes in blank samples (Appendix B, Table D). Only reported concentrations of eight of the seventeen 2,3,7,8-substituted analytes (i.e., 2,3,7,8-TCDD, 1,2,3,7,8-PeCDD, 2,3,7,8-TCDF, 1,2,3,7,8-PeCDF, 2,3,4,7,8-PeCDD, 1,2,3,6,7,8-HxCDF, 1,2,3,7,8,9-HxCDF and 1,2,3,4,7,8,9-HpCDF) were unaffected by blank contamination. Blank contamination may contribute more than 10 percent of concentrations of some analytes in specific samples. Where blanks may be a significant contributor to analyte concentrations of some samples, the B qualifier was applied to summary data. Blank values may have the most impact on concentrations of OCDD, 1,2,3,4,6,7,8-HpCDF and OCDF. The presence of analytes in blanks, if deducted from reported analyte concentrations, had no appreciable impact on computations of 2,3,7,8-TCDD toxic equivalents.

Internal standards recovery for chlorinated dioxins and furans were generally within acceptance limits but, overall, recoveries tended to be lower than desired. The laboratory, in their data reports, reported adjusted data based on recovery of internal standards, thereby negating the concern for low analyte recovery. Recovery of lab control spikes were acceptable and recoveries ([RPD]) of duplicate spike recovery samples were all acceptable.

Interferences by polychlorinated diphenyl ethers (PCDE) were present for at least one analyte in all samples. PCDE interference was present for many samples for 1,2,3,4,7,8-PeCDF and 1,2,3,6,7,8-PeCDF, frequently for 2,3,7,8-tetrachlorodibenzofuran (2,3,7,8-TCDF) in samples from Areas 1 and 3, and to a much lesser extent 2,3,7,8-tetrachloro-*p*-dioxin (2,3,7,8-TCDD) and other analytes. Indeed, for 2,3,7,8-TCDF, 9 of 14 samples from Area 1 and 4 of 20 samples from Area 3 were affected. Estimated maximum possible concentrations were provided by the laboratory for these samples but there is no basis for placing reliance on these values. Where interferences were found, the detection limit was substituted since there was no way to separate or

quantify the magnitude of the interference. This substitution equated to zero for most statistical computations. Therefore, concentrations of analytes with interferences in the affected samples are likely to be underestimated. Assuming negligible PCDE interference and that EMPC values were correct, average 2,3,7,8-TCDD toxic equivalent concentrations could increase by as much as 40, 8.7, 15 and 5.8 percent in Areas 1 through 4, respectively.

### Data overview

Table 2 presents summaries of the sample length, weight, lipid, PCB and mercury data for each species by Area and year of collection. The size categories for bluefish reflect those in the original sampling design and roughly parallel those used by NOAA/EPA/FDA (1986a, 1986b). Similarly, Table 3 presents summary data for cadmium and Table 4 presents chlorinated dioxin and furan summaries for American lobster. Aspects of the data for the analytes are summarized for length-contaminant relationships (Table 5), seasonality (Tables 6, 7 and 8), spatial differences (Table 9), and sex (Table 10) for each species. PCB congener data for bluefish are found in Table 11.

The lack of sample availability did present issues for data analysis. For American eels, there were insufficient sample numbers in three areas precluding spatial comparisons. Further, sex was not determined for American eels. Similarly, for weakfish, spatial data comparisons are valid only for Areas 2 and 3 due to lack of sufficient sample numbers in Areas 1 and 4.

As a special note regarding American lobster, it was noted that there were no differences in length distributions for either sex among sampling areas. However, male lobster from Area 4, eastern Long Island Sound, were significantly ( $P = 0.0004$ ) heavier than their counterparts in the remaining three areas. No differences in weight were encountered for females from Areas 1 through 3; there were no females taken in Area 4. In Areas 1 through 3, male and female weights were statistically indistinguishable.

### Lipids

Lipid concentrations in striped bass in 2006 over all areas averaged ( $\pm$  standard deviation [ $\pm$  SD hereafter])  $1.51 \pm 1.22$  percent, whereas 2007 collections had a lipid content of  $2.23 \pm 2.09$  percent. On an annual basis the distributions of concentrations were not significantly different ( $P = 0.109$ ). There was no correlation with length of fish (Table 5). Within years there were no spatial differences in lipid content (Area 4 samples in 2007 were excluded due to insufficient sample numbers, i.e.,  $n = 2$ ) (Table 9). In 2006, a barely significant seasonal difference in lipid content was apparent ( $P = 0.047$ ;  $n = 103$ ) but a greater difference was present in the limited sampling in 2007 ( $P = 0.028$ ;  $n = 29$ ) (Table 6). There was a significant difference ( $P = 0.002$ ) by sex (males < females) in 2006 ( $1.12 \pm 1.18$  percent in males versus  $1.66 \pm 1.22$  percent in females), but not in 2007 ( $P = 0.131$ ), indeed, the relationship appeared to be reversed (males > females on a numeric basis) (Table 10). In 2006, there were no differences in lipid content between areas by sex.

Lipids in all bluefish sampled by year were correlated with length in 2006 but not in 2007 (Table 5). In smaller (305 to 508 mm) bluefish, the bluefish in 2006 had a lipid content of  $0.83 \pm 0.69$  percent whereas in 2007 they contained  $1.35 \pm 0.73$  percent lipid, a 62 percent increase ( $P = 0.004$ ). In Area 3, lipids in 2007 collections were 170 percent greater than in 2006 samples, i.e., 0.57 percent in 2006 versus 1.54 percent in 2007 (Table 2). There were no significant ( $P = 0.755$ ) differences in lipid content spatially within year of collection. In 2006, spring samples ( $0.44 \pm$

0.082 percent) had significantly lower ( $P < 0.001$ ) lipid content than fall samples ( $1.05 \pm 0.79$  percent) (Table 6). In 2007, there were insufficient spring samples to make a meaningful seasonal comparison. No difference by sex was apparent in either year of collection.

In large bluefish (greater than 508 mm), lipid content was more variable with several fish having lipid contents greater than 10 percent. These high lipid content fish occurred primarily in Areas 2 and 4 in 2006 and in Area 3 in 2007. Thus, mean concentrations for these areas appear to be biased high (Figure 2). Overall lipid content in 2007 samples was 26 percent greater than 2006 samples on an arithmetic basis ( $3.79 \pm 3.42$  percent in 2006 versus  $4.78 \pm 3.93$  percent in 2007), however, the difference was not significant ( $P = 0.123$ ). In both years, lipid content was lower in spring than in fall samples ( $P = 0.003$  and  $P = 0.015$  in 2006 and 2007, respectively) (Table 6). There were no differences in lipid content by sex in 2006 but a weak association in 2007, i.e., lipids in females were greater than males (Table 10). Spatial differences in lipids were present in both 2006 and 2007 (Table 9), but spatial differences were inconsistent between years. There were no differences by sex within seasons of a year.

In American eels, the overall ( $n = 15$ ) lipid concentration was  $10.36 \pm 4.12$  percent. There was a significant size-lipid relationship despite the small number of samples (Table 5). Sex was not determined.

Overall lipid content of weakfish ( $n = 25$ ) was  $5.97 \pm 3.80$  percent. There was no size-lipid relationship (Table 5). Lipids in weakfish from Area 3 were greater than for weakfish in Area 2 ( $P = 0.012$ ), i.e.,  $8.47 \pm 3.85$  % versus  $4.86 \pm 3.35$  % for Areas 3 and 2, respectively (Table 9). There was no difference in lipid by sex in smaller fish ( $P = 0.392$ ) and insufficient sample number for larger fish (Table 10).

The hepatopancreas of American lobster displayed substantial variability (Table 2, Figure 3) of lipid content by Area with means ranging from 9.6 % in Area 2 to 15.8 % in Area 1; spatial differences were not statistically different when all 65 samples were considered. However, in males, Area 4 specimens had significantly lower lipid concentrations than the remaining three areas ( $9.90 \pm 6.09$  percent in Area 4 vs  $17.3 \pm 8.89$  percent for Areas 1 through 3) (Table 9). The overall ( $n = 65$ ) mean and standard deviation lipid concentration in lobster hepatopancreas in 2007 samples was  $12.1 \pm 7.65$  percent. Differences in lipid content by sex were present ( $P < 0.001$ ). In Areas 1 through 3, males had  $17.31 \pm 8.89$  % lipid while females had  $8.56 \pm 3.80$  % lipid. Indeed, thirty-five percent of males had lipid concentrations greater than the maximum for females (16.2 %); the maximum lipid concentration in males was 36.1 %. Similarly, females did not display a difference in lipid concentrations between sampling Areas 1 through 3. Lipids were not related to carapace length of the specimens (Table 5).

## Polychlorinated biphenyls

### Striped bass

#### *Wet weight PCB evaluation:*

There were significant ( $P < 0.001$ ) differences in PCB concentrations between years ( $0.253 \pm 0.193$   $\mu\text{g/g}$  in 2006 and  $0.511 \pm 0.247$   $\mu\text{g/g}$  in 2007), therefore, the data for each year must be treated separately. Length-PCB relationships (wet weight) do not exist for striped bass from Long Island Sound (Table 5; Figure 4) in either year. Regressions of length and PCB showed non-significant correlation coefficients of 0.0035 ( $n = 103$ ) and 0.0977 ( $n = 29$ ) for 2006 and

2007 samples, respectively (Table 5). Therefore, the PCB data for striped bass may be examined without further reference to length within each year.

Seasonal differences within year of collection were absent ( $P = 0.474$  and  $P = 0.179$  for 2006 and 2007, respectively) (Table 7). Therefore, seasonal data within Areas for each year can be combined for spatial comparisons.

Within each year of collection, no spatial differences in total PCB concentration were present ( $P = 0.920$  for 2006 samples and  $P = 0.859$  for 2007) (Table 9). In 2007, there were insufficient sample numbers in Area 4 for inclusion in statistical testing. Therefore, within years there is no rationale for segregating striped bass PCB data by Area within the Sound during 2006 or 2007. In 2006, there was a barely significant difference ( $P = 0.046$ ) in total PCB concentrations by sex of striped bass (males  $0.330 \pm 0.290 \mu\text{g/g}$ , females  $0.226 \pm 0.136 \mu\text{g/g}$ ) but the difference was absent in 2007 ( $P = 0.069$ ) (Table 10). Sex was not a strong determinant of total PCB levels in striped bass.

#### *Lipid based PCB evaluation:*

Since PCB concentrations in 2007 were over twice the 2006 value, and there were increased lipid concentrations in 2007 (noted previously), further analyses were conducted with comparisons on a lipid normalized basis to assess the influence of inter-annual differences and other lipid based data aspects further.

The greater PCB wet weight concentrations in 2007 were reinforced by the PCB-lipid assessment, i.e.,  $24.1 \pm 28.2 \mu\text{g/g}$  in 2006 vs  $47.3 \pm 52.0 \mu\text{g/g}$  in 2007. In both years, spring lipid PCB levels were less than fall concentrations ( $P < 0.02$  in both years) (Table 7).

Due to the seasonal differences within each year, the spatial analysis was carried to each season's collection. A spatial difference ( $P = 0.0139$ ) occurred in Spring 2006 only with lipid based PCB concentrations increasing from the eastern Sound to western Sound ( $10.02 \pm 2.61 \mu\text{g/g}$  to  $26.87 \pm 22.92 \mu\text{g/g}$ , respectively). Eighteen fish had  $50 \mu\text{g/g}$  PCB (lipid basis) or more of which half were from the western Sound (Area 1) and another third were in the north-central Sound (Area 2) and the remaining fish from the south-central Sound (Area 3).

Males had greater lipid based PCB concentration than females in 2006 only ( $P < 0.001$ ) ( $43.77 \pm 46.42 \mu\text{g/g}$  for males and  $17.10 \pm 12.12 \mu\text{g/g}$  for females) and the difference occurred in the two seasons as well. There were no males collected in spring 2007, and no sex difference in fall 2007 although sample numbers were limited.

### Bluefish

#### *Wet weight PCB evaluation:*

In a contrast to striped bass, bluefish show an exponential length-PCB relationship in each sample year (Figure 5;  $R^2 = 0.6081$  in 2006, and  $R^2 = 0.6885$  in 2007), therefore, statistical analyses of bluefish must be cognizant of the size of fish. Figure 5 suggests bluefish smaller than 508 mm contain relatively constant PCB concentrations, thus, they will be treated as one size group while larger fish will be examined separately.

Within the smaller size group (305 to 508 mm) of bluefish, Sound-wide PCB concentrations on a wet weight basis were  $0.069 \pm 0.047 \mu\text{g/g}$  ( $n = 25$ ) in 2006 and  $0.211 \pm 0.089 \mu\text{g/g}$  ( $n = 10$ ) in

2007, a significant difference ( $P < 0.001$ ). There were no significant differences in PCBs among areas by year of collection and no differences by season within years (Tables 9 and 7, respectively). Males tended to have greater PCB concentrations than females in 2006 ( $P = 0.025$ ) but there were insufficient samples in 2007 to conduct a meaningful test. The values for females in 2006 are influenced by an outlier value.

For bluefish over 508 mm, 2006 fish had significantly lower ( $P < 0.001$ ) PCB concentrations than 2007 collections ( $0.483 \pm 0.445 \mu\text{g/g}$  in 2006 and  $0.858 \pm 0.561 \mu\text{g/g}$  in 2007), i.e., PCBs in 2006 bluefish were about one-half 2007 fish. Seasonal comparisons are not meaningful due to limited spring collections. There were no differences by sex in either year (Table 10). On an arithmetic basis, fish from Area 2 in 2006 appear to have somewhat greater average PCB concentrations on a wet weight basis ( $0.667 \mu\text{g/g}$  versus about  $0.40 \mu\text{g/g}$  elsewhere). However, the Kruskal-Wallis test showed the fish from all areas were statistically equivalent ( $P = 0.0866$ ) (Table 9), albeit the average PCB level for Area 2 was influenced by outlier values. In 2007, sufficient samples for spatial comparison are present for Areas 2 and 3; there was no difference in PCB concentration.

#### *Lipid based PCB evaluation:*

In bluefish 305 to 508 mm, comparison of lipid normalized total PCB concentrations confirmed the difference between years ( $P = 0.009$ ), i.e.,  $10.49 \pm 7.90 \mu\text{g/g}$  in 2006 versus  $18.08 \pm 8.96 \mu\text{g/g}$  in 2007. Differences between areas were not apparent although suggested in the small sample size in 2007 (Table 9). In 2006, spring bluefish had greater lipid based PCB concentrations than fall fish ( $P = 0.031$ ), a contrast to wet weight based PCBs. Sex differences were not present in lipid based PCB in either year (Table 10).

In larger bluefish, interannual differences were confirmed ( $P = 0.002$ ) with lipid based PCB concentrations in 2006 bluefish at  $16.36 \pm 14.07 \mu\text{g/g}$  and 2007 fish having  $32.20 \pm 41.26 \mu\text{g/g}$ . Seasonal differences were absent in 2006 but present in 2007, however, spring sample numbers in 2006 were small, thus, the presence or absence of any relationship may be questioned (Table 7). There were no spatial differences in 2006 but a spatial difference (Area 2 > Area 3) is suggested by the small sample numbers in 2007 (Table 9). No difference in lipid based PCB by sex was present in 2006 but the limited sample numbers in 2007 suggest males had greater lipid based PCB levels than females ( $P = 0.026$ ;  $49.36 \pm 60.82 \mu\text{g/g}$  in males,  $21.34 \pm 16.50 \mu\text{g/g}$  in females) (Table 10).

#### American eels

Total PCB concentrations in the 15 American eels collected averaged  $0.506 \pm 0.097 \mu\text{g/g}$  on a wet weight basis, and  $5.601 \pm 2.215 \mu\text{g/g}$  on a lipid basis. Insufficient samples were available to examine spatial differences and sex was not determined. No length-PCB relationship was apparent ( $R^2 = 0.0505$ ) on a wet weight basis but a negative length-PCB relationship ( $R^2 = 0.7947$ ) was present on a lipid basis.

#### Weakfish

Twenty-four of the 25 weakfish were taken from the central portion of the Sound, Areas 2 and 3. Average PCB concentrations were  $0.388 \mu\text{g/g}$  and  $0.791 \mu\text{g/g}$  in Areas 2 and 3, respectively. However, a length-PCB relationship ( $R^2 = 0.7193$ ; Table 5 and Figure 6) exists which influences the comparison of PCB findings for Areas 2 and 3. For weakfish in the 380 to 508 mm size group, the mean PCB concentrations were  $0.313 \pm 0.089 \mu\text{g/g}$  and  $0.505 \pm 0.206$

$\mu\text{g/g}$  for Areas 2 and 3, respectively, which are significantly different ( $P = 0.008$ ). The five larger weakfish had a mean total PCB concentration of  $1.09 \pm 0.663 \mu\text{g/g}$ . Alternatively, expression of PCB values on a lipid basis reversed spatial differences for weakfish 380 to 508 mm so that Area 2 > Area 3 ( $P = 0.017$ ) with concentrations of  $9.14 \pm 4.74 \mu\text{g/g}$  and  $5.61 \pm 1.52 \mu\text{g/g}$ , respectively (Table 9). There was a difference by sex (males > females) in lipid-based PCBs in smaller weakfish although the difference was barely significant (Table 10). By Area, there was no difference by sex in Area 2 within the smaller sized fish ( $P = 0.3861$ ) while in Area 3 a difference (males > females) was suggested but the small sample numbers ( $n = 3$  for each sex) negates meaningful comparison.

### American lobster

On a wet weight basis, the average PCB concentration was  $1.31 \pm 0.553 \mu\text{g/g}$  for the entire Sound. However, there were significant differences ( $P < 0.001$ ) in PCB concentration by sex, i.e., males > females in Areas 1 through 3 (Table 10). Within males, the eastern Sound specimens (Area 4) had significantly lower PCB levels than in the remainder of the Sound (Areas 1 through 3). In females, there were no spatial differences in Areas 1 through 3 but there were no females collected in Area 4. For Areas 1 through 3, the 24 males had  $1.704 \pm 0.522 \mu\text{g/g}$  PCB while the 26 females had  $1.023 \pm 0.399 \mu\text{g/g}$  PCB; the 15 Area 4 males had  $1.195 \pm 0.484 \mu\text{g/g}$  PCB and are statistically the same as females from other areas. No carapace length-PCB relationship within each sex (Table 5), nor weight-PCB relationship for females was present. Males did show a positive weight-PCB association ( $R^2 = 0.449$ ,  $n = 24$ ).

On a lipid PCB basis, the average lipid based PCB concentration for all 65 samples was  $13.69 \pm 8.431 \mu\text{g/g}$ . Sex differences in lipid PCB concentrations were not apparent ( $P = 0.0575$ ) in Areas 1 through 3 (Table 10), nor were there spatial differences in males from Areas 1 through 4 (Table 9). PCBs in females were greater in Area 2 than in Areas 1 and 3. This spatial difference in females caused a spatial difference ( $P = 0.009$ ) in the combined dataset with Area 2 having the greatest concentrations and Areas 1 and 3 the lowest levels; Area 4 was similar but lower than Area 2. No carapace length-lipid PCB or weight-lipid PCB relationship existed within each sex.

### Mercury

#### Striped bass

Neither year of collection ( $P = 0.092$ ) nor sex (Table 10) had an impact on mercury concentrations in striped bass. Season of collection had no effect ( $P = 0.515$ ) on mercury concentrations in 2006 but was marginally significant ( $P = 0.040$ ) in 2007 where sample size was small. Due to a general lack of these associations, mercury data were combined for further examination. Length-mercury regressions show a general increase in mercury concentration with size of fish in each year of collection ( $R^2 = 0.5613$  in 2006,  $R^2 = 0.8155$  in 2007,  $R^2 = 0.5848$  for combined data) (Table 5, Figure 7). Because of the length-mercury relationship, there was a significant ( $P < 0.001$ ) spatial difference in mercury concentration. Areas 1 and 3 had the smallest fish (each at 714 mm mean length) and the lowest average mercury concentrations ( $0.273 \mu\text{g/g}$  and  $0.268 \mu\text{g/g}$ , respectively), followed by Area 4 with 808 mm fish and  $0.342 \mu\text{g/g}$  mercury, and Area 2 had the largest fish (mean of 881 mm) with the greatest mercury levels (mean of  $0.528 \mu\text{g/g}$ ).

### Bluefish

Mercury in bluefish did not show differences in concentration between 2006 and 2007 within the two size categories, therefore, the data within size groups for the two years were combined for analysis. Bluefish in the 305 to 508 size category did not demonstrate significant spatial variation in mercury concentrations ( $P = 0.485$ ) (Table 9). No length-mercury relationship was present within this size group (Table 5). There were no differences based on sex of the fish (Table 10). However, spring fish had greater mercury concentrations than fall fish ( $P < 0.001$ ), i.e.,  $0.324 \pm 0.094 \mu\text{g/g}$  in spring and  $0.216 \pm 0.070 \mu\text{g/g}$  in fall (Table 8).

Mercury concentrations in bluefish over 508 mm were statistically the same ( $P = 0.275$ ) for the two years and sex differences were not present (Table 10). Seasonal differences were absent in 2006 but were present in 2007, and combined data showed seasonal differences ( $P = 0.008$ ) (Table 8). However, the difference in the average concentrations was only  $0.07 \mu\text{g/g}$  (i.e.,  $0.412 \pm 0.158 \mu\text{g/g}$  in spring versus  $0.342 \pm 0.129 \mu\text{g/g}$  in fall), a difference which is very small. A positive length-mercury relationship is present (Table 5; Figure 8). As with striped bass, the generally larger fish were collected in Areas 2 and 4 (mean lengths of 707 and 702 mm), and smaller fish in Areas 1 and 3 (mean lengths of 671 and 655 mm, respectively). Mercury concentrations were greater ( $P = 0.008$ ) for the north central (Area 2;  $0.400 \pm 0.146 \mu\text{g/g}$ ) and eastern (Area 4;  $0.368 \pm 0.125 \mu\text{g/g}$ ) Sound compared with the remaining two areas ( $0.299 \pm 0.078 \mu\text{g/g}$  and  $0.317 \pm 0.158 \mu\text{g/g}$  in Areas 1 and 3, respectively) (Table 9).

### American eel

Mercury in American eels did not exceed  $0.2 \mu\text{g/g}$  in any of the 15 samples; the mean  $\pm$  SD concentration was  $0.110 \pm 0.037 \mu\text{g/g}$ . Length and mercury content were positively correlated ( $R^2 = 0.4149$ ) (Table 5).

### Weakfish

The twenty-five weakfish contained average mercury concentrations of  $0.141 \pm 0.094 \mu\text{g/g}$  (Table 2). Spatial differences were not apparent between Areas 2 and 3; insufficient samples were available for comparisons with Areas 1 and 4. Mercury concentrations are positively correlated with length of fish ( $R^2 = 0.7447$ ) (Table 5, Figure 9). There were no sex differences in mercury concentrations for weakfish 508 mm or less in length taken in Areas 2 and 3 ( $P = 0.689$ ); sample size for each sex of larger weakfish was too small to test (Table 10).

### American lobster

All mercury concentrations in the hepatopancreas of lobster, regardless of area of collection, were less than  $0.200 \mu\text{g/g}$ . The average mercury concentration for the combined data was  $0.0726 \pm 0.0324 \mu\text{g/g}$  (Table 2). No carapace length-mercury relationship was found ( $R^2 = 0.0949$ ) (Table 5). For Areas 1 through 3, there were no differences in mercury concentration by sex ( $P = 0.356$ ). American lobster in Area 4 (all males) appear to contain significantly ( $P < 0.001$ ) greater concentrations of mercury than in Areas 1, 2 and 3 (Figure 10), which have equivalent concentrations (Table 9). Despite the spatial difference, the difference in mean concentrations of  $0.05 \mu\text{g/g}$  (i.e.,  $0.111 \pm 0.028 \mu\text{g/g}$  in Area 4 versus  $0.061 \pm 0.024 \mu\text{g/g}$  in Areas 1 through 3 combined) is generally considered inconsequential.

### Cadmium in American lobster hepatopancreas

Mean cadmium concentrations in lobster hepatopancreas from the four collection Areas ranged from 3.5 µg/g to 4.9 µg/g; maximum concentrations ranged as high as 17.3 µg/g (Table 3). There were no relationships between cadmium and carapace length ( $R^2 = 0.0077$ ) of the specimens (Table 5). Spatial differences did not occur within either sex or when data were combined (Table 9). Males contained significantly more cadmium than females ( $P < 0.001$ ) (Table 10). For Areas 1 through 3, 24 males contained an average of  $5.28 \pm 2.753$  µg/g while 26 females contained  $3.563 \pm 3.959$  µg/g cadmium. Removal of two outliers for females reduced concentrations to  $2.496 \pm 1.232$  µg/g. Males from Area 4 had  $4.323 \pm 1.446$  µg/g cadmium which was not significantly different from males in the other three Areas.

### Chlorinated dioxins and furans in American lobster hepatopancreas

Table 4 summarizes data by Area for the 17 chlorinated dioxins and furans with 2,3,7,8-chlorine substitution, 2,3,7,8-TCDD toxic equivalents, and total tetra- through octa-chlorodibenzo-*p*-dioxins and dibenzofurans in American lobster hepatopancreas. By observation, concentrations of chlorinated dioxins were generally lowest in eastern Long Island Sound whereas such a tendency was lacking for most chlorinated dibenzofurans.

2,3,7,8-TCDF concentrations were the greatest of all analytes with an overall average concentration of 39.9 pg/g, maximum of 120 pg/g. 2,3,4,7,8-PeCDF was second most abundant at 14.1 pg/g followed by 1,2,3,7,8-PeCDF at 9.0 pg/g. 2,3,7,8-TCDD concentrations ranged up to 5.8 pg/g although the mean ( $\pm$  SD) was  $1.36 \pm 1.14$  pg/g for the 64 samples. Three furans were not detected in most samples, i.e., 1,2,3,6,7,8-HxCDF, 1,2,3,7,8,9-HxCDF and 1,2,3,4,7,8,9-HpCDF (Table 4).

For total 2,3,7,8-TCDD toxic equivalents (TEQ), males have greater TEQ concentrations than females within Areas 1 through 3 ( $P = 0.006$ ) (Table 10) with males averaging 18.2 pg/g while females had 12.3 pg/g. In Area 4 (only males were collected) the TEQ averaged 12.5 pg/g. Within sex, there were no significant area differences in TEQ concentration and when data for both sexes are combined, no spatial differences in TEQ concentrations were present ( $P = 0.922$ ) (Table 9). The mean  $\pm$  SD for all samples was  $14.6 \pm 6.96$  pg/g TEQs. TEQs totaling 10 pg/g were exceeded by 76 percent of the samples.

Eighty-seven percent of TEQs were contributed by 2,3,4,7,8-PeCDF (29 %), followed by 2,3,7,8-TCDF (27 %), 1,2,3,7,8-PeCDD (22 %) and 2,3,7,8-TCDD (9 %). The remaining 13 % was made up by the other 13 compounds.

The total dioxin and furan homologs had distinctly greater concentrations than their corresponding 2,3,7,8-substituted congeners; concentrations were three to ten times their respective congener levels.

### PCB congeners

A subset of 25 bluefish samples were semi-randomly selected (representation of the full range of total PCB - by Aroclor - concentrations was desired) from 2006 collections and analyzed for the full suite of 209 PCB congeners. The objectives were to test the relationship between total PCB concentrations quantified on Aroclor versus congener bases, identify important congeners

contributing to total PCB concentrations, and to make a limited assessment of coplanar (dioxin-like) PCB congener contributions to 2,3,7,8-TCDD toxic equivalents. Since the chemical analyses were conducted independently by two different laboratories using different analytical methods, there was the ability to independently examine the validity of the total PCB concentrations.

There were 163 PCB congener peaks quantified of which 32 contained coeluting PCB congeners representing 78 PCB compounds (Table 11, Figure 11). Total PCB congener concentrations ranged from 33,800 pg/g to nearly 3,920,000 pg/g and provided an approximate representation of the full range of total PCB concentrations observed on an Aroclor basis. Since the sample with the maximum PCB concentration may be considered an outlier, comparisons with and without the outlier were made for total PCB on Aroclor versus congener bases. Figure 12 graphically compares the data for each fish sample. There was a strong correlation between total PCBs quantified as Aroclors and congeners, i.e.,  $R^2 = 0.8642$  without the outlier sample and  $R^2 = 0.9736$  when the outlier sample was included. Overall, there was a nearly 1:1 concentration relationship.

The relative contribution of individual PCB congeners to total PCB concentrations was categorized based on the average congener concentrations for all samples in Table 11. Individual samples may occasionally have a congener or congeners with a classification that is somewhat higher or lower than the classification given based on mean concentrations. Seventy to 133 congener peaks were detected (mean 107 congener peaks). Twenty-four congener peaks (up to 46 possible congeners) were present at concentrations representing 1.0 percent or more of the total PCB concentration. The classifications are:

<u>Class</u>	<u>No. of Peaks</u>	<u>PCB congener (IUPAC numbers)* peaks</u>
≥ 10 %	1	153/168
≥5.0 < 10 %	3	90/101/113, 129/138/163, 147/149
≥1.0 <5.0 %	20	44/47/65, 49/69, 52, 61/70/74/76, 66, 86/87/97/108/119/125, 92, 95, 99, 105, 110/115, 118, 128/166, 132, 135/151, 146, 177, 180/193, 183/185, 187
<1.0 %		All other congeners

\* ## indicate coeluting PCB congener numbers.

Only two of the congeners above are coplanar PCBs, i.e., congeners 105 and 118. However, there are a total of 12 PCB congeners that are coplanar and have potential dioxin-like toxicity. 2,3,7,8-TCDD toxic equivalents were computed for each sample using mammalian/human toxicity equivalency factors of Van den Berg *et al.* (2006). Mean TEQs with and without the outlier were:

<u>n</u>	<u>TEQ (pg/g wet weight)</u>
25	8.47 ± 23.2
24	3.99 ± 6.30

Twenty percent of the TEQs exceeded 10 pg/g; indeed, the outlier value was 116 pg/g.

## DISCUSSION

### Temporal changes in PCBs

PCBs in striped bass from Long Island Sound have been measured episodically since 1984. A size-PCB relationship has been absent in nearly all years in which sampling has occurred. Spatial differences were reported in the period 1985 through 1994 with striped bass from the western Sound having somewhat greater PCB concentrations than striped bass from the eastern Sound (Sloan *et al.* 1995). Since spatial differences were absent in 2006 and 2007, and the spatial differences in earlier years would not materially affect a temporal assessment, the data within each year was combined for this temporal assessment. Further, the earlier studies used a different boundary for samples from eastern Long Island Sound, i.e., Peconic Bay, Napeague Bay and portions of Gardiners Bay and Block Island Sound were excluded from the definition of Long Island Sound. To achieve spatial comparability with the current study for this temporal comparison, the raw data for total PCBs in striped bass from these additional waters have been included for the years 1984 through 1994. Figure 13 shows 2006-2007 striped bass samples represent an 82 percent decline in PCB concentrations from the 1985-1987 period. The 1984 samples were not included in this computation because of a sampling bias for western Long Island Sound which caused an overestimate of Sound-wide PCB concentrations. Declines in PCB concentrations in striped bass of a similar magnitude have been reported for the Hudson River (Sloan *et al.* 2005; Interstate Workgroup 2008), New Jersey coastal waters and the Delaware River estuary (Interstate Workgroup 2008).

In striped bass, the lipid content has declined 65 percent from 4.86 percent lipid (n = 560) in 1985-1987 to 1.67 percent in 2006-2007 (n = 132) (Figure 13). Since PCBs are lipophilic, this change may affect total PCB concentrations expressed on a wet weight basis. Lipid normalized total PCBs declined by 50 percent from an average of 59.31  $\mu\text{g/g}$  in 1985-1987 to 29.19  $\mu\text{g/g}$  in 2006-2007 (Figure 14). Average PCB and lipid concentrations were strongly correlated ( $R^2 = 0.826$ ;  $P < 0.01$ ) (Figure 15), therefore, changes in lipid content appear to be a primary factor controlling PCB concentrations in Long Island Sound between 1984 and 2007. [Figures 13, 14 and 15 provide the mean PCB concentration  $\pm$  the 95 % confidence interval of the mean.]

PCB levels in bluefish from Long Island Sound were examined in a 1985 federal study (NOAA/FDA/EPA 1986a, 1986b) as well as in the current assessment. The first study showed there was little difference in PCB concentrations within size groups between seasons and areas along the Atlantic Coast. By selecting only bluefish taken from Long Island Sound, direct comparisons of total PCB concentrations are possible. Only fish greater than 508 mm were available for this comparison. The Federal study examined PCB concentrations in individual fish samples and in five fish composite samples, therefore, the information is provided separately for the two types of samples (Table 12). On a wet weight basis, total PCB concentrations declined by an average of 70 percent, however, on a lipid basis, there were no declines in total PCBs apparent ( $P > 0.05$  for all comparisons). The declines in total PCB concentrations on a wet weight basis appear to be controlled by declines in lipid content averaging 64 percent (average lipid contents in 1985 were 14.94 percent for 16 individual samples and 10.48 percent for 62 composite samples).

The association between total PCB concentrations and lipid content over time in striped bass and bluefish suggests PCB exposures in the marine environment have changed relatively little. In the absence of significant declines in PCB exposures, as lipids change in response to availability of food sources and dissolved PCB concentrations in marine waters, so will PCB concentrations change in striped bass and bluefish. Significant declines in PCB concentrations in North Atlantic

surface waters were documented in the 1970s following controls of open uses of PCBs (Harvey *et al.* 1974), and later in the 1970s and early 1980s with phase-out of all PCB uses (Fensterheim 1993). Concentrations of PCBs in air and seawater are in equilibrium (Harvey *et al.* 1974; Schreitmüller *et al.* 1994; Gioia *et al.* 2008). In the north Atlantic, there has been little, if any, change in air and seawater PCB concentrations between 1990 and 2005 (Gioia *et al.* 2008). This is in contrast to declining PCB exposures in freshwater environments documented in the Hudson River (Sloan *et al.* 2005), Great Lakes (Scheider *et al.* 1998; Hickey *et al.* 2006), and other locations (e.g., Fensterheim 1993; Brown *et al.* 1998; Villeneuve *et al.* 1999). However, this limited assessment for the marine environment needs to be supported, or refuted, by closer examination of this relationship for these and other species in the marine environment. Further, this association suggests revisions of health advisories based on current or future levels of PCBs in these fish species must be conducted with caution.

There are either insufficient numbers of samples or no historical data for PCBs in American eels, weakfish and American lobster from Long Island Sound, therefore, temporal comparisons were not possible.

### Mercury

Sixty-two striped bass from Long Island Sound were examined for mercury in 1985 (Sloan *et al.* 1991) and had  $0.298 \pm 0.169$  µg/g mercury. In 2006-2007, the overall mercury concentration for 132 samples were  $0.365 \pm 0.214$  µg/g, which is not significantly different ( $p > 0.05$ ) by testing differences between means. Since mercury is bound to proteins (Bloom 1992) rather than lipids, changes in lipid content would have no impact on mercury concentrations.

### Cadmium

Sloan and Karcher (1984) measured cadmium in the hepatopancreas of 10 lobster from Eatons Neck, NY in western Long Island Sound (Area 1). The cadmium mean and standard deviation concentrations were  $6.65 \pm 6.61$  µg/g (based on the original data but incorrectly reported as  $4.85 \pm 4.70$  µg/g by Sloan and Karcher 1984) in 1981. In 2007, the Area 1 cadmium was  $3.49 \pm 2.41$  µg/g ( $n = 9$ ) which was not significantly ( $P = 0.232$ ) different from 1981. Sloan and Karcher (1984) also reported males had significantly greater concentrations of cadmium in the hepatopancreas than females, i.e.,  $10.96 \pm 7.14$  µg/g in males ( $n = 5$ ) versus  $2.35 \pm 1.03$  µg/g in females ( $n = 5$ ). This difference by sex is more dramatic than found within the current study for Area 1 ( $4.200 \pm 2.688$  µg/g in males ( $n = 6$ ) and  $2.071 \pm 0.823$  µg/g in females ( $n = 3$ ). Despite the differences, there were no significant differences ( $p > 0.05$ ) in cadmium between 1981 and 2007 within sex. Therefore, while the 1981 concentrations are numerically greater than reported in the current investigation for Area 1, in the absence of information for other years, there appears to be no significant change in cadmium concentrations in hepatopancreas of American lobster over the intervening 26 year time span.

Like lobster, blue crabs have a health advisory to avoid consumption of the hepatopancreas which is due, in part, to excessive cadmium concentrations (NYSDOH 2008). Blue crabs from the Hudson River have experienced a 70 percent decline in cadmium concentrations following partial control (in 1990 and 1991) of a cadmium source associated with the former Marathon Battery site in Cold Spring, NY. Cadmium in the hepatopancreas declined significantly ( $P < 0.001$ ) river-wide from  $8.13 \pm 5.67$  µg/g ( $n = 65$ ) in the 1979-1981 period to  $2.39 \pm 2.01$  µg/g ( $n = 58$ ) in the 2000-2004 period (Levinton *et al.* (2006). Despite the reduction in cadmium concentrations, the NYS Department of Health believes the health threats from cadmium levels (plus PCBs and

chlorinated dioxins and furans as well) in blue crab hepatopancreas remain too great for any relaxation of the health advisory (NYSDOH 2009). In contrast to the Hudson River, blue crab from Jamaica Bay, an area without known sources of cadmium, have had consistently low levels of cadmium in the hepatopancreas, i.e., means of 0.39  $\mu\text{g/g}$  in 1981 ( $n = 5$ ; Sloan and Karcher 1984) and 0.38  $\mu\text{g/g}$  in 2005 ( $n = 15$ ; NYSDEC unpublished data).

Sloan and Karcher (1984) suggest blue crab and lobster having elevated hepatopancreas to muscle cadmium ratios - greater than about 15 - indicate exposure to cadmium sources in the environment, and by observation, specimens from these areas – Hudson River, Flushing Bay, Eatons Neck - have cadmium levels in the hepatopancreas greater than 1.5  $\mu\text{g/g}$ . Although there is an apparent lack of information on cadmium concentrations in lobster from areas without known cadmium sources, average cadmium levels in hepatopancreas of Long Island Sound lobster in excess of 1.5  $\mu\text{g/g}$  (indeed, 4.37  $\mu\text{g/g}$ ) suggests sources of cadmium are present within the Sound's drainage basin.

Several investigators (Sloan and Karcher 1984, McReynolds *et al.* 2004b, Levinton *et al.* 2006) have shown muscle in lobster and blue crab contain lower concentrations of cadmium than the hepatopancreas. Cadmium in muscle ranged from 0.7 to 22 percent of concentrations in hepatopancreas, with an average of about six percent or a median of about three percent. Therefore, in the absence of cadmium data for lobster muscle in this study, it can be estimated that the average cadmium concentrations in muscle may be approximately 0.12 to 0.25  $\mu\text{g/g}$ .

Cadmium levels in lobster hepatopancreas in this study are two to three orders of magnitude greater than levels observed in edible muscle of fish. McReynolds *et al.* (2004b) found less than 0.002  $\mu\text{g/g}$  cadmium in 22 striped bass from Eatons Neck, NY, and less than 0.04  $\mu\text{g/g}$  in striped bass and four other fish species from the Hudson River and New York Harbor. There is no reason to believe that such a relationship has changed.

#### Chlorinated dioxins and furans in lobster

The presence of dioxins and furans is a contributor to the cause for health advice to avoid consumption of the hepatopancreas of lobster (NYSDOH 2008). However, this study is the first investigation of chlorinated dioxins and furans in lobster from Long Island Sound. Previous works with lobster and blue crab in New York, Connecticut and New Jersey waters (Belton *et al.* 1985; Rappe *et al.* 1991; Hauge *et al.* 1994; Skinner *et al.* 1997; Skinner 2001) were limited primarily to the New York-New Jersey Harbor and New York Bight. These areas may be impacted by a known 2,3,7,8-TCDD source in the Passaic River watershed (Belton *et al.* 1985; Umbreit *et al.* 1986; USEPA 1987; Rappe *et al.* 1991), which is tributary to the harbor complex.

Several authors (previously cited) have demonstrated that chlorinated dioxins and furans (PCDD/Fs) preferentially accumulate in the hepatopancreas of lobster and blue crab. PCDD/F concentrations in muscle tissues are typically one to two orders of magnitude lower than in the hepatopancreas, and are often not detected in muscle even in some of the most contaminated environments. It is instructive to compare concentrations of major PCDD/Fs in lobster hepatopancreas from the New York-New Jersey Harbor estuary and New York Bight with those from Long Island Sound in the current study (Table 13). Lobster from Long Island Sound contain some of the lowest levels of each of the chlorinated dioxins and furans. The co-dominance of 2,3,7,8-TCDD apparent in the samples from the Passaic River, Newark Bay and the Arthur Kill-Kill Van Kull system, near a source of 2,3,7,8-TCDD, and of 2,3,7,8-TCDF in New York Harbor and New York Bight sites (related to PCB sources in the Hudson River and in metropolitan New York City) is lacking in Long Island Sound lobster specimens.

### Relationships to human health and environmental criteria

Various criteria for chemical residues are present or proposed for the protection of human or wildlife consumers of fish and other aquatic life. Table 14 includes some criteria for chemical residues in fish and shellfish recommended for use or used in New York and Connecticut for the protection of human health, or the regulation of commercial fisheries, or for assessments of environmental health threats. The US Environmental Protection Agency and the US Food and Drug Administration (USFDA) have agreed that the use of USFDA action levels or tolerances for the purposes of making local advisory determinations is inappropriate (USEPA 2009). USFDA tolerance or action levels are acceptable for regulatory use where commercial fisheries contain chemical adulteration. Health advisory assessments by health professionals using one or more of a variety of possible evaluation mechanisms (e.g., assessments of compliance with established or proposed criteria, risk assessment, risk-benefit analysis, judgment by health professionals) may be employed. These assessments are not within the purview of this study. However, the criteria do provide the reader with an opportunity to compare the data obtained in this study with the various criteria to obtain an idea of the relative importance of the concentrations of the analytes that were determined.

The ecological criteria cited are for protection of fish consuming wildlife. However, the criteria were designed for protection of sensitive terrestrial wildlife and may not necessarily be protective of wildlife associated with the marine environment. Criteria for protection of marine wildlife are generally not available.

### Health advisories

As a consequence of health professionals' assessments of the data obtained by this study, the Connecticut Department of Public Health and the New York State Department of Health have modified their health advisories for consumers of fish (CTDPH 2009, NYSDOH 2009). The new health advisories, based in part on the findings of this study, are provided in Table 15. Changes from the health advisories prior to 2009 can be determined by examining Tables 1 and 15.

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**Table 1:** Health advisories<sup>1</sup> for people consuming fish or lobster taken from the marine district of New York and Connecticut, including Long Island Sound, in 2008.

<u>State</u>	<u>Species</u>	<u>Health advice</u>
New York	Striped bass	Women of childbearing age and children under the age of 15 should eat no striped bass from Upper and Lower Bays of New York Harbor, Raritan Bay or Long Island Sound west of Wading River. Other people should eat no more than one meal per month of striped bass from these waters. Everyone should eat no more than one meal per week of striped bass taken from Jamaica Bay, eastern Long Island Sound, Block Island Sound, Peconic/Gardiners Bay or Long Island south shore waters.
	Bluefish & American eel	Women of childbearing age and children under the age of 15 years should eat no bluefish or American eel (or any other fish) from the Upper Bay of New York Harbor. Other people should eat no more than one meal per month of bluefish or American eel from Upper Bay. The general advisory (eat no more than one meal (one-half pound) per week) applies to bluefish and American eels but not to most other fish from Long Island Sound, Block Island Sound, Peconic/Gardiners Bays, the Lower Bay of New York Harbor, Raritan Bay, Jamaica Bay and other Long Island south shore waters.
	Lobster & Blue crab	The hepatopancreas (sometimes called mustard, tomalley or liver) of crabs and lobsters from any waters should not be eaten because of high contaminant levels. Because contaminants in the hepatopancreas are transferred to cooking liquid, crab and lobster cooking liquid should also be discarded.
Connecticut	Striped bass	High risk group <sup>2</sup> – Do not eat Low risk group – One meal per two months
	Bluefish > 25"	High risk group – Do not eat Low risk group – One meal per two months
	13" – 25"	High Risk group – One meal per month Low risk group – One meal per month

<sup>1</sup> Sources: NYSDOH 2008; CTDPH 2008.

<sup>2</sup> CTDPH high risk group is defined as pregnant women, women planning to become pregnant within one year, nursing mothers, and children under six years of age. The low risk group includes all other people.

Table 2: Concentrations of polychlorinated biphenyls, mercury and lipids in four species of fish and the hepatopancreas of American lobster taken from Long Island Sound in 2006 and 2007.

Year	Area	n	Length (mm) <sup>1</sup>	Weight (g)	Concentration			
					Lipid (%)	PCB (µg/g wet wt.)	PCB (µg/g lipid)	Mercury (µg/g wet wt.)
Striped bass								
2006	1	24	717 ± 108	3981 ± 2290	1.18 ± 1.04	0.223 ± 0.150	25.46 ± 20.17	0.297 ± 0.155
			608 - 1001	730 - 10295	0.36 - 4.97	0.090 - 0.605	8.50 - 90.15	0.110 - 0.808
	2	37	877 ± 144	6716 ± 3804	1.51 ± 1.24	0.250 ± 0.180	21.01 ± 14.99	0.530 ± 0.280
			648 - 1150	1074 - 16010	0.17 - 6.70	0.053 - 0.845	6.32 - 79.05	0.194 - 1.55
	3	21	701 ± 75	3505 ± 1239	1.63 ± 1.35	0.307 ± 0.293	30.45 ± 44.94	0.264 ± 0.114
			610 - 918	2410 - 7850	0.48 - 5.54	0.094 - 1.45	7.63 - 215.7	0.110 - 0.466
	4	21	796 ± 154	5527 ± 3250	1.79 ± 1.25	0.240 ± 0.123	21.58 ± 33.17	0.329 ± 0.145
			622 - 1100	2300 - 13100	0.23 - 5.06	0.019 - 0.445	5.83 - 160.9	0.129 - 0.613
All	103		787 ± 146	5197 ± 3216	1.51 ± 1.22	0.283 ± 0.159	24.09 ± 28.22	0.381 ± 0.230
			622 - 1150	730 - 16010	0.17 - 6.70	0.019 - 1.45	5.83 - 215.7	0.110 - 1.55
2007	1	13	709 ± 72	3485 ± 1163	1.91 ± 2.29	0.492 ± 0.261	56.37 ± 59.07	0.248 ± 0.0789
			604 - 840	2050 - 6535	0.23 - 8.17	0.208 - 1.12	13.49 - 228.3	0.120 - 0.351
	2	4	919 ± 150	7721 ± 3789	2.14 ± 1.65	0.529 ± 0.219	31.25 ± 11.70	0.507 ± 0.133
			726 - 1084	3100 - 12020	0.60 - 4.40	0.269 - 0.716	16.27 - 44.83	0.355 - 0.636
	3	10	740 ± 104	4349 ± 2244	2.37 ± 2.21	0.498 ± 0.275	47.74 ± 57.42	0.277 ± 0.113
			617 - 962	2470 - 9875	0.12 - 6.47	0.227 - 0.955	14.76 - 195.8	0.166 - 0.443
	4	2	944	8240	3.72	0.670	18.44	0.479
			898 - 990	6910 - 9570	3.04 - 4.40	0.632 - 0.708	16.09 - 20.79	0.386 - 0.572
All	29		765 ± 125	4695 ± 2606	2.23 ± 2.09	0.511 ± 0.247	47.32 ± 52.01	0.309 ± 0.138
			604 - 1084	2050 - 12020	0.12 - 8.17	0.208 - 1.12	13.49 - 228.3	0.120 - 0.636

Table 2 continued

Year	Area	n	Length (mm) <sup>1</sup>	Weight (g)	Concentration			
					Lipid (%)	PCB (µg/g wet wt.)	PCB (µg/g lipid)	Mercury (µg/g wet wt.)
Bluefish (305 to 508 mm)								
2006	1	9	404 ± 54	611 ± 248	0.97 ± 1.02	0.070 ± 0.030	11.70 ± 9.14	0.254 ± 0.117
			323 - 480	285 - 1005	0.27 - 3.51	0.030 - 0.121	3.45 - 31.85	0.128 - 0.447
	2	10	417 ± 53	689 ± 247	0.86 ± 0.51	0.076 ± 0.061	8.75 ± 2.73	0.302 ± 0.115
			322 - 476	310 - 980	0.42 - 1.87	0.028 - 0.237	6.21 - 13.60	0.149 - 0.495
	3	6	410 ± 29	698 ± 185	0.57 ± 0.19	0.057 ± 0.046	11.59 ± 11.93	0.223 ± 0.104
			384 - 463	540 - 1050	0.42 - 0.94	0.018 - 0.148	3.46 - 35.24	0.118 - 0.395
	4	0	--	--	--	--	--	--
	All	25	411 ± 47	663 ± 228	0.83 ± 0.69	0.069 ± 0.047	10.49 ± 7.90	0.266 ± 0.114
			322 - 480	285 - 1050	0.27 - 3.51	0.018 - 0.237	3.45 - 35.24	0.118 - 0.495
2007	1	1	479	970	0.92	0.082	8.91	0.266
			--	--	--	--	--	--
	2	0	--	--	--	--	--	--
	3	4	468 ± 36	920 ± 178	1.54 ± 0.77	0.219 ± 0.082	15.02 ± 3.97	0.271 ± 0.063
			431 - 504	725 - 1090	0.86 - 2.65	0.140 - 0.315	11.83 - 20.08	0.231 - 0.363
	4	5	457 ± 37	835 ± 181	1.28 ± 0.81	0.229 ± 0.091	22.36 ± 10.76	0.300 ± 0.0342
			414 - 499	665 - 1040	0.66 - 2.38	0.156 - 0.375	8.38 - 34.93	0.252 - 0.331
	All	10	464 ± 33	883 ± 167	1.35 ± 0.73	0.211 ± 0.089	18.08 ± 8.96	0.285 ± 0.046
			431 - 504	665 - 1090	0.66 - 2.65	0.082 - 0.375	8.38 - 34.93	0.231 - 0.363

Table 2 continued

Year	Area	N	Length (mm) <sup>1</sup>	Weight (g)	Concentration			
					Lipid (%)	PCB (µg/g wet wt.)	PCB (µg/g lipid)	Mercury (µg/g wet wt.)
Bluefish (>508 mm)								
2006	1	26	668 ± 62	2501 ± 613	2.86 ± 2.32	0.390 ± 0.264	15.25 ± 6.39	0.294 ± 0.0789
			533 - 787	1365 - 3719	0.60 - 9.25	0.080 - 1.01	8.57 - 32.17	0.169 - 0.495
	2	30	706 ± 88	3085 ± 1207	4.77 ± 4.19	0.667 ± 0.711	15.25 ± 7.50	0.392 ± 0.141
			539 - 845	1370 - 5865	0.55 - 17.6	0.051 - 3.17	5.69 - 39.30	0.223 - 0.694
	3	13	610 ± 62	1721 ± 764	2.54 ± 3.07	0.353 ± 0.301	27.23 ± 35.55	0.287 ± 0.155
			524 - 737	461 - 3295	0.29 - 10.4	0.086 - 1.17	7.94 - 132.8	0.016 - 0.595
	4	42	702 ± 52	2892 ± 625	4.06 ± 3.33	0.449 ± 0.260	14.47 ± 7.34	0.368 ± 0.125
			631 - 816	2010 - 4390	0.69 - 13.50	0.106 - 1.28	3.54 - 34.06	0.216 - 0.689
All	111		684 ± 73	2716 ± 925	3.79 ± 3.42	0.483 ± 0.445	16.36 ± 14.07	0.348 ± 0.130
			524 - 816	461 - 5865	0.29 - 17.6	0.051 - 3.17	3.54 - 132.8	0.016 - 0.694
2007	1	2	710	3306	5.47	1.37	26.35	0.366
			708 - 711	2980 - 3632	3.92 - 7.01	1.22 - 1.51	21.54 - 31.15	0.364 - 0.369
	2	12	710 ± 68	2876 ± 811	2.39 ± 1.96	0.852 ± 0.680	51.07 ± 59.73	0.419 ± 0.162
			633 - 816	1975 - 4155	0.66 - 7.43	0.252 - 2.70	15.58 - 228.4	0.268 - 0.775
	3	17	690 ± 63	2857 ± 701	6.37 ± 4.37	0.802 ± 0.485	19.56 ± 17.22	0.340 ± 0.162
			580 - 833	1855 - 4505	0.35 - 14.00	0.217 - 1.90	5.99 - 62.00	0.166 - 0.853
All	31		--	--	--	--	--	--
			699 ± 62	2893 ± 723	4.78 ± 3.93	0.858 ± 0.561	32.20 ± 41.26	0.372 ± 0.159
			580 - 833	1855 - 4505	0.35 - 14.00	0.217 - 2.70	5.99 - 228.4	0.166 - 0.853

Table 2 continued

Year	Area	N	Length (mm) <sup>1</sup>	Weight (g)	Concentration			
					Lipid (%)	PCB (µg/g wet wt.)	PCB (µg/g lipid)	Mercury (µg/g wet wt.)
American eel								
2007	1	2	616	375	5.32	0.385	7.25	0.0539
			606 - 625	330 - 420	5.15 - 5.49	0.375 - 0.395	6.83 - 7.67	0.0535 - 0.0543
	2	12	621 ± 68	511 ± 231	10.95 ± 3.97	0.513 ± 0.083	5.37 ± 2.37	0.116 ± 0.032
			499 - 724	210 - 910	4.44 - 16.5	0.380 - 0.657	2.50 - 11.28	0.0898 - 0.175
	3	0						
	4	1	671	605	13.3	0.668	5.02	0.147
	All	15	624 ± 62	499 ± 213	10.36 ± 4.12	0.506 ± 0.097	5.60 ± 2.21	0.110 ± 0.037
			499 - 724	210 - 910	4.44 - 16.5	0.375 - 0.668	2.50 - 11.28	0.0535 - 0.175
Weakfish								
2007	1	1	467	1015	3.69	0.245	6.64	0.160
	2	16	479 ± 120	1223 ± 1064	4.86 ± 3.35	0.388 ± 0.273	9.53 ± 4.34	0.133 ± 0.068
			406 - 844	625 - 4850	0.85 - 12.2	0.178 - 1.35	4.01 - 20.94	0.0548 - 0.337
	3	8	527 ± 177	1854 ± 1832	8.47 ± 3.85	0.791 ± 0.568	11.01 ± 10.13	0.156 ± 0.141
			387 - 830	605 - 5365	5.65 - 17.7	0.335 - 1.85	4.51 - 28.86	0.0602 - 0.430
	4	0						
	All	25	494 ± 136	1417 ± 1335	5.97 ± 3.80	0.512 ± 0.424	9.89 ± 6.53	0.141 ± 0.094
			387 - 844	605 - 5365	0.85 - 17.7	0.178 - 1.85	4.01 - 28.86	0.0548 - 0.430

Table 2 continued

Year	Area	N	Length (mm) <sup>1</sup>	Weight (g)	Concentration			
					Lipid (%)	PCB (µg/g wet wt.)	PCB (µg/g lipid)	Mercury (µg/g wet wt.)
American lobster – hepatopancreas								
2007	1	9	85.1 ± 2.0	428 ± 83	15.8 ± 8.70	1.49 ± 0.503	10.49 ± 2.56	0.0503 ± 0.015
			83.0 – 89.3	345 - 610	5.1 – 33.4	0.779 – 2.22	6.65 – 15.28	0.0283 – 0.0721
	2	15	86.8 ± 2.1	439 ± 52	9.61 ± 4.83	1.37 ± 0.609	15.25 ± 3.56	0.0636 ± 0.0191
			84.3 – 92.7	365 - 555	3.9 – 18.6	0.790 – 2.51	8.33 – 20.85	0.0370 – 0.0982
	3	26	86.6 ± 3.8	482 ± 117	13.5 ± 8.84	1.29 ± 0.585	12.73 ± 10.23	0.0636 ± 0.0283
			83.0 – 95.5	285 - 745	1.86 – 36.1	0.520 – 2.83	4.85 – 50.27	0.0298 – 0.162
	4	15	87.7 ± 2.9	588 ± 51	9.90 ± 6.09	1.20 ± 0.484	15.73 ± 10.30	0.111 ± 0.0275
			83.2 – 93.0	510 - 670	1.99 – 23.0	0.367 – 1.94	5.54 – 39.49	0.0661 – 0.145
	All	65	86.7 ± 3.10	489 ± 104	12.10 ± 7.65	1.31 ± 0.553	13.69 ± 8.43	0.073 ± 0.032
			83.0 – 95.5	285 - 745	1.86 – 36.1	0.367 – 2.83	4.85 – 50.27	0.0283 – 0.162

<sup>1</sup> Total length for fish; carapace length for lobster.

Table 3: Cadmium concentrations in the hepatopancreas of American lobster taken from Long Island Sound in 2007.

Year	Area	n	Cadmium ( $\mu\text{g/g}$ wet weight)	
			Mean $\pm$ SD	Min. – Max.
2007	1	9	3.49 $\pm$ 2.41	1.36 – 9.01
	2	15	4.88 $\pm$ 3.97	0.848 – 15.5
	3	26	4.42 $\pm$ 3.60	0.879 – 17.3
	4	15	4.32 $\pm$ 1.45	2.08 – 6.79
	All areas	65	4.374 $\pm$ 3.143	0.848 – 17.3

Table 4: Chlorinated dibenzo-*p*-dioxins and dibenzofurans in the hepatopancreas of American lobster taken from Long Island Sound in 2007.

Parameter	Concentration (pg/g wet weight) in <sup>1</sup> :				
	Area 1	Area 2	Area 3	Area 4	Entire Sound
N	14	15	20	15	64
2,3,7,8-TCDD	1.99 ± 1.06 0.75 – 4.5	1.24 ± 1.09 <0.048 – 2.8 5 (33)	1.42 ± 1.28 <0.33 – 5.8 1 (5.0)	0.793 ± 0.793 <0.058 – 2.1 6 (40)	1.36 ± 1.14 <0.058 – 5.8 12 (19)
1,2,3,7,8-PeCDD	4.17 ± 1.60 2.0 – 7.6	2.70 ± 2.07 <0.24 – 7.5 4 (26)	3.73 ± 2.04 1.6 – 10	2.40 ± 1.61 <0.078 – 5.3 2 (13)	3.27 ± 1.95 <0.078 – 10 6 (9.4)
1,2,3,4,7,8-HxCDD	1.93 ± 0.933B <sup>2</sup> <0.15 – 3.4 1 (7.1)	2.19 ± 0.883 1.1 – 4.3	1.81 ± 0.899B 0.66 – 4.6	0.953 ± 0.903 <0.10 – 3.1	1.72 ± 0.991B <0.10 – 4.6 1 (1.6)
1,2,3,6,7,8-HxCDD	6.48 ± 3.00 2.9 – 13	8.85 ± 3.78 4.2 – 16	6.94 ± 3.42 2.7 – 17	4.56 ± 3.86 0.53 – 16	6.73 ± 3.75 0.53 – 17
1,2,3,7,8,9-HxCDD	2.24 ± 1.17 <0.55 – 4.3 1 (7.1)	2.89 ± 1.19 1.5 – 6.1	2.22 ± 1.23 <0.15 – 5.3 2 (10)	1.07 ± 1.10B <0.083 – 3.5 5 (33)	2.11 ± 1.32B <0.083 – 6.1 8 (12)
1,2,3,4,6,7,8-HpCDD	7.75 ± 3.71B 1.8 – 14	9.09 ± 3.23 4.5 – 15	6.44 ± 3.45B <0.20 – 17 1 (5.0)	2.75 ± 1.93 <0.084 – 6.4 2 (13)	6.48 ± 3.86B <0.084 – 17 3 (4.6)
OCDD	10.3 ± 4.54B 2.5 – 19	11.1 ± 3.92B 6.8 – 20	8.28 ± 3.10B 3.9 – 15	3.37 ± 2.18 <0.23 – 7.6 2 (13)	8.23 ± 4.49B <0.23 – 20 2 (3.1)
2,3,7,8-TCDF	20.9 ± 34.0 <0.08 – 110 9 (64)	53.1 ± 21.8 30 – 100	38.8 ± 30.7 <0.072 – 120 4 (20)	46.1 ± 22.8 11 – 85	39.9 ± 29.6 <0.072 – 120 13 (20)
1,2,3,7,8-PeCDF	7.44 ± 3.87B 3.8 – 15	11.4 ± 4.72 6.5 – 22	9.14 ± 6.20B <0.14 – 26 1 (5.0)	7.96 ± 6.02 1.0 – 24	9.02 ± 5.46B <0.14 – 26 1 (1.6)
2,3,4,7,8-PeCDF	14.2 ± 6.52 6.8 – 30	16.7 ± 7.15 10 – 33	13.5 ± 5.92 7.1 – 32	12.2 ± 7.53 3.0 – 30	14.1 ± 6.78 3.0 – 33
1,2,3,4,7,8-HxCDF	1.76 ± 0.77B 0.58 – 2.9	0.475 ± 0.574 <0.043 – 1.5 8 (53)	1.33 ± 0.967B <0.077 – 3.6 5 (25)	0.365 ± 0.397 <0.061 – 1.2 6 (40)	0.998 ± 0.915B <0.061 – 3.6 19 (29)

1,2,3,6,7,8-HxCDF	<0.38 <0.080 - <0.38 14 (100)	<0.29 <0.054 - <0.29 14 (100)	<0.51 <0.066 - <0.51 20 (100)	0.016 ± 0.062 <0.043 - 0.24 14 (93)	<0.51 <0.043 - 0.24 63 (98)
2,3,4,6,7,8-HxCDF	2.66 ± 1.24B 1.4 - 5.8	2.73 ± 1.12 1.4 - 5.1	2.44 ± 0.823B 0.95 - 4.2	0.970 ± 0.870B <0.14 - 3.0 2 (13)	2.21 ± 1.21B <0.14 - 5.8 2 (3.1)
1,2,3,7,8,9-HxCDF	<0.40 <0.084 - <0.40 14 (100)	<0.24 <0.049 - <0.24 15 (100)	<0.44 <0.085 - <0.44 20 (100)	<0.28 <0.066 - <0.28 15 (100)	<0.44 <0.049 - <0.44 64 (100)
1,2,3,4,6,7,8-HpCDF	0.719 ± 0.376B <0.15 - 1.3 1 (7.1)	0.237 ± 0.230B <0.057 - 0.62 6 (40)	0.697 ± 0.435B <0.15 - 1.7 3 (15)	0.102 ± 0.147B <0.063 - 0.48 8 (53)	0.455 ± 0.422B <0.057 - 1.7 18 (28)
1,2,3,4,7,8,9-HpCDF	<0.57 <0.14 - <0.57 (0.23B) <sup>3</sup> 13 (92)	<0.34 <0.049 - <0.34 15 (100)	<0.58 <0.060 - <0.58 20 (100)	<0.24 <0.080 - <0.24 15 (100)	<0.58 <0.049 - <0.58 (0.23B) <sup>3</sup> 63 (98)
OCDF	0.323 ± 0.461B <0.23 - 1.3 8 (57)	0.239 ± 0.353B <0.078 - 1.3 7 (46)	0.296 ± 0.316B <0.12 - <1.1 (1.0) <sup>3</sup> 9 (45)	0.453 ± 0.211B <0.23 - 0.70 2 (13)	0.325 ± 0.343B <0.078 - 1.3 26 (40)
2,3,7,8-TCDD TEQ <sup>4</sup> (DL = 0)	14.3 ± 6.06 5.91 - 26.8	16.4 ± 6.19 10.0 - 31.7	14.9 ± 7.81 6.52 - 38.3	12.5 ± 7.32 2.71 - 26.7	14.6 ± 6.95 2.71 - 38.3
2,3,7,8-TCDD TEQ <sup>4</sup> (½DL)	14.3 ± 6.11 5.92 - 27.1	16.5 ± 6.20 10.1 - 31.8	14.9 ± 7.81 6.52 - 38.3	12.6 ± 7.28 2.75 - 26.7	14.6 ± 6.94 2.75 - 38.3
Total TCDD	20.9 ± 11.1 7.1 - 43	17.6 ± 7.95 9.5 - 34	16.6 ± 7.91 1.5 - 31	5.79 ± 4.18 0.78 - 18	15.2 ± 9.61 0.78 - 43
Total PeCDD	30.8 ± 14.5 13 - 61	34.3 ± 14.1 19 - 68	30.0 ± 11.8 15 - 64	9.13 ± 5.45 0.50 - 22	26.3 ± 15.2 0.50 - 68
Total HxCDD	35.1 ± 17.4 14 - 69	64.0 ± 27.1 38 - 130	31.7 ± 24.6 3.4 - 110	19.0 ± 14.0 2.8 - 54	37.1 ± 26.7 2.8 - 130
Total HpCDD	20.1 ± 8.68B 4.6 - 33	22.3 ± 8.60 12 - 42	16.1 ± 7.59 <0.20 - 32 1 (5.0)	5.80 ± 3.59 0.59 - 14	16.0 ± 9.50 <0.20 - 42 1 (1.6)

Total TCDF	122 ± 72.4 29 – 280	150 ± 60.9 74 – 280	106 ± 54.5 46 – 280	81.0 ± 43.0 11 – 180	114 ± 61.7 11 – 280
Total PeCDF	111 ± 51.2 46 – 220	101 ± 47.5 53 – 200	97.6 ± 45.8 46 – 230	42.6 ± 24.6 8.6 – 95	88.4 ± 49.9 8.6 – 230
Total HxCDF	20.9 ± 10.9B 8.1 – 43	14.7 ± 11.7 1.9 – 35	17.4 ± 8.54B 5.8 – 45	3.50 ± 3.25B 0.74 – 13	14.3 ± 10.9B 0.74 – 45
Total HpCDF	0.757 ± 0.359B <0.18 – 1.3 1 (7.1)	0.237 ± 0.230B <0.063 – 0.62 6 (40)	0.697 ± 0.435B <0.17 – 1.7 4 (20)	0.102 ± 0.147B <0.069 – 0.48 8 (53)	0.463 ± 0.424B <0.063 – 1.7 19 (29)

<sup>1</sup> The data presented for each analyte on each line are:

Mean ± standard deviation concentrations;

Minimum – maximum concentrations; and

If a third line is present, the number and (%) of non-detects.

For computations, and unless otherwise specified in the Parameter column, a zero was substituted where a reportable concentration was not found.

<sup>2</sup> B = Blank contamination; blank may represent 10 percent or more of the reported value.

<sup>3</sup> The maximum detected value (in parenthesis) is less than the greatest detection limit.

<sup>4</sup> Human and mammalian TEQs calculated per Van den Burg (2005).

Table 5: Length-lipid and length-contaminant relationships (wet weight basis) for several Long Island Sound fish and the hepatopancreas of American lobster.

<u>Species</u>	<u>Year</u>	<u>n</u>	<u>R<sup>2</sup></u>	<u>Best fit type</u>	<u>Equation (where significant)</u>
<u>Lipids</u>					
Striped bass	2006	103	0.0009	Exponential	
	2007	29	0.0917	Linear	
Bluefish	2006	136	0.4497	Exponential	$y = 0782e^{0.0052x}$
	2007	41	0.1597	Power	
American eel	2007	15	0.5620	Linear	$y = 0.0496x - 20.573$
Weakfish	2007	24	0.0435	Power	
American lobster	2007	65	0.0011	Linear	
<u>Total PCBs</u>					
Striped bass	2006	103	0.0035	Linear	
	2007	29	0.0977	Linear	
Bluefish	2006	136	0.6081	Exponential	$y = 0.0051e^{0.0062x}$
	2007	41	0.6885	Exponential	$y = 0.012e^{0.0059x}$
American eel	2007	15	0.0505	Linear	
Weakfish	2007	24	0.7193	Linear	$y = 0.0026x - 0.7912$
American lobster <sup>1</sup>	2007	65	0.0224	Exponential	

<u>Species</u>	<u>Year</u>	<u>n</u>	<u>R<sup>2</sup></u>	<u>Best fit type</u>	<u>Equation (where significant)</u>
<u>Mercury</u>					
Striped bass	2006	103	0.5613	Linear	$y = 0.0012x - 0.5597$
	2007	29	0.8155	Linear	$y = 0.007x - 0.1962$
	Combined	132	0.5848	Exponential	$y = 0.0327e^{0.0029x}$
Bluefish 305 to 508 mm	2006	25	0.0439	Linear	
	2007	10	0.0014	Exponential	
	Combined	142	0.4597	Linear	
> 508 mm	2006	111	0.4302	Linear	$y = 0.0012x - 0.4545$
	2007	31	0.6072	Linear	$y = 0.0020x + 1.0123$
	Combined	142	0.4597	Linear	$y = 0.0013x - 0.5479$
All sizes	2006	136	0.2471	Linear	$y = 0.005x + 0.0093$
	2007	41	0.3290	Linear	$y = 0.0007x - 0.1042$
	Combined	177	0.2606	Linear	$y = 0.005x - 0.0045$
American eel	2007	15	0.4149	Linear	$y = 0.004x + 0.1321$
Weakfish	2007	24	0.7447	Linear	$y = 0.0006x - 0.1526$
American lobster <sup>1</sup>	2007	65	0.0949	Power	
<u>Cadmium</u>					
American lobster <sup>1</sup>	2007	65	0.0077	Power	
<u>2,3,7,8-TCDD toxic equivalents<sup>2</sup></u>					
American lobster <sup>1</sup>	Male	39	0.0689	Linear	
	Female	25	0.2367	Linear	

<sup>1</sup> Carapace length vs contaminant in hepatopancreas.

<sup>2</sup> Chlorinated dibenzo-*p*-dioxins and dibenzofurans only.

Table 6: Influence of season on lipid content in striped bass and bluefish taken from Long Island Sound in 2006 and 2007.

Species	Year	Concentration (%)				p
		Spring		Fall		
		n	Mean ± SD	n	Mean ± SD	
Striped bass	2006	53	1.61 ± 1.10	50	1.41 ± 1.35	0.047
	2007	11	3.06 ± 2.39	18	1.72 ± 1.76	0.028
Bluefish 305 to 508 mm	2006	9	0.44 ± 0.082	16	1.05 ± 0.79	<0.001
	2007	2	2.01	8	1.18 ± 0.64	nc
Bluefish >508 mm	2006	10	1.50 ± 0.96	101	4.02 ± 3.49	0.003
	2007	14	2.94 ± 2.18	17	6.29 ± 4.45	0.015

Table 7: Influence of season on total PCB concentrations in striped bass and bluefish taken from Long Island Sound, 2006 and 2007.

Species	Year	Area	Basis	Concentration ( $\mu\text{g/g}$ ) <sup>1</sup>				P	
				Spring		Fall			
				n	Mean $\pm$ SD	n	Mean $\pm$ SD		
Striped bass	2006	1	Wet	17	0.258 $\pm$ 0.166	7	0.139 $\pm$ 0.032		
			Lipid	17	26.87 $\pm$ 22.92	7	22.05 $\pm$ 11.77		
		2	Wet	19	0.275 $\pm$ 0.188	18	0.585 $\pm$ 0.223		
			Lipid	19	17.92 $\pm$ 9.75	18	24.28 $\pm$ 18.79		
		3	Wet	9	0.209 $\pm$ 0.098	12	0.380 $\pm$ 0.368		
			Lipid	9	12.92 $\pm$ 3.75	12	43.61 $\pm$ 56.76		
	4	Wet	8	0.194 $\pm$ 0.159	13	0.269 $\pm$ 0.090			
		Lipid	8	10.02 $\pm$ 2.61	13	28.70 $\pm$ 41.06			
	All	Wet	53	0.246 $\pm$ 0.163	50	0.261 $\pm$ 0.221	0.474		
		Lipid	53	18.75 $\pm$ 15.40	50	29.75 $\pm$ 36.64	0.019		
	2007	All	Wet	11	0.577 $\pm$ 0.284	18	0.471 $\pm$ 0.220		0.179
			Lipid	11	25.53 $\pm$ 16.96	18	60.63 $\pm$ 61.59		0.010
Bluefish (305 – 508 mm)	2006	All	Wet	9	0.0633 $\pm$ 0.0401	16	0.0728 $\pm$ 0.0510	0.213	
			Lipid	9	15.34 $\pm$ 11.19	16	7.77 $\pm$ 3.30	0.031	
	2007	All	Wet	8	0.204 $\pm$ 0.0911	2	0.239	Isn <sup>2</sup>	
			Lipid	8	19.63 $\pm$ 9.46	2	11.86	Isn	
	2006	All	Wet	10	0.271 $\pm$ 0.129	101	0.504 $\pm$ 0.460	0.024	
			Lipid	10	28.26 $\pm$ 37.26	101	15.18 $\pm$ 8.7	0.061	
2007	All	Wet	(9)	(16.65 $\pm$ 6.74)					
		Lipid	14	0.932 $\pm$ 0.718	17	0.797 $\pm$ 0.404	0.358		
			14	43.91 $\pm$ 56.09	17	22.55 $\pm$ 20.44	0.018		
			(13)	(29.72 $\pm$ 18.81)					

<sup>1</sup> Parenthetic values exclude outliers.

<sup>2</sup> Isn = Insufficient sample numbers.

Table 8: Influence of season on mercury concentrations in striped bass and bluefish taken from Long Island Sound, 2006 and 2007.

Species	Year	Concentration ( $\mu\text{g/g}$ wet weight)				P
		Spring		Fall		
		n	Mean $\pm$ SD	n	Mean $\pm$ SD	
Striped bass	2006	53	0.361 $\pm$ 0.178	50	0.401 $\pm$ 0.274	0.515
	2007	11	0.352 $\pm$ 0.115	18	0.284 $\pm$ 0.148	0.040
Bluefish 305 – 508 mm	2006 + 2007	18	0.324 $\pm$ 0.0941	17	0.216 $\pm$ 0.0702	< 0.001
Bluefish >508 mm	2006	10	0.370 $\pm$ 0.110	101	0.346 $\pm$ 0.132	0.063
	2007	14	0.443 $\pm$ 0.183	17	0.314 $\pm$ 0.109	0.010
	2006 + 2007	24	0.412 $\pm$ 0.158	118	0.342 $\pm$ 0.129	0.008

Table 9: Spatial differences in lipids and chemical residue concentrations in three species of fish and in the hepatopancreas of American lobster in Long Island Sound, 2006 and 2007.

Analyte	Species	Year	Was there a spatial difference?	P	Area description <sup>1</sup>
Lipids	Striped bass	2006	No	0.298	
		2007	No	0.676	
	Bluefish 305 - 508 mm	2006	No	0.755	
		2007	Insufficient samples		
	Bluefish > 508 mm	2006	Yes	0.0355	<u>3 1</u> 4 2
		2007	Yes	0.039	— 2 < 3
Weakfish <508 mm	2007	Yes	0.012	2 < 3	
Am. lobster	2007	Male – Yes	0.039	4 < 3 = 2 = 1	
		Female - No	0.332		
PCBs (wet weight)	Striped bass	2006	No	0.920	
		2007	No	0.859	
	Bluefish 305 - 508 mm	2006	No	0.439	
		2007	Insufficient samples		
	Bluefish > 508 mm	2006	No	0.0866	
		2007	No	0.235	
Weakfish <508 mm	2007	Yes	0.008	3 < 2	
Am. lobster	2007	Male - Yes	0.0412	4 < 3 = 1 = 2	
		Female - No	0.329		
PCBs (lipid basis)	Striped bass	2006	No	0.168	
		2007	No	0.583	
	Bluefish 305 - 508 mm	2006	No	0.846	
		2007	Insufficient samples		
	Bluefish > 508 mm	2006	No	0.235	
		2007	Yes	<0.001	3 < 2
Weakfish <508 mm	2007	Yes	0.017	3 < 2	
Am. lobster	2007	Male – No	0.386	3 = 1 < 2	
		Female – Yes	0.0045		
Mercury	Striped bass	2006 + 2007	Yes	<0.001	3 = 1 = 2 < 4
		2006 + 2007	No	0.485	
	Bluefish > 508 mm	2006 + 2007	Yes	0.0081	<u>1 3</u> 4 2
		2007	No	0.095	—
	Am. lobster	2007	Yes	<0.001	1 = 3 = 2 < 4
Cadmium	Am. lobster	2007	No	0.505	
		2007	No	0.922	
PCDD/F (as TEQs)	Am. lobster	2007	No	0.922	

<sup>1</sup> Areas are ranked from lowest to highest. Generalized areas are:

Area 1 = western Long Island Sound (shared waters),

Area 2 = north-central (Connecticut) Long Island Sound,

Area 3 = south-central (New York) Long Island Sound, and

Area 4 = eastern Long Island Sound (shared waters).

A line connecting area numbers indicates the concentrations are statistically ( $P > 0.05$ ) the same.

Table 10: Sex as an influence on chemical residue levels in Long Island Sound fish and American lobster.

Species	Chemical	Year	Areas	n	Males		Females		P
					Mean ± SD	n	Mean ± SD	n	
Striped bass	Lipids	2006	All	27	1.12 ± 1.18	76	1.66 ± 1.22	0.002	
		2007	All	7	2.85 ± 1.96	22	2.03 ± 2.13	0.131	
	Total PCBs (wet weight)	2006	All	27	0.330 ± 0.290	76	0.226 ± 0.136	0.046	
		2007	All	7	0.636 ± 0.216	22	0.472 ± 0.247	0.069	
	Total PCBs (lipid based)	2006	All	27	43.77 ± 46.42	76	17.10 ± 12.12	<0.001	
2007		All	7	32.20 ± 18.76	22	52.13 ± 58.35	0.324		
	Mercury	2006/2007	All	34	0.345 ± 0.153	98	0.372 ± 0.232	0.482	
Bluefish 305 – 508 mm	Lipids	2006	All	10	0.99 ± 0.95	14	0.75 ± 0.47	0.270	
		2007	All	6	1.16 ± 0.77	4	1.61 ± 0.66	0.101	
	Total PCBs (wet weight)	2006	All	10	0.0732 ± 0.0272	14	0.0610 ± 0.0544	0.025	
		2007	All	6	0.205 ± 0.0680	4	0.219 ± 0.127	0.416	
	Total PCBs (lipid based)	2006	All	10	11.35 ± 8.67	14	8.11 ± 3.09	0.279	
2007		All	6	21.28 ± 9.74	4	13.28 ± 5.64	0.068		
	Mercury	2006/2007	All	16	0.251 ± 0.0794	18	0.283 ± 0.112	0.176	
>508 mm	Lipids	2006	All	36	4.20 ± 4.09	71	3.50 ± 3.06	0.448	
		2007	All	11	3.23 ± 3.75	19	5.55 ± 3.95	0.041	
	Total PCBs (wet weight)	2006	All	36	0.498 ± 0.505	71	0.459 ± 0.418	0.482	
		2007	All	12	0.938 ± 0.647	19	0.795 ± 0.509	0.274	
	Total PCBs (lipid based)	2006	All	36	18.15 ± 21.39	71	15.48 ± 8.86	0.297	
2007		All	11	49.36 ± 60.82	71	21.34 ± 16.50	0.026		
	Mercury	2006/2007	All	47	0.362 ± 0.151	90	0.346 ± 0.132	0.277	

Species	Chemical	Year	Areas	n	Males		Females		P
					Mean ± SD	n	Mean ± SD	n	
Weakfish 387 – 508 mm	Lipids	2007	All	9	5.99 ± 5.09	11	5.83 ± 2.96	0.392	
	Total PCBs								
	- wet weight	2007	All	9	0.416 ± 0.211	11	0.328 ± 0.0899	0.247	
	- lipid based	2007	All	9	9.88 ± 5.48	11	6.38 ± 1.83	0.044	
	Mercury	2007	All	9	0.105 ± 0.0370	11	0.108 ± 0.0468	0.689	
	>508 mm	Lipids	2007	All	4	7.05 ± 3.59	1	2.98	nc <sup>1</sup>
	Total PCBs								
	- wet weight	2007	All	4	1.28 ± 0.575	1	0.305	nc	
	- lipid based	2007	All	4	19.48 ± 9.06	1	10.24	nc	
	Mercury	2007	All	4	0.312 ± 0.113	1	0.147	nc	
American eels (presumptive females)	Lipids	2007	All	0		15	10.36 ± 4.12	nc	
	Total PCBs								
	- wet weight	2007	All	0		15	0.506 ± 0.0973	nc	
	- lipid based	2007	All	0		15	5.60 ± 2.21	nc	
Mercury	2007	All	0		15	0.110 ± 0.0374	nc		
American lobster - hepatopancreas	Lipids	2007	1-3	24	17.31 ± 8.89	26	8.56 ± 3.80	< 0.001	
		2007	4	15	9.90 ± 6.09	0		nc	
	Total PCBs								
	(wet weight)	2007	1-3	24	1.704 ± 0.522	26	1.023 ± 0.399	< 0.001	
			4	15	1.195 ± 0.484	0		nc	
	Total PCBs	2007	1-3	24	12.41 ± 8.66	26	13.71 ± 7.03	0.0575	
(lipid based)		4	15	15.73 ± 10.30	0		nc		

<u>Species</u>	<u>Chemical</u>	<u>Year</u>	<u>Areas</u>	<u>n</u>	<u>Males</u>	<u>Females</u>	<u>P</u>	
					<u>Mean ± SD</u>	<u>n</u>		<u>Mean ± SD</u>
American lobster - hepatopancreas	Mercury	2007	1-3	24	0.0650 ± 0.0293	26	0.0577 ± 0.0177	0.356
			4	15	0.111 ± 0.0275	0	nc	
	Cadmium	2007	1-3	24	5.28 ± 2.75	26	3.56 ± 3.96	< 0.001
			4	15	4.32 ± 1.45	0	nc	
	2,3,7,8-TCDD TEQs	2007	1-3	24	18.2 ± 7.87	25	12.3 ± 3.90	0.006
			4	15	12.5 ± 7.31	0	nc	

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<sup>1</sup> nc = Not calculated due to insufficient sample numbers.

Table 11: Polychlorinated biphenyl congeners in bluefish from Long Island Sound in 2006.

Homolog Group	IUPAC No.	PCB congener concentration (ng/g wet weight)		No. of detections	PCB homolog concentration (ng/g wet weight)	
		Mean	Min. – Max.		Mean	Min. – Max.
Mono-	1	<50	<50 - <50	0	<150	<150 - <150
	2	<50	<50 - <50	0		
	3	<50	<50 - <50	0		
Di-	4	62.3	<50 – 547	5	248	82 – 1220
	5	<50	<50 - <50	0		
	6	8.00	<50 – 150	2		
	7	<50	<50 - <50	0		
	8	43.6	<50 – 435	8		
	9	<50	<50 - <50	0		
	10	5.92	<50 – 78.6	2		
	11	122	16 – 360	25		
	12/13	<50	<50 - <50	0		
	14	<50	<50 - <50	0		
	15	2.48	<50 – 62	1		
Tri-	16	84.3	<50 – 985	9	7098	181 - 74200
	17	386	<50 – 4360	20		
	18/30	569	<50 – 6580	21		
	19	206	<50 – 2920	7		
	20/28	2394	127 – 23800	25		
	21/33	219	<50 – 2180	20		
	22	322	<50 – 3390	21		
	23	<50	<50 - <50	0		
	24	<50	<50 - <50	0		
	25	305	<50 – 3680	19		
	26/29	723	<50 – 7190	21		
27	117	<50 – 1200	8			
31	1351	53.8 – 15400	25			

Homolog Group	IUPAC No.	PCB congener concentration (ng/g wet weight)		No. of detections	PCB homolog concentration (ng/g wet weight)	
		Mean	Min. – Max.		Mean	Min. – Max.
Tri-	32	376	<50 – 3870	20	44476	2090 – 447000
	34	11.1	<50 – 203	2		
	35	<50	<50 - <50	0		
	36	<50	<50 - <50	0		
	37	33.7	<50 – 551	3		
	38	<50	<50 - <50	0		
	39	<50	<50 - <50	0		
Tetra-	40/41/71	1762	106 – 19200	25		
	42	1344	60.8 – 14700	25		
	43	98.0	<50 – 1310	10		
	44/47/65	5438	294 – 54600	25		
	45/51	418	<50 – 4570	22		
	46	69.3	<50 – 919	8		
	48	319	<50 – 3670	20		
	49/69	5874	255 – 60100	25		
	50/53	433	<50 – 4530	21		
	52	7515	374 – 75800	25		
	54	3.32	<50 – 83.1	1		
	55	<50	<50 - <50	0		
	56	1184	64.8 – 11900	25		
	57	57.0	<50 – 621	8		
	58	70.0	<50 – 908	9		
	59/62/75	503	<50 – 5060	23		
	60	798	<50 – 7510	24		
61/70/74/76	8223	440 – 80800	25			
63	374	<50 – 3540	22			
64	1890	85.8 – 19600	25			
66	6915	405 – 65900	25			
67	199	<50 – 2050	18			

Homolog Group	IUPAC No.	PCB congener concentration (ng/g wet weight)		No. of detections	PCB homolog concentration (ng/g wet weight)	
		Mean	Min. – Max.		Mean	Min. – Max.
Tetra-	68	245	<50 – 2350	21		
	72	332	<50 – 3240	22		
	73	<50	<50 - <50	0		
	77	271	<50 – 2840	20		
	78	<50	<50 - <50	0		
	79	119	<50 – 1220	16		
	80	2.76	<50 – 68.9	1		
	81	7.95	<50 – 144	2		
	Penta-	82	1001	65.3 – 11200	25	118688
83		711	<50 – 8240	23		
84		1723	102 – 18700	25		
85/116/117		3300	257 – 33500	25		
86/87/97/108/ 119/125		9622	680 – 102000	25		
88/91		2732	184 – 32200	25		
89		26.0	<50 – 451	3		
90/101/113		22016	1590 – 232000	25		
92		4397	311 – 46700	25		
93/98/100/102		744	<75 – 7850	24		
94		49.4	<50 – 722	7		
95		7662	503 – 81700	25		
96		20.8	<50 – 369	3		
99		20441	1620 – 188000	25		
103		518	<50 – 5850	23		
104		<50	<50 - <50	0		
105		4682	334 – 45200	25		
106		<50	<50 - <50	0		
107/124		447	<50 – 4920	23		
109		2639	209 – 22600	25		
110/115	15120	988 – 163000	25			
111	110	<50 – 1100	17			

Homolog Group	IUPAC No.	PCB congener concentration (ng/g wet weight)		No. of detections	PCB homolog concentration (ng/g wet weight)	
		Mean	Min. – Max.		Mean	Min. – Max.
Penta-	112	<50	<50 - <50	0		
	114	211	<50 – 2320	20		
	118	19430	1460 – 169000	25		
	120	513	<50 – 4920	24		
	121	27.8	<50 – 436	5		
	122	73.0	<50 – 960	10		
	123	415	<50 – 5040	22		
	126	75.9	<50 – 1080	8		
	127	17.8	<50 – 362	2		
Hexa-	128/166	4234	356 – 38300	25	158964	12900 – 1450000
	129/138/163	36730	2900 – 316000	25		
	130	2323	178 – 22300	25		
	131	186	<50 – 2170	20		
	132	5045	346 – 48900	25		
	133	1189	<50 – 11300	24		
	134/143	1005	<50 – 11000	24		
	135/151	9849	733 – 101000	25		
	136	2077	145 – 22300	25		
	137	847	<50 – 8450	24		
	139/140	631	<50 – 6520	24		
	141	2234	139 – 22300	25		
	142	<50	<50 - <50	0		
	144	925	58.3 – 9900	25		
	145	<50	<50 - <50	0		
	146	11600	1100 – 101000	25		
	147/149	21791	1730 – 217000	25		
148	372	<50 – 3520	24			
150	178	<50 – 1870	20			
152	<50	<50 - <50	0			
153/168	48354	4230 – 414000	25			
154	2044	199 – 19300	25			

Homolog Group	IUPAC No.	PCB congener concentration (ng/g wet weight)		No. of detections	PCB homolog concentration (ng/g wet weight)	
		Mean	Min. – Max.		Mean	Min. – Max.
Hexa-	155	308	<50 – 2920	23		
	156/157	2102	154 – 20200	25		
	158	1930	129 – 19400	25		
	159	59.7	<50 – 1150	5		
	160	<50	<50 - <50	0		
	161	<50	<50 - <50	0		
	162	279	<50 – 3340	20		
	164	1173	75.3 – 12900	25		
	165	149	<50 – 1330	20		
	167	1342	104 – 12700	25		
	169	<50	<50 - <50	0		
	Hepta-	170	3920	338 – 36900	25	62341
171/173		1974	210 – 16800	25		
172		1188	127 – 10500	25		
174		3092	317 – 29100	25		
175		530	61.5 – 4590	25		
176		764	78.8 – 7130	25		
177		4717	549 – 40700	25		
178		3783	473 – 31300	25		
179		3240	310 – 31200	25		
180/193		11096	1160 – 101000	25		
181		19.9	<50 – 413	2		
182		113	<50 – 936	19		
183/185		6448	740 – 53200	25		
184		95.2	<50 – 815	19		
186		<50	<50 - <50	0		
187		20257	2650 – 152000	25		
188		284	<50 – 2470	23		
189		174	<50 – 1620	22		
190	463	<50 – 5370	24			
191	193	<50 – 1810	22			

Homolog Group	IUPAC No.	PCB congener concentration (ng/g wet weight)		No. of detections	PCB homolog concentration (ng/g wet weight)	
		Mean	Min. – Max.		Mean	Min. – Max.
Hepta-	192	<50	<50 - <50	0		
Octa-	194	1981	133 – 19400	25	16190	1650 - 146000
	195	707	55.0 – 6650	25		
	196	1681	156 – 16100	25		
	197/200	630	<250 – 5320	21		
	198/199	3999	412 – 40000	25		
	201	1642	253 – 11700	25		
	202	3155	424 – 24100	25		
	203	2338	217 – 21700	25		
	204	5.64	<50 – 141	1		
	205	100	<50 – 1010	18		
Nona-	206	3882	426 – 28900	25	6764	770 - 52900
	207	591	70.8 – 4880	25		
	208	2289	274 – 19100	25		
Deca-	209	2946	289 - 25800	25	2945	289 - 25800
Total congeners		417891	33837 – 3916861			

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Table 12: Temporal comparison of polychlorinated biphenyl concentrations in bluefish greater than 508 mm taken from Long Island Sound.

<u>Sample type</u>	<u>Year</u>	<u>n</u>	<u>Concentration (<math>\mu\text{g/g}</math>)</u>		<u>Change (%)<sup>1</sup></u>		<u>Reference</u>
			<u>Wet weight</u> <u>Mean <math>\pm</math> SD</u>	<u>Lipid basis</u> <u>Mean <math>\pm</math> SD</u>	<u>Wet weight</u>	<u>Lipid basis</u>	
Individuals	1985	16	2.693 $\pm$ 1.405	21.44 $\pm$ 11.64			NOAA/EPA/FDA 1986a
	2006	111	0.483 $\pm$ 0.445	16.36 $\pm$ 14.07	- 82 (- 74)	- 23 (- 23)	This study
	2007	31 30 <sup>2</sup>	0.858 $\pm$ 0.561	32.20 $\pm$ 41.26 25.66 $\pm$ 19.75	- 68 (- 54)	+ 50 (+ 50) + 19 (+ 28)	This study
Composites of 5 fish	1985	62 61 <sup>2</sup>	1.894 $\pm$ 1.137	21.37 $\pm$ 13.48 19.99 $\pm$ 7.98			NOAA/FDA/EPA 1986a

<sup>1</sup> Percentage change from 1985 to the year indicated. Comparisons are of individuals to individuals and, in parenthesis, individuals to composites. For lipid based data excluding outliers (i.e., the + 28 value), the individual to composite comparison is with 1985 data without the outlier.

<sup>2</sup> Data excludes one outlier value.

Table 13: Comparison of major chlorinated dioxin and furan concentrations and 2,3,7,8-TCDD toxic equivalents in hepatopancreas of American lobster and blue crabs from marine waters of New York, New Jersey and Connecticut.

Location	Year	n	Concentration (pg/g wet weight)					2,3,7,8-TCDD TEQs <sup>2</sup>	Reference
			2,3,7,8-TCDD	2,3,7,8-TCDF	1,2,3,7,8-PeCDD	1,2,3,7,8-PeCDF	2,3,4,7,8-PeCDF		
<u>American lobster</u>									
Long Island Sound	2007	64	1.36 ± 1.14	39.9 ± 29.6	3.27 ± 1.93	9.02 ± 5.46	14.1 ± 6.78	14.6 ± 6.95	This study
NY Bight									
- nearshore	1985-86	2	290	320	na <sup>1</sup>	na	na	322+	Hauge <i>et al.</i> 1991
- offshore	1985-86	4	<39	<33 (1)	na	na	na	0+	Hauge <i>et al.</i> 1991
NY Bight	~ 1989	2	434	366	84.6	79.5	179	736	Rappe <i>et al.</i> 1991
- offshore near former sewage sludge dump site									
NY Bight Apex (nearshore)	1993	2	79.8	332	26.2	36.6	69.3	173	Skinner <i>et al.</i> 1997
<u>Blue crab</u>									
Hudson River									
- Poughkeepsie	1999	7	13.4 ± 30.5	57.1 ± 54.0	1.08 ± 2.86	4.68 ± 5.96	9.65 ± 12.2	24.3 ± 43.5	McReynolds <i>et al.</i> 2004c
- Haverstraw	1999	6	13.0 ± 27.6	39.2 ± 24.6	0.0	1.58 ± 1.78	11.9 ± 7.09	21.2 ± 30.5	McReynolds <i>et al.</i> 2004c
Upper Bay	1993	2	29.2	274	13.1	28.0	53.2	96.0	Skinner <i>et al.</i> 1997
	1999	6	24.2 ± 38.1	44.4 ± 24.5	0.74 ± 1.82	4.03 ± 4.56	11.6 ± 7.68	34.5 ± 45.8	McReynolds <i>et al.</i> 2004c
Passaic River	~ 1989	2	4954	638	102	186 <sup>3</sup>	391	5284	Rappe <i>et al.</i> 1991
	1999	6	346 ± 243	31.6 ± 37.5	3.41 ± 3.79	10.6 ± 6.79	48.9 ± 38.9	375 ± 253	McReynolds <i>et al.</i> 2004c
Newark Bay	1995	6	135 ± 53.9	182 ± 5.69	6.81 ± 1.67	14.7 ± 4.60	31.6 ± 10.5	175 ± 61.7	Skinner <i>et al.</i> 1997
	1999	6	145 ± 64.5	104 ± 63.7	0.833 ± 2.04	8.92 ± 4.15	26.0 ± 10.7	170 ± 67.2	McReynolds <i>et al.</i> 2004c

<u>Location</u>	<u>Year</u>	<u>n</u>	<u>Concentration (pg/g wet weight)</u>						<u>Reference</u>
			<u>2,3,7,8-TCDD</u>	<u>2,3,7,8-TCDF</u>	<u>1,2,3,7,8-PeCDD</u>	<u>1,2,3,7,8-PeCDF</u>	<u>2,3,4,7,8-PeCDF</u>	<u>2,3,7,8-TCDD TEQs<sup>2</sup></u>	
<u>Blue crab (continued)</u>									
Arthur Kill/ Kill Van Kull	1993	2	155	138	8.40	15.3	38.2	200	Skinner <i>et al.</i> 1997
Raritan Bay	1999	6	16.7 ± 16.8	42.4 ± 26.6	3.07 ± 2.50	3.41 ± 3.32	9.16 ± 4.42	27.9 ± 18.8	McReynolds <i>et al.</i> 2004c
Jamaica Bay	1993	2	1.05	13.6	1.85	1.95	1.85	1.79	Skinner <i>et al.</i> 1997
	1999	6	19.6 ± 28.2	19.1 ± 10.8	0.0	1.73 ± 1.91	4.16 ± 5.31	23.2 ± 31.3	McReynolds <i>et al.</i> 2004c

<sup>1</sup> na = Not analyzed.

<sup>2</sup> Calculated 2,3,7,8-TCDD TEQs for mammals and humans using original data and toxicity equivalency factors of Van den Berg *et al.* (2006).

<sup>3</sup> Coelution with 1,2,3,4,8-PeCDF.

Table 14: Criteria for chemical residues in fish and shellfish for the protection of human health or piscivorous wildlife.

<u>Chemical(s)</u>	<u>Source</u>	<u>Quantity</u>
		<u>Criteria for Protection of Human Health</u>
PCBs	USFDA (1984a)	2.0 mg/kg (ppm) as a tolerance for PCBs in edible fish and shellfish <sup>1</sup> in interstate and international commerce
	USEPA (2002)	0.05 mg/kg Remedial Action Objective for Hudson River fish assuming ½ lb fish meal per week 0.2 mg/kg Remedial Action Objective for Hudson River fish assuming ½ lb. fish meal per month 0.4 mg/kg Remedial Action Objective for Hudson River fish assuming ½ lb. fish meal/2 months
	USEPA (2000)	Assuming consumption of an 8 oz. fish meal and noncarcinogenic risks, selected categories are: ≤ 0.0059 mg/kg for unrestricted fish consumption > 0.023 – 0.047 mg/kg for one meal per week > 0.094 – 0.19 mg/kg for one meal per month > 0.19 – 0.38 mg/kg for one meal per two months > 0.38 for eat none  Assuming consumption of an 8 oz. fish meal and an additional lifetime carcinogenic risk of 10 <sup>-6</sup> , selected categories are: ≤ 0.0015 mg/kg for unrestricted consumption > 0.0059 – 0.012 mg/kg for one meal per week > 0.023 – 0.047 mg/kg for one meal per month > 0.047 – 0.094 mg/kg for one meal per two months > 0.094 for eat none
	GLSFATF (1993)	Assuming consumption of an 8 oz. fish meal, ≤ 0.05 mg/kg for unrestricted fish consumption 0.06 – 0.2 mg/kg for one meal/week advice 0.21 – 1.0 mg/kg for one meal/month advice 1.1 - 1.9 mg/kg for six meals/year advice > 1.9 mg/kg for eat none advice

Mercury	USFDA (1984b)	1.0 mg/kg as methylmercury as an action level for edible fish and shellfish <sup>1</sup> in interstate and international commerce
	IJC (1988)	0.5 mg/kg
	USEPA (2000)	Assuming consumption of an 8 oz. fish meal and noncarcinogenic risks (as methylmercury), selected categories are: $\leq 0.029$ mg/kg for unrestricted fish consumption $> 0.12 - 0.23$ mg/kg for one meal per week $> 0.47 - 0.94$ mg/kg for one meal per month $> 0.94 - 1.9$ mg/kg for one meal per two months $> 1.9$ mg/kg for eat none
	USEPA (2001b)	0.3 mg/kg recommended methylmercury concentration in fish as an ambient water quality criterion
	GLSFATF (2007)	Assuming consumption of an 8 oz. fish meal $\leq 0.05$ mg/kg for unrestricted consumption $> 0.05 \leq 0.11$ mg/kg for two meals per week advice $> 0.11 \leq 0.22$ mg/kg for one meal per week advice $> 0.22 \leq 0.95$ mg/kg for one meal per month advice $> 0.95$ mg/kg for eat none advice
Cadmium	NYSDOH	1.0 mg/kg – level at which specific health advice may be recommended
	USFDA (1993) <sup>2</sup>	3.0 mg/kg – guidance level for consumption of crustaceans
	USFDA (2001)	4.0 mg/kg – guidance level for consumption of molluscan bivalves
	USFDA (2007)	
USEPA (2000)	Assuming consumption of an 8 oz. fish meal and noncarcinogenic risks, selected categories are: $\leq 0.088$ mg/kg for unrestricted fish consumption $> 0.35 - 0.7$ mg/kg for one meal per week $> 1.4 - 2.8$ mg/kg for one meal per month $> 2.8 - 5.6$ mg/kg for one meal per two months $> 5.6$ mg/kg for eat none	

Chlorinated dioxins/ furans	NYSDOH (1981)	10 pg/g as 2,3,7,8-TCDD toxic equivalents – level at which specific health advice may be recommended
	USEPA (2000)	Assuming consumption of an 8 oz. fish meal and an additional lifetime carcinogenic risk of 10 <sup>-6</sup> (as 2,3,7,8-TCDD toxic equivalents), selected categories are: ≤ 0.019 pg/g for unrestricted fish consumption > 0.075 – 0.15 pg/g for one meal per week > 0.3 – 0.6 pg/g for one meal per month > 0.6 – 1.2 pg/g for one meal per two months > 1.2 pg/g for eat none
<u>Criteria for Protection of Wildlife (whole fish)</u>		
PCBs	Newell <i>et al.</i> (1987)	0.11 mg/kg for protection of piscivorous wildlife
	IJC (1988)	0.1 mg/kg for protection of piscivorous wildlife
	USEPA (2002)	0.3 – 0.03 mg/kg in fish for protection of river otter; Hudson River Remedial Action Objective 0.7 – 0.07 mg/kg in fish for protection of mink; Hudson River Remedial Action Objective
Mercury	IJC (1988)	0.5 mg/kg for protection of piscivorous wildlife
2,3,7,8-TCDD	Newell <i>et al.</i> (1987)	2.3 pg/g for protection of piscivorous wildlife

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<sup>1</sup> In practice, US Food and Drug Administration guidance is that chemical residues in fish and shellfish must be below the tolerance for PCBs, or below action levels for other chemical residues for which there are recommended limits, with a probability of 95 percent or greater.

<sup>2</sup> USFDA now issues a disclaimer with the Guidance Documents for cadmium, arsenic, chromium, lead, and nickel. The disclaimer says:  
“‘This Guidance Document represented current agency thinking in regards to the available science at the time it was issued. It no longer represents the current state of science and is presented here for the historical record only.’”  
However, the values in the Guidance Documents continue to be included in other guidance documents (cited above), without qualification, for regulation of commercial shellfisheries. The USFDA has proposed to withdraw these guidance values.

Table 15: The 2009 health advisories for human consumers of fish and crustaceans taken from Connecticut and New York waters of Long Island Sound.

<u>State</u>	<u>Health advice<sup>1</sup></u>
Connecticut	<p>Striped bass and large bluefish (over 25 inches) caught in Long Island Sound should not be eaten by those in the high risk group: pregnant women, women of childbearing age, nursing mothers and children under the age of 6. The remainder of the general population should eat no more than one meal per month of these fish.</p> <p>All people should eat no more than one meal per month of bluefish 13 to 25 inches in total length.</p>
New York	<p>Women of childbearing age and children under the age of 15 should:</p> <ul style="list-style-type: none"> <li>• eat no weakfish greater than 25 inches in total length;</li> <li>• eat no more than one meal per month of American eel, striped bass, bluefish greater than 20 inches, and weakfish less than 25 inches; and</li> <li>• eat no more than one meal per week of smaller bluefish (less than or equal to 20 inches).</li> </ul> <p>Women beyond childbearing age and adult males should:</p> <ul style="list-style-type: none"> <li>• eat no more than one meal per month of weakfish greater than 25 inches in total length; and</li> <li>• eat no more than one meal per week of American eel, bluefish, striped bass and smaller weakfish.</li> </ul> <p>Everyone should avoid consuming the hepatopancreas ("the green stuff" also known as mustard, tomalley, liver) of crabs and lobsters, and discard crab or lobster cooking liquid.</p>

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<sup>1</sup> Sources: CTDPH 2009; NYSDOH 2009.

Figure 1: Sampling areas for fish and lobster in Long Island Sound.



Figure 2: Lipids in bluefish greater than 508 mm by sampling area in Long Island Sound

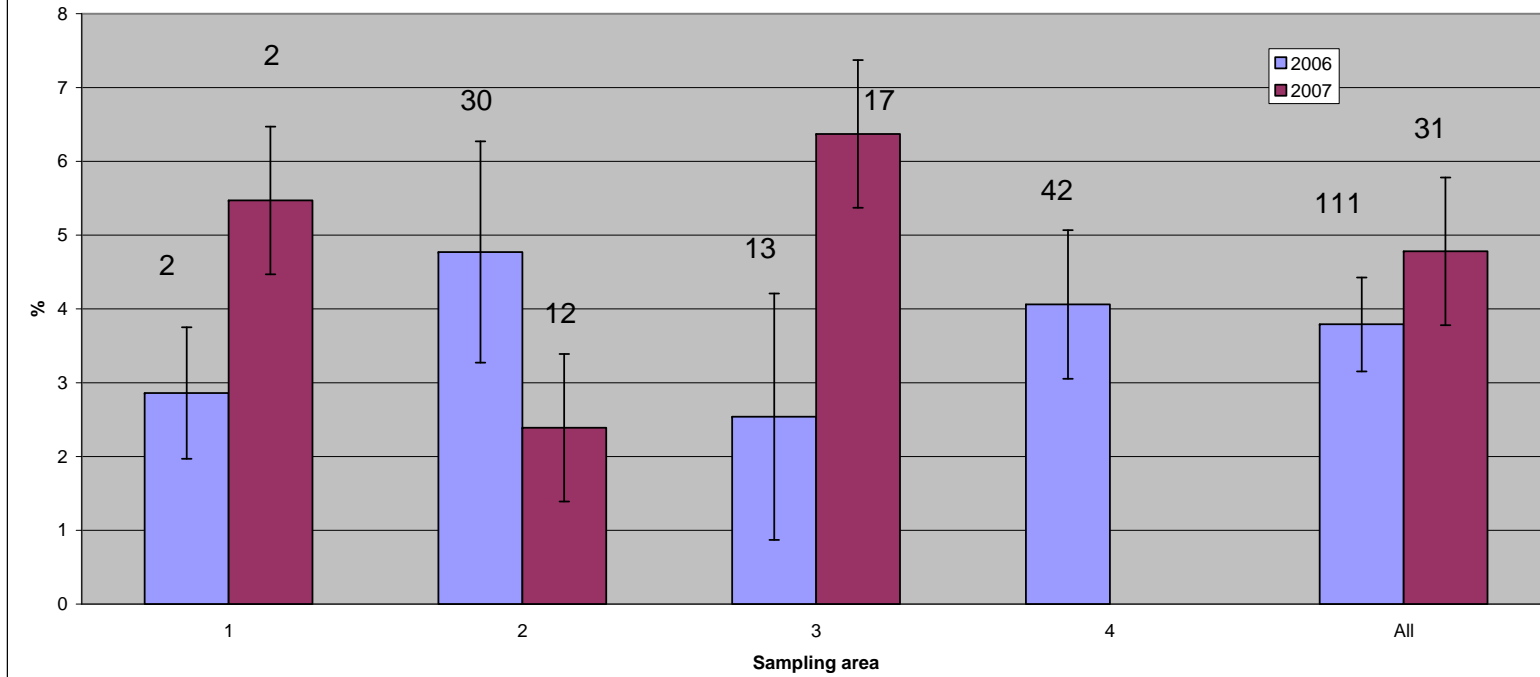
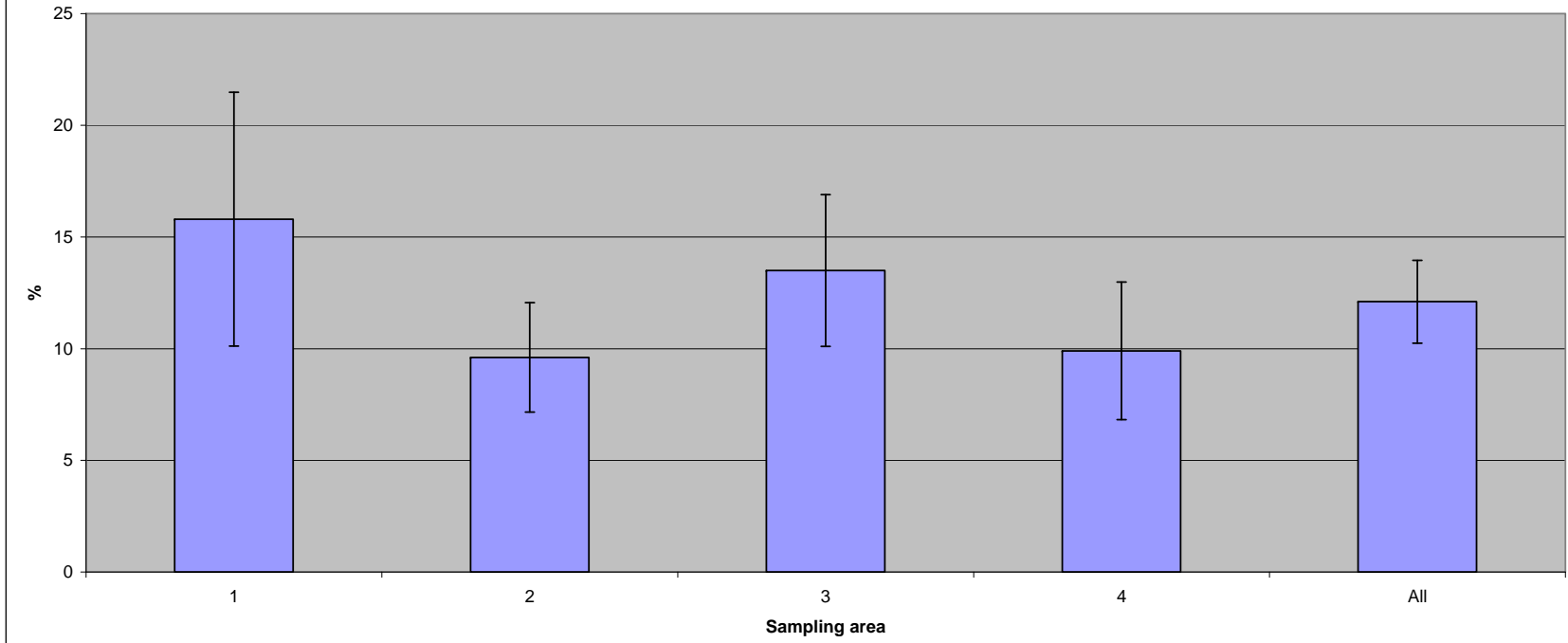


Figure 3: Lipids in the hepatopancreas of American lobster taken from Long Island Sound in 2007



**Figure 4: Length-total PCB relationships in striped bass taken from Long Island Sound**

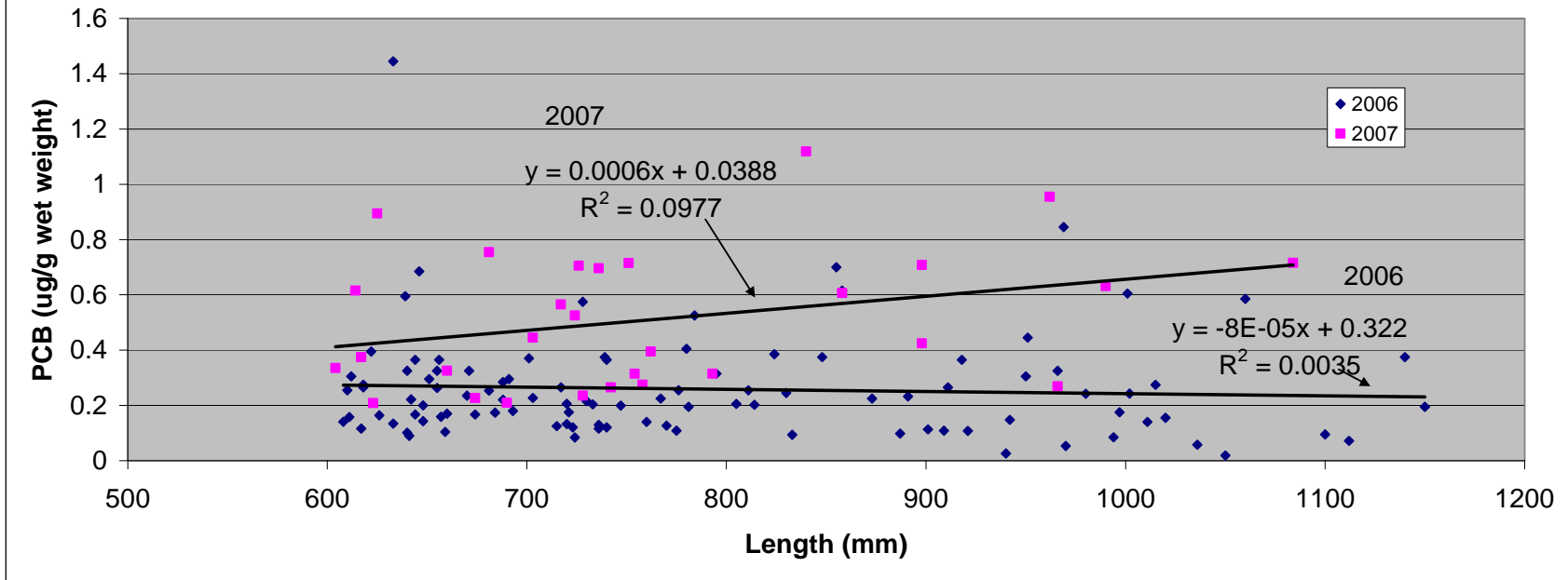


Figure 5: Length-total PCB relationships in bluefish taken from Long Island Sound

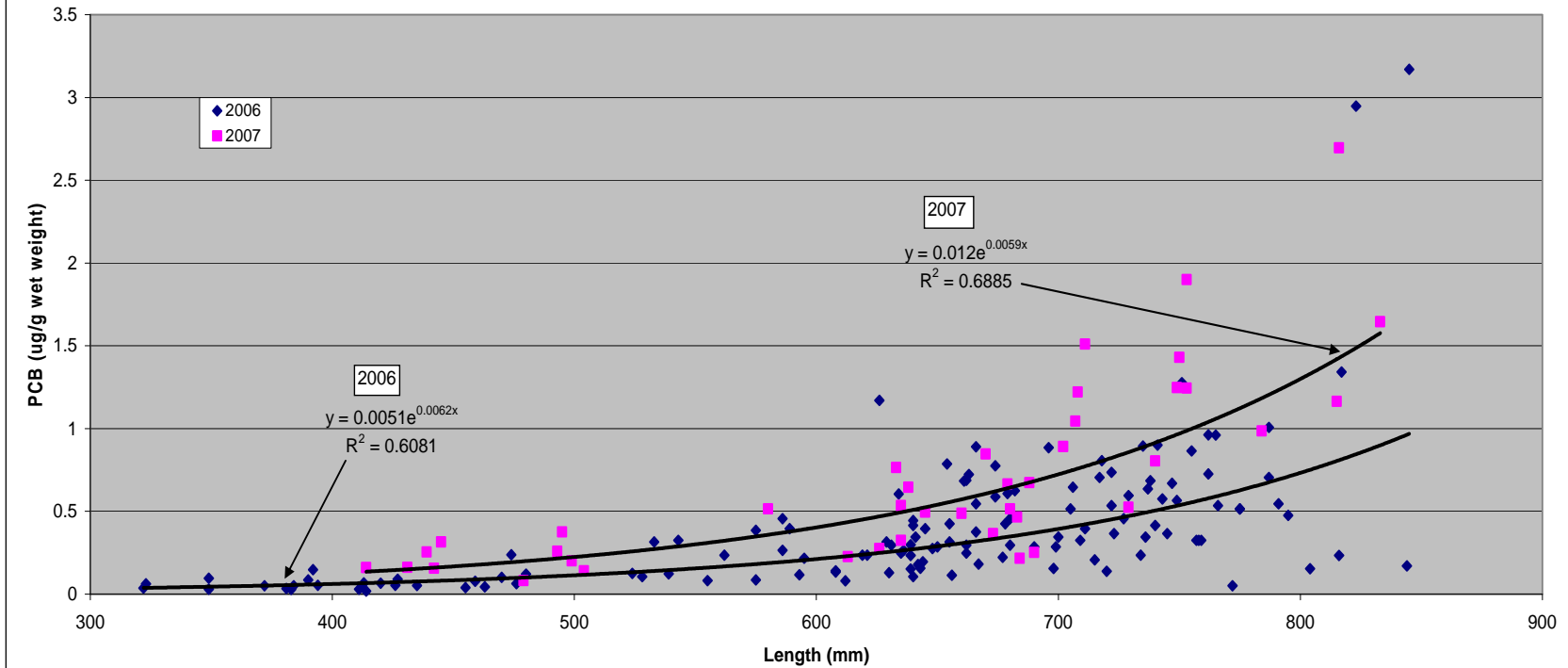


Figure 6: Length-total PCB relationship in weakfish taken from Long Island Sound in 2007

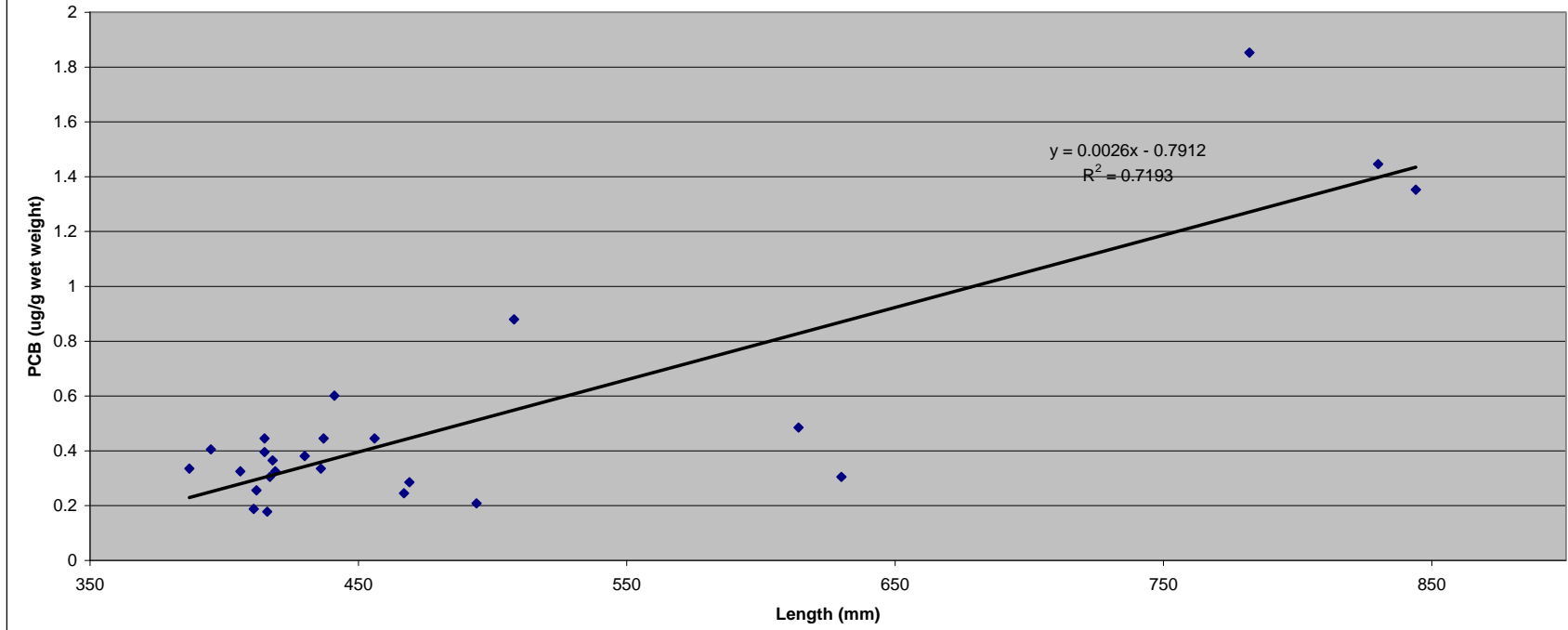


Figure 7: Length-mercury relationship in striped bass taken from Long Island Sound (2006 and 2007 combined)

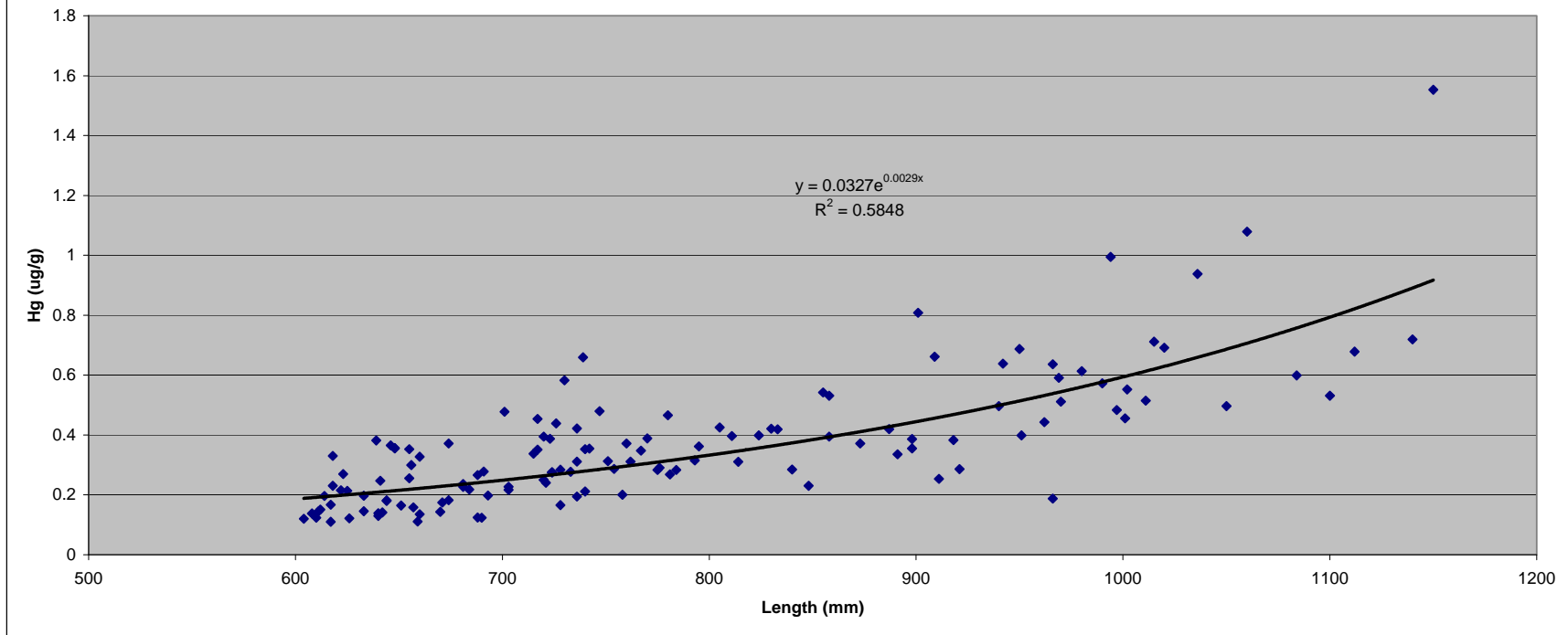


Figure 8: Length-mercury relationship in bluefish greater than 508 mm taken from Long Island Sound (2006 and 2007 combined)

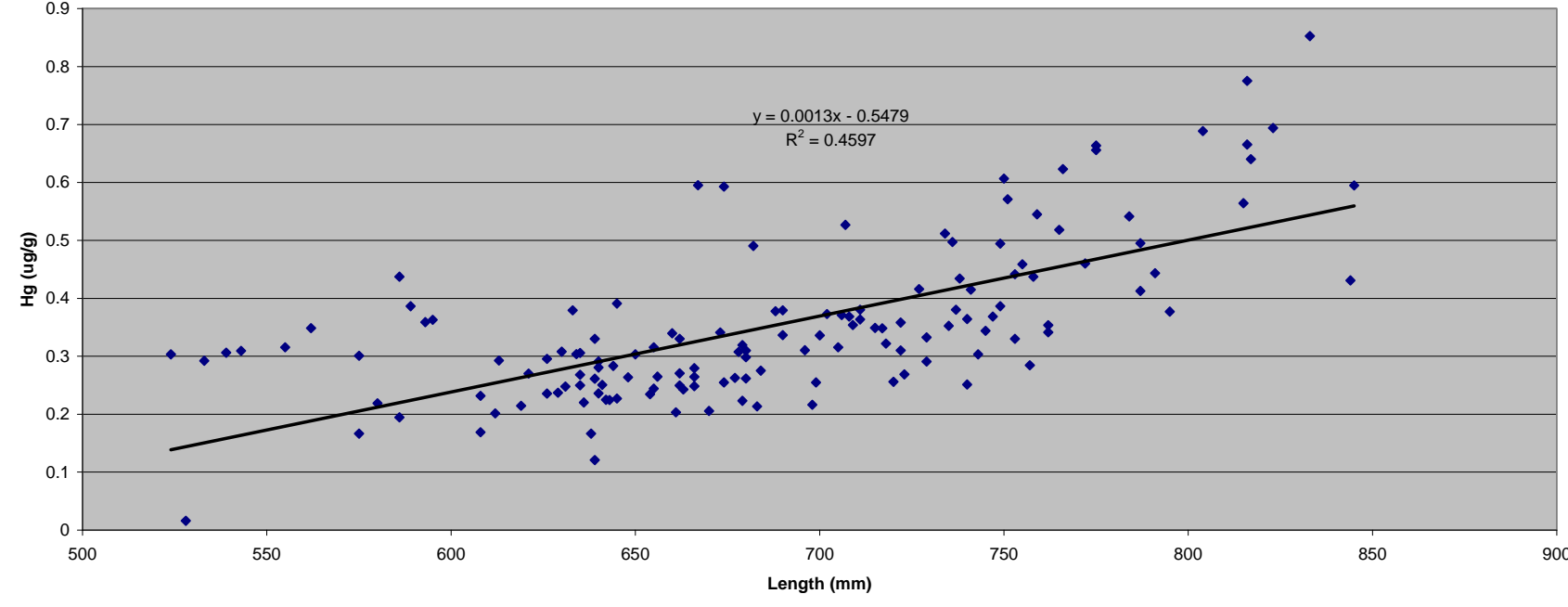


Figure 9: Length-mercury relationship in weakfish taken from Long Island Sound in 2007

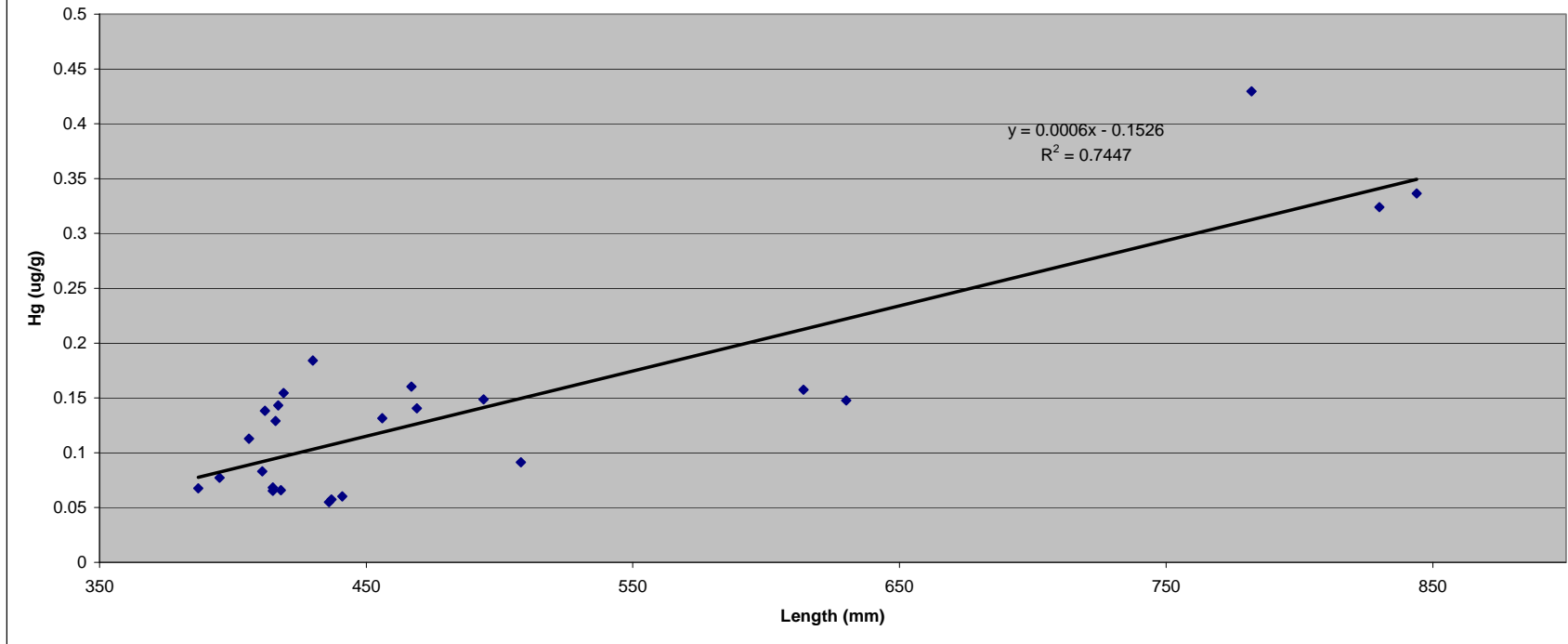
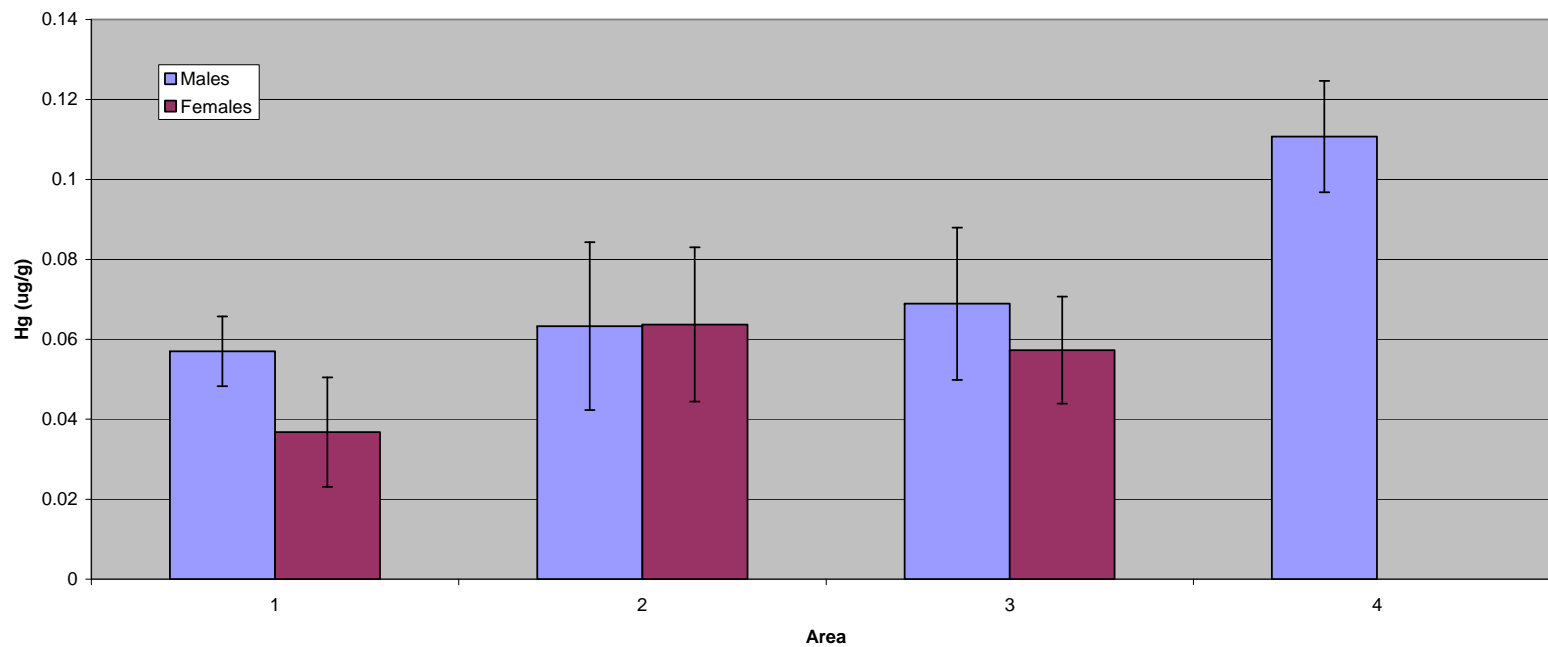


Figure 10: Mercury concentrations by area and sex in hepatopancreas of American lobster taken from Long Island Sound in 2007



**Figure 11: Contributions by PCB congener to total PCB in bluefish taken from Long Island Sound**

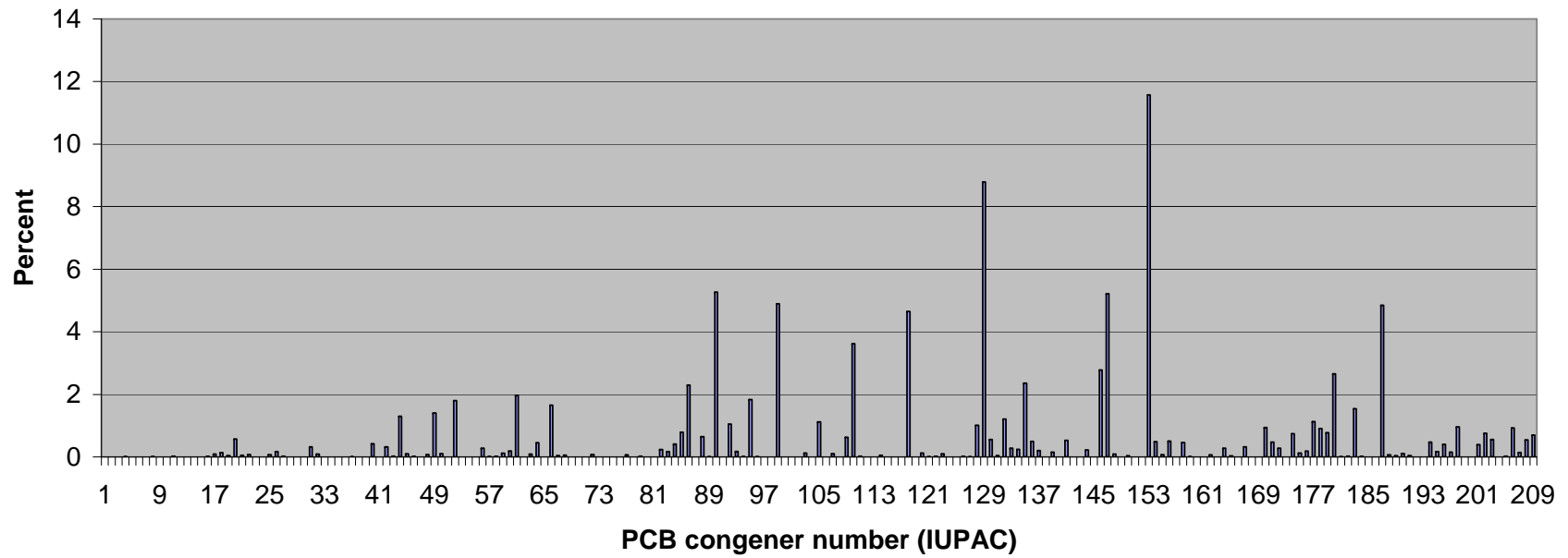
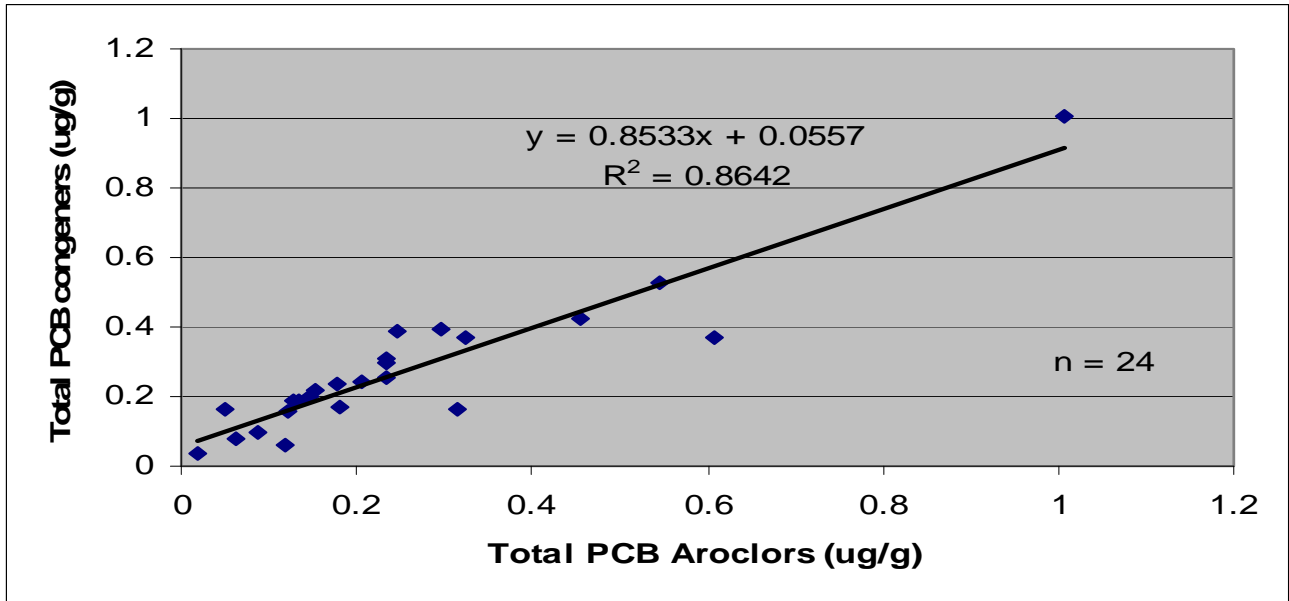


Figure 12: Total PCB concentrations in bluefish quantified as Aroclors vs congeners.

Without extreme value



With extreme value

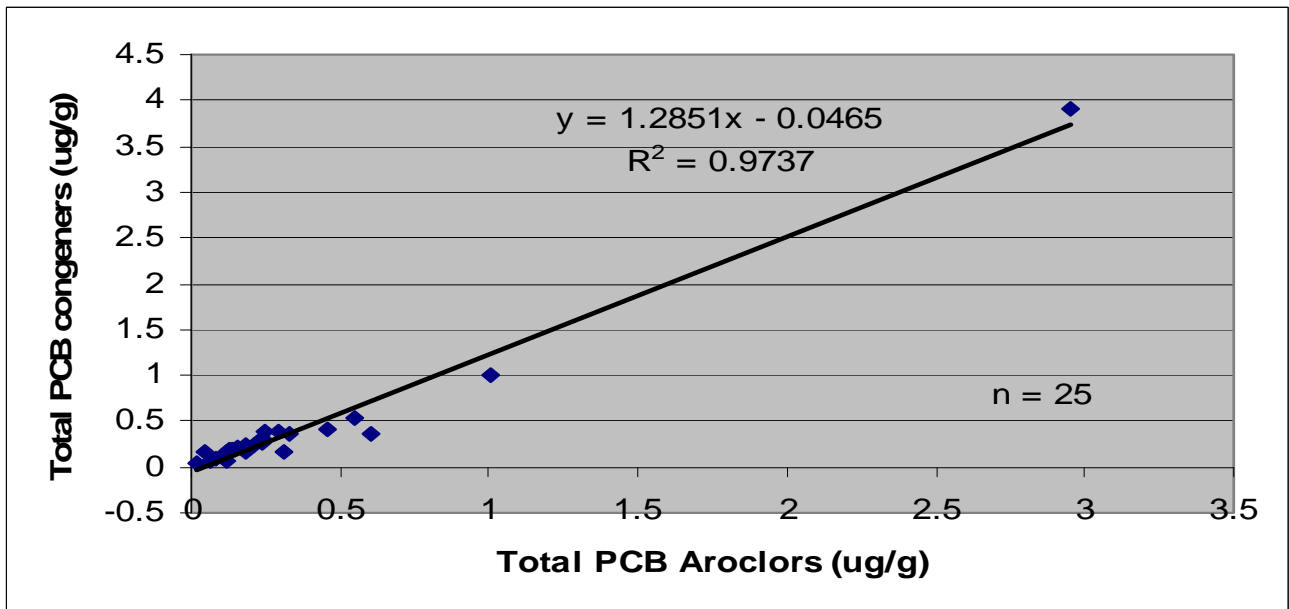


Figure 13: Temporal changes in lipid and PCB concentrations in striped bass from Long Island Sound

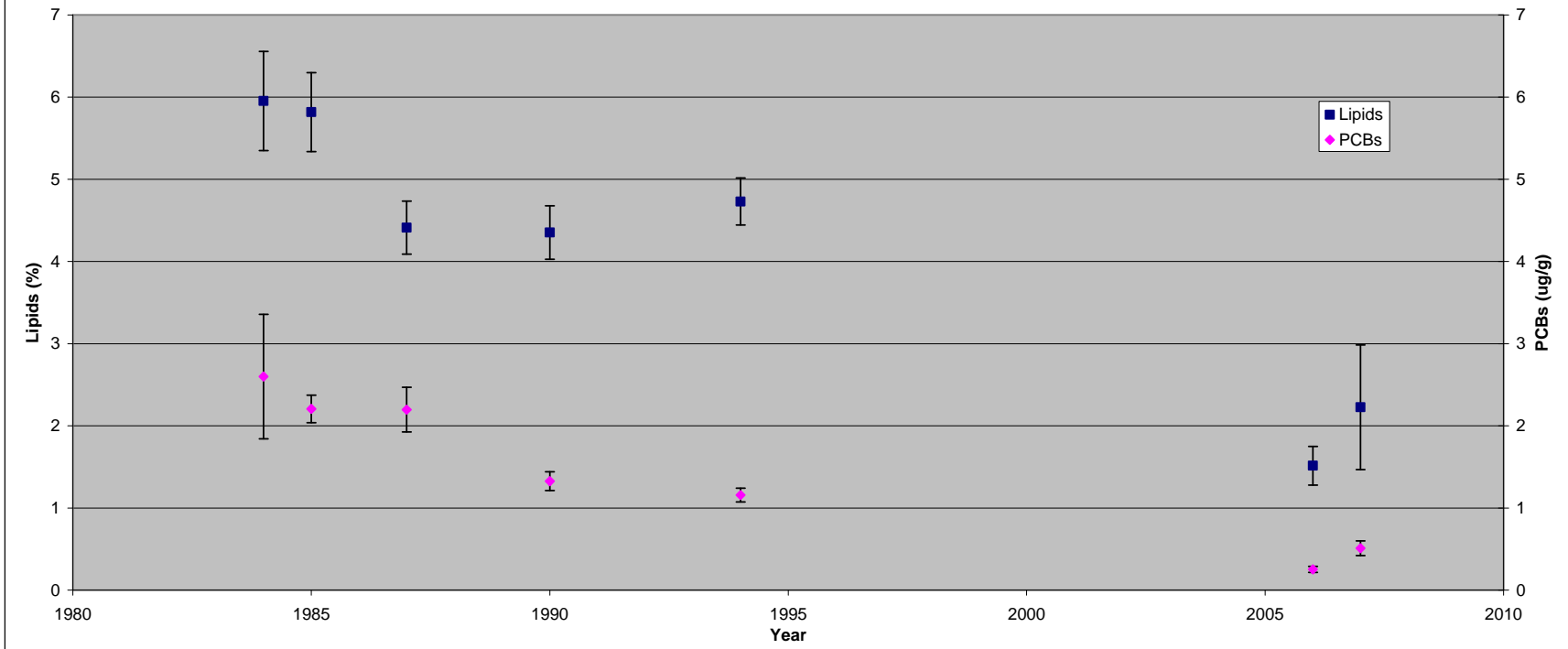
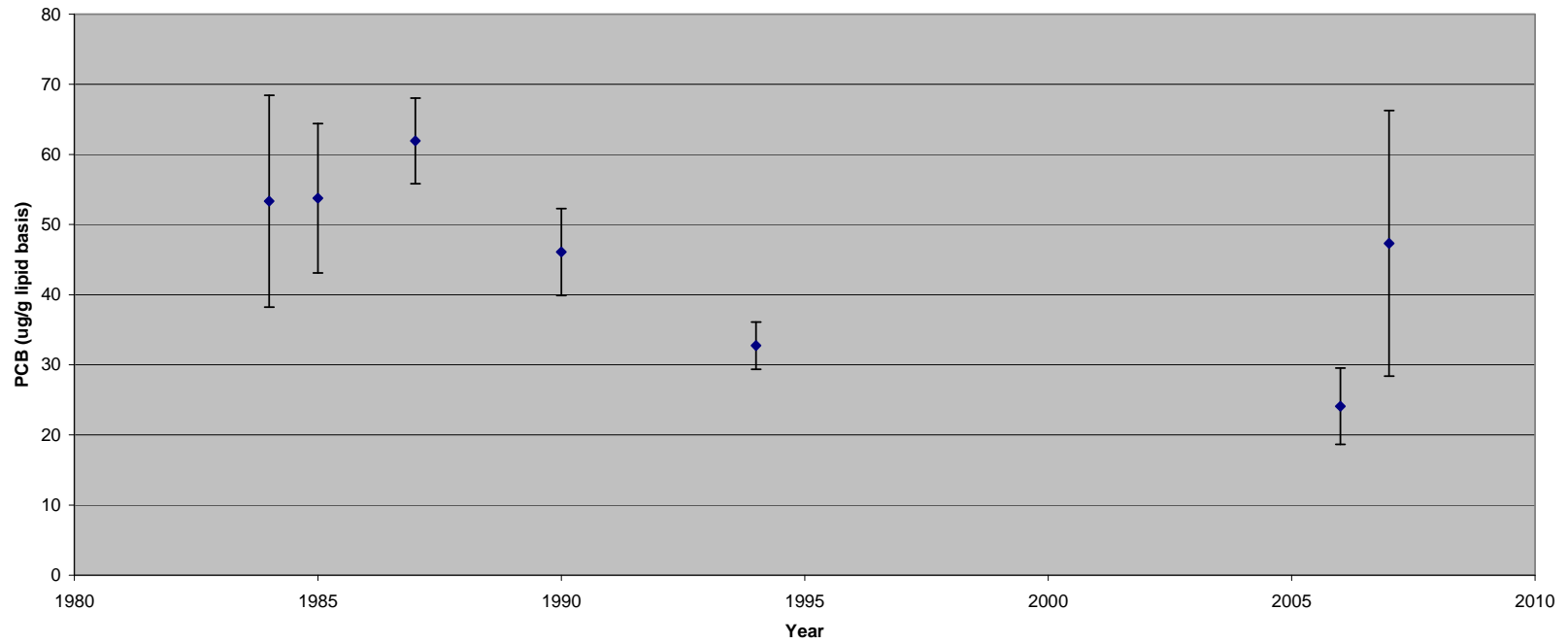
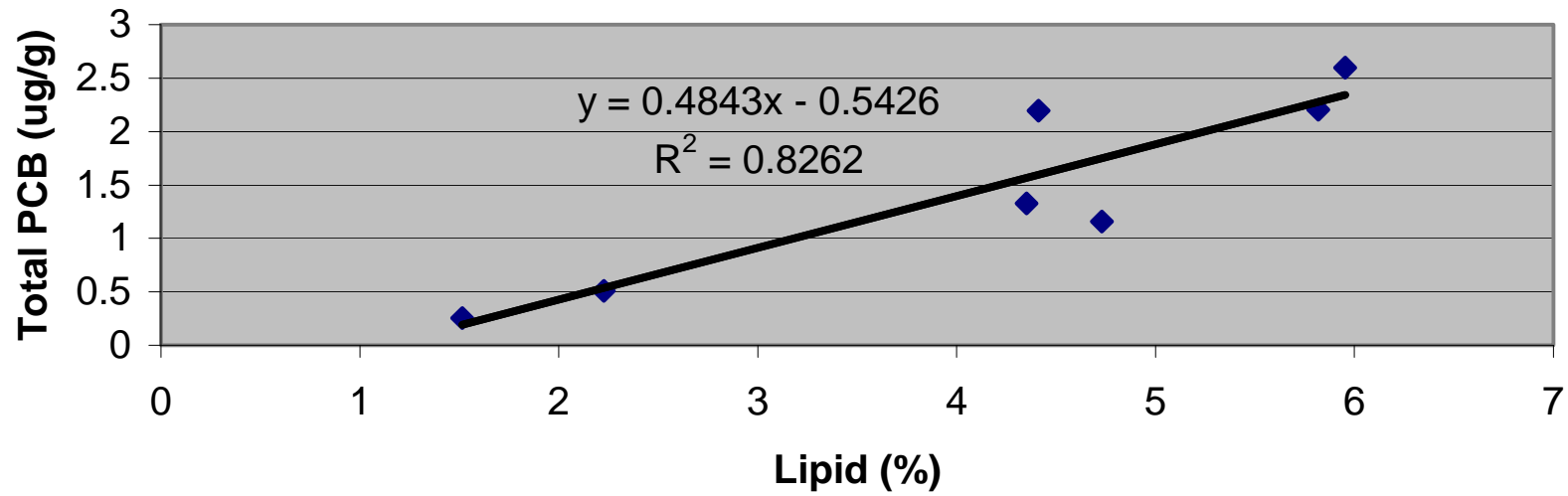


Figure 14: Temporal changes in total PCBs (lipid basis) in striped bass taken from Long Island Sound



**Figure 15: Total PCB-lipid relationship in striped bass from Long Island Sound between 1984 and 2007**



## **APPENDICES**

## Appendix A: MSCL Method NY-4

### Analysis for Organochlorine Pesticides and PCBs in Animal Tissue

Five-gram fish samples or two-gram egg samples are weighed into 250 ml beaker then thoroughly mixed with 150 grams (5 g samples) or 75 grams (2 g samples) of anhydrous sodium sulfate (SOP 1.255). The samples are stored in a desiccator overnight. The samples are then Soxhlet extracted (SOP 1.259) with 600 ml hexane (SOP 1.255) for seven hours. The extract is concentrated by rotary evaporation (SOP 1.129); transferred to a tarred test tube through a Pasteur pipette containing sodium sulfate, and further concentrated to dryness for lipid determination (SOP 1.264).

The weighed lipid sample is dissolved in 4 ml of methylene chloride and the fat removed by injecting 2 ml on a Waters high pressure GPC (Gel Permeation Chromatography) (EPA Method 3640A). The fraction is concentrated by Turbovap and then exchanged into hexane.

The sample is transferred to a 300 ml glass chromatographic column (Kontes # 420280-0242) containing 20 g Florisil (SOP 1.255) topped with 1 cm sodium sulfate and the sample tube rinsed three times with about 2 ml petroleum ether. The column is eluted with 200 ml 6 % diethyl ether (SOP 1.255)/94 % petroleum ether (Fraction I) followed by 200 ml 15 % diethyl ether/85 % petroleum ether (Fraction II). If Endosulfan II and/or Endosulfan Sulfate analysis is required, then 200 ml 50 % diethyl ether/50 % petroleum ether (Fraction III) is required. The diethyl ether used in this analysis contains 2 % ethanol (SOP 1.255). Fractions II and III are concentrated to an appropriate volume for quantification of residues by megabore column electron capture gas chromatography (SOP 1.265) (DB-608 and DB-5 dual columns). Dieldrin and Endrin are in Fraction II, and some delta-BHC. Fraction I is concentrated to 5 ml and transferred to a silicic acid (SOP 1.255) chromatographic column (custom columns 1 cm OD x 40 cm with a 100 ml reservoir on top, Ace Glass) for additional cleanup required for separation of PCBs from other organochlorines. Five grams of hot silicic acid is put into the column, which already has a glass wool plug and about 3-mm sodium sulfate in the bottom. The silicic acid is topped with 10-mm sodium sulfate and prewashed with 10 ml hexane. Three fractions are eluted from the silicic acid column. The sample in 5 ml solvent is added to the column and rinsed into the column with 3, 1, 1-ml hexane. Then the sample is eluted with 20 ml petroleum ether (Fraction SAI). Fraction SAI is 150 ml petroleum ether. Fraction SAIII is 20 ml of a mixed solvent consisting of 1 part acetonitrile, 19 parts hexane and 80 parts methylene chloride (SOP 1.255). Each is concentrated to appropriate volume for quantification of residues by megabore column, electron capture gas chromatography. HCB and Mirex are in SAI. PCBs are found in SAI. The rest of the compounds are in SAIII.

GC determinations were run on a Varian 3600 GC with a Varian Star Data System ver 5 and a Varian 8200 Autosampler. All GCs were equipped with dual DB-608 (0.83  $\mu$  film thickness, J & W Scientific # 125-1730) and DB-5 (1.5  $\mu$  film thickness, J & W Scientific # 125-0532) 30 m megabore columns. All compounds were calculated using a three point standard curve forced through the origin using external standards (SOP 1.267).

PCBs were determined by shooting SAI fractions on a Varian 3400 GC with a Varian Star Data System ver 5 and a Varian 8200 Autosampler. This GC is equipped with a 60 m DB-5 0.25 ID capillary column. Another Varian 3400 GC equipped with a 60 m DB-XLB 0.25 ID capillary column is also used as a second system for PCBs.

The compounds were calculated in the following manner. All the Aroclor standards are at 0.5 ng/ $\mu$ l with one  $\mu$ l shot.

Starting with Aroclor 1260, 4 peaks that are unique to this mixture are located. The areas of the standards are summed and the same peaks located in the sample and also summed. Aroclor 1260 is calculated by the following formula to obtain PPM 1260.

$$\frac{(\text{Area sample})(\text{weight of standard shot in ng})}{(\text{Area 1260 standard})(\text{basis shot in mg})}$$

Aroclor 1254 is calculated by locating the major peaks in the mixture that are normally found in samples. The areas of these peaks are summed. Because some of this area comes from Aroclor 1260 and not all from Aroclor 1254, the contribution from Aroclor 1260 has to be subtracted from the total area. Aroclor 1254 is calculated by using the formula:

$$\frac{\{(\text{Area sample}) - [(\text{PPM 1260})(\text{basis})(\text{area from 1260})]/\text{ng 1260 std}\}(\text{wt 1254 std in ng})}{(\text{Area 1254 standard})(\text{basis shot in mg})}$$

Results are in PPM.

Aroclor 1248 and Aroclor 1242 are calculated in a similar fashion, subtracting the contribution from 1254 in the 1248, and the 1248 in the 1242.

Total PCBs are calculated by adding the sum of Aroclor 1242, 1248, 1254 and 1260.  
Basis = (weight of the sample mg/final volume of sample  $\mu$ l)( $\mu$ l of sample shot)

Appendix B, Table A: Quality control data summary for lipid and polychlorinated biphenyl (Aroclor) analyses.

Analyte	Quality Control Measure	Unit	n	Mean ± SD	Min. – Max.
Lipids	Duplicates	RPD	23	6.06 ± 4.22	0.42 – 14.4
PCBs – Aroclor 1242	Blanks	µg/g	23	<0.010	<0.010 - <0.010
	Duplicates	RPD	23	1.10 ± 4.59	0.0 – 21.9
	Matrix spikes	% recovery	23	93.22 ± 7.12	78 – 104
PCBs – Aroclor 1248	Blanks	µg/g	23	<0.010	<0.010 - <0.010
	Duplicates	RPD	23	7.01 ± 23.25	0.0 – 108
			21 <sup>1</sup>	0.894 ± 2.82	0.0 – 9.52
PCBs – Aroclor 1254	Blanks	µg/g	23	<0.010	<0.010 - <0.010
	Duplicates	RPD	23	7.97 ± 5.78	0.0 – 20.0
	Matrix spikes	% recovery	23	95.70 ± 15.27	63 – 115
PCBs – Aroclor 1260	Blanks	µg/g	23	<0.010	<0.010 - <0.010
	Duplicates	RPD	23	7.72 ± 5.41	0.0 – 18.8
	Matrix spikes	% recovery	23	96.65 ± 10.36	71 – 110
Total PCBs	Blanks	µg/g	23	<0.010	<0.010 - <0.010
	Duplicates	RPD	23	7.00 ± 4.14	0.0 – 19.4
	Matrix spikes	% recovery	23	95.20 ± 8.62	76 – 105.7
	Reference material <sup>4</sup>	µg/g	2	1.07	1.05 – 1.09
PCBs - Surrogates	PCB 209 <sup>2</sup>	µg/g	203	0.187 ± 0.020	0.110 – 0.269
	Tetrachloro-m-xylene <sup>3</sup>	µg/g	195	0.0665 ± 0.0130	0.009 – 0.094

<sup>1</sup> Excludes two outlier values.

<sup>2</sup> Surrogate concentration spiked was 0.20 µg/g.

<sup>3</sup> Surrogate concentration spiked was 0.10 µg/g.

<sup>4</sup> Hudson River Reference Material. Reference material total concentration is 0.948 ± 0.253 µg/g. Source: Sloan *et al.*, 2007.

Appendix B, Table B-1: Quality control data summary for polychlorinated biphenyl congener analyses.

Quality control measure	PCB congener (IUPAC No.)	n	Units	Mean ± SD	Min. – Max.
Blanks	All except 11	2	pg/g	All less than their respective reporting limit	
	11	2	pg/g		61.6 - 141
Surrogates - radiolabeled PCBs	1	29	% recovery	28.66 ± 12.95	
	3	29	% recovery	47.79 ± 9.36	28 – 70
	4	29	% recovery	35.52 ± 10.28	11 – 55
	15	29	% recovery	69.07 ± 9.57	42 – 86
	19	29	% recovery	52.90 ± 8.85	33 – 72
	37	29	% recovery	79.69 ± 9.80	52 – 100
	54	29	% recovery	63.14 ± 6.50	41 – 72
	77	29	% recovery	81.76 ± 9.33	54 – 100
	81	29	% recovery	82.45 ± 10.06	54 – 101
	104	29	% recovery	58.93 ± 5.53	41 – 66
	105	29	% recovery	86.41 ± 9.60	58 – 102
	114	29	% recovery	83.28 ± 9.09	56 – 99
	118	29	% recovery	86.00 ± 9.63	58 – 101
	123	29	% recovery	86.66 ± 9.39	58 – 102
	126	29	% recovery	83.76 ± 9.47	57 – 99
	155	29	% recovery	58.93 ± 6.26	43 – 70
	156/157	29	% recovery	67.69 ± 8.52	50 – 87
	167	29	% recovery	73.72 ± 81.9	54 – 89
	169	29	% recovery	37.24 ± 14.75	19 – 85
	188	29	% recovery	181.00 ± 62.55	79 – 288
189	29	% recovery	100.72 ± 12.19	74 – 124	
202	29	% recovery	180.24 ± 57.26	81 – 290	
205	29	% recovery	83.17 ± 7.03	60 – 91	
206	29	% recovery	110.52 ± 16.55	70 – 133	
208	29	% recovery	109.14 ± 13.53	74 – 138	
209	29	% recovery	111.66 ± 25.46	60 - 158	
Cleanup standards – radiolabeled PCBs	18	29	% recovery	78.21 ± 10.46	50 – 103
	111	29	% recovery	83.38 ± 7.62	55 – 95
	118	29	% recovery	74.86 ± 7.14	52 – 84
Lab control spikes - native PCBs	1	2	% recovery	107	103 – 111
	3	2	% recovery	106.5	105 – 108
	4	2	% recovery	116.5	113 – 120
	15	2	% recovery	105.5	105 – 106
	19	2	% recovery	111	109 – 113
	37	2	% recovery	104	102 – 106
	54	2	% recovery	102.5	102 – 103
	77	2	% recovery	100	99 – 101
	81	2	% recovery	99	98 – 100
	104	2	% recovery	108.5	108 – 109
	105	2	% recovery	101.5	98 – 105
114	2	% recovery	99	98 – 100	

	118	2	% recovery	110	107 – 113
	123	2	% recovery	99	97 – 101
	126	2	% recovery	96.5	95 – 98
	155	2	% recovery	110	110 – 110
	156/157	2	% recovery	99.5	98 – 101
	167	2	% recovery	108	108 – 108
	169	2	% recovery	97.5	96 – 99
	188	2	% recovery	108.5	108 – 109
	189	2	% recovery	94	94 – 94
	202	2	% recovery	108	108 – 108
	205	2	% recovery	102	101 – 103
	206	2	% recovery	104	103 – 105
	208	2	% recovery	104.5	104 – 105
	209	2	% recovery	105	105 – 105
Lab control spikes - radiolabeled PCBs	1	2	% recovery	20	10 – 30
	3	2	% recovery	48	43 – 53
	4	2	% recovery	30.5	25 – 36
	15	2	% recovery	75.5	63 – 88
	19	2	% recovery	53	51 – 55
	37	2	% recovery	91	81 – 101
	54	2	% recovery	66	60 – 72
	77	2	% recovery	93	84 – 102
	81	2	% recovery	92.5	86 – 99
	104	2	% recovery	63	62 – 64
	105	2	% recovery	97	90 – 104
	114	2	% recovery	95	88 – 102
	118	2	% recovery	97	90 – 104
	123	2	% recovery	97.5	90 – 105
	126	2	% recovery	97	90 – 104
	155	2	% recovery	65	62 – 68
	156/157	2	% recovery	88	85 – 91
	167	2	% recovery	89	85 – 93
	169	2	% recovery	84.5	82 – 87
	188	2	% recovery	95.5	89 – 102
189	2	% recovery	100.5	96 – 105	
202	2	% recovery	100.5	97 – 104	
205	2	% recovery	85.5	85 – 86	
206	2	% recovery	85.5	81 – 90	
208	2	% recovery	104	98 – 110	
209	2	% recovery	72	65 – 79	
Matrix spikes - native PCBs	1	4	% recovery	197.5 ± 181.7	103 – 470
	3	4	% recovery	115.5 ± 10.25	106 – 130
	4	4	% recovery	133.8 ± 24.85	120 – 171
	15	4	% recovery	108.3 ± 2.22	105 – 110
	19	4	% recovery	120.3 ± 4.79	115 – 126
	37	4	% recovery	118.0 ± 12.75	103 – 230
	54	4	% recovery	105.5 ± 1.29	104 – 107
	77	4	% recovery	195.5 ± 11.27	119 – 275
	81	4	% recovery	109.8 ± 87.28	100 – 120

	104	4	% recovery	112.0 ± 4.24	110 – 116
	105	4	% recovery	1925 ± 1804	349 – 3572
	114	4	% recovery	199.8 ± 97.89	114 – 287
	118	4	% recovery	8027 ± 7829	1175 – 15156
	123	4	% recovery	230.3 ± 154.7	109 – 434
	126	4	% recovery	143.5 ± 38.79	108 – 180
	155	4	% recovery	212.5 ± 86.95	137 – 297
	156/157	4	% recovery	508.3 ± 403.5	155 – 874
	167	4	% recovery	625.3 ± 497.5	190 – 1066
	169	4	% recovery	124.3 ± 22.85	103 – 145
	188	4	% recovery	204.3 ± 76.63	137 – 276
	189	4	% recovery	164.5 ± 65.26	108 – 223
	202	4	% recovery	1508 ± 1252	405 – 2666
	205	4	% recovery	140.0 ± 40.62	105 – 180
	206	4	% recovery	1665 ± 1463	384 – 3095
	208	4	% recovery	1093 ± 907.0	293 – 1931
	209	4	% recovery	1400 ± 1242	314 – 2497

- continued on next page -

Quality control measure	PCB congener (IUPAC No.)	n	Units	Original values		Background subtracted	
				Mean $\pm$ SD	Min. – Max.	Mean $\pm$ SD	Min. – Max.
Matrix spikes - native PCBs	1	2	RPD	64.5	4.5 – 124.5	71.0	4.5 – 137.5
	3	2	RPD	9.25	5.4 – 13.1	9.6	5.5 – 13.7
	4	2	RPD	17.9	2.0 – 33.8	20.1	2.0 – 38.2
	15	2	RPD	2.70	1.4 – 4.0	2.8	1.5 – 4.1
	19	2	RPD	2.40	2.0 – 2.8	2.9	2.2 – 3.6
	37	2	RPD	1.15	1.1 – 1.2	1.0	0.8 – 1.2
	54	2	RPD	1.85	0.30 – 3.4	1.9	0.3 – 3.5
	77	2	RPD	0.40	0.40 – 0.40	0.35	0.2 – 0.5
	81	2	RPD	3.95	2.7 – 5.2	7.35	6.3 – 8.4
	104	2	RPD	1.80	0.10 – 3.5	1.8	0.1 – 3.5
	105	2	RPD	6.65	4.9 – 8.4	76.2	42.7 – 109.7
	114	2	RPD	1.70	1.6 – 1.8	3.4	2.0 – 4.8
	118	2	RPD	8.50	4.8 – 12.2	23.1	0.0 – 46.2
	123	2	RPD	24.65	1.7 – 47.6	51.25	2.7 – 99.8
	126	2	RPD	3.25	3.2 – 3.3	22.75	3.4 – 42.1
	155	2	RPD	3.50	0.30 – 6.7	8.45	0.7 – 16.2
	156/157	2	RPD	4.30	3.8 – 4.8	15.8	8.4 – 23.2
	167	2	RPD	3.15	2.0 – 4.3	12.05	8.1 – 16
	169	2	RPD	2.20	1.3 – 3.1	2.2	1.3 – 3.1
	188	2	RPD	2.90	1.6 – 4.2	6.5	1.8 – 11.2
189	2	RPD	1.35	0.60 – 2.1	3.75	0.9 – 6.6	
202	2	RPD	7.45	5.8 – 9.1	126.65	51.3 – 200	
205	2	RPD	3.00	0.40 – 5.6	5.55	0.5 – 10.6	
206	2	RPD	10.75	9.9 – 11.6	79.05	0.0 – 158.1	
208	2	RPD	7.75	5.7 – 9.8	69.4	49.3 – 89.5	
209	2	RPD	4.00	1.7 – 6.3	39.7	23 – 56.4	

Appendix B, Table B-2: Reference material analyses for PCB congeners.

PCB congener (IUPAC number)	Concentration (ng/g wet weight) <sup>1</sup>		Contract lab concentration (ng/g wet weight)
	Overall mean	Uncertainty	
8	1.72	0.11	0.752
18	11.77	0.82	
18/30			9.11
20/28			18.35
28	15.8	2.6	
31	17.2	2.7	17.75
44	25.0	1.3	
44/47/65			57.75
45	5.34	0.3	
45/51			8.215
49	54.0	2.2	
49/69			52.9
52	58.3	3.9	66.2
56	12.91	0.47	7.29
63	3.97	0.25	4.525
66	26.9	2.8	
66/70/74/76			23.8
70	18.69	0.83	
74	16.2	0.77	
82	5.03	0.27	3.4
86/87/97/108/119/125			25.95
87	13.1	1.3	
90/101/113			45.4
92	12.46	0.46	16.6
95	30.8	1.3	30.95
99	29.1	2.2	42.15
101	45.4	3.2	
105	8.5	1.4	9.675
107	4.27	0.32	
107/124			1.12
110	35.28	2.4	
110/115			44.4
118	29.1	2.4	32.5
128	7.27	0.29	
128/166			7.99
129/138/163			74.05
132	11.92	0.25	
135/151			25.65
138	29.8	1.2	
146	13.0	1.3	18.6
147/149			42.6
149	28.7	2.4	
151	9.8	1.1	
153	57.5	1.4	

153/132	58.9	4.6	
153/168			75.15
154	11.76	0.68	3.005
156	3.28	0.52	
156/157			4.48
157	0.88	0.15	
158	3.87	0.34	5.155
163	15.1	1.1	
170	7.25	0.57	5.405
174	4.76	0.31	6.75
180	18.79	0.82	
180/193	21.12	0.54	14.25
183	6.68	0.48	
183/185			11.05
187	19.1	1.1	32.0
193	0.901	0.057	
194	3.327	0.082	1.505
195	1.35	0.079	1.045
201	<1		1.205
206	3.37	0.21	3.835
209	1.50	0.15	1.56

<sup>1</sup> Values for Hudson River Reference Material from Schantz *et al.* 2004.

Appendix B, Table C: Quality control data summary mercury and cadmium analyses.

Analyte	Quality Control Measure	Unit	n	Mean ± SD	Min. – Max.
Mercury	Blanks	µg/g	18 <sup>1</sup>	<0.0005 <sup>1</sup>	<0.0005 – 0.0006
	Duplicates	RPD	27	4.57 ± 3.66	0.0 – 12.8
	Matrix spikes	% recovery	24	98.34 ± 6.72	87.5 – 113.7
	Matrix spike duplicates	% recovery	24	100.1 ± 5.09	89.4 – 109.5
	Reference materials	% recovery	9	96.41 ± 2.40	92.8 – 100.8
Cadmium	Blanks	µg/g	6	<0.005	<0.005 - <0.005
	Duplicates	RPD	4	1.00 ± 0.75	0.2 – 1.9
	Matrix spikes	% recovery	4	83.00 ± 3.96	78.2 – 86.9
	Matrix spike duplicates	% recovery	4	89.15 ± 2.83	85.2 – 91.4
	Reference materials	% recovery	2	91.05	85.2 – 96.9

<sup>1</sup> Fourteen of 18 blanks were non-detect (<0.0005 µg/g), three were at the method detection limit (0.0005 µg/g), and one at 0.0006 µg/g. All blanks were less than 1.0 percent of fish sample concentrations.

Appendix B, Table D: Quality control data summaries for analysis of chlorinated dibenzo-*p*-dioxins and dibenzofurans.

Quality Control Measure	Analyte	Unit	n	Largest reporting limit	Blanks < reporting limits (%)	Min. – Max.
Blanks	2,3,7,8-TCDD	pg/g	7	0.40	100	<0.13 – <0.40
	1,2,3,7,8-PeCDD	pg/g	7	0.22	100	<0.076 - <0.22
	1,2,3,4,7,8-HxCDD	pg/g	7	0.17	57	<0.067 – 0.31
	1,2,3,6,7,8-HxCDD	pg/g	7	0.18	71	<0.069 – 0.28
	1,2,3,7,8,9-HxCDD	pg/g	7	0.28	85	<0.07 – 0.14, <0.28
	1,2,3,4,6,7,8-HpCDD	pg/g	7	0.25	42	<0.11 – 0.39
	OCDD	pg/g	7	0.83	14	<0.13 – 3.0
	2,3,7,8-TCDF	pg/g	7	0.23	71	<0.11 – 0.56
	1,2,3,7,8-PeCDF	pg/g	7	0.20	57	<0.094 – 0.70
	2,3,4,7,8-PeCDF	pg/g	7	0.23	100	<0.066 - <0.23
	1,2,3,4,7,8-HxCDF	pg/g	7	0.22	57	<0.066 – 0.39
	1,2,3,6,7,8-HxCDF	pg/g	7	0.19	57	0.075, <0.079 – 0.28
	2,3,4,6,7,8-HxCDF	pg/g	7	0.11	57	<0.071 – 0.32
	1,2,3,7,8,9-HxCDF	pg/g	7	0.20	57	<0.065 – 0.34
	1,2,3,4,6,7,8-HpCDF	pg/g	7	0.21	71	<0.072 – 0.43
	1,2,3,4,7,8,9-HxCDF	pg/g	7	0.29	57	0.14 – 0.35
	OCDF	pg/g	7	0.54	28	<0.14 – 1.5
	∑TCDD	pg/g	7	0.20	85	<0.16 – 0.92
	∑PeCDD	pg/g	7	0.20	71	<0.16 – 0.27
	∑HxCDD	pg/g	7	0.21	42	<0.069 – 0.89
	∑HpCDD	pg/g	7	0.25	14	<0.11 – 1.0
	∑TCDF	pg/g	7	0.17	57	<0.11 – 1.5
	∑PeCDF	pg/g	7	0.20	42	<0.08 – 1.4
	∑HxCDF	pg/g	7	0.17	28	<0.16, 0.075 – 2.3
	∑HpCDF	pg/g	7	0.25	28	<0.25, 0.14 – 0.78

Quality Control Measure	Analyte	Unit	N	Mean ± SD	Min. – Max.
Internal standards	2,3,7,8-TCDD-13C	% recovery	74	65.28 ± 12.76	23 - 88
	1,2,3,7,8-PeCDD-13C	% recovery	74	83.07 ± 16.62	53 - 125
	1,2,3,4,7,8-HxCDD-13C	% recovery	74	73.61 ± 10.79	53 - 107
	1,2,3,6,7,8-HxCDD-13C	% recovery	74	70.92 ± 9.99	50 - 96
	1,2,3,4,6,7,8-HpCDD-13C	% recovery	74	71.07 ± 12.12	48 - 111
	OCDD-13C	% recovery	74	58.68 ± 15.23	31 - 117
	2,3,7,8-TCDF-13C	% recovery	74	61.38 ± 12.53	22 - 88
	1,2,3,7,8-PeCDF-13C	% recovery	74	66.51 ± 12.77	39 - 94
	2,3,4,7,8-PeCDF-13C	% recovery	74	68.72 ± 13.03	44 - 97
	1,2,3,4,7,8-HxCDF-13C	% recovery	74	67.43 ± 9.32	48 - 108
	1,2,3,6,7,8-HxCDF-13C	% recovery	74	63.43 ± 8.69	45 - 95
	2,3,4,6,7,8-HxCDF-13C	% recovery	74	62.41 ± 9.09	42 - 85
	1,2,3,7,8,9-HxCDF-13C	% recovery	74	67.64 ± 11.71	45 - 97
	1,2,3,4,6,7,8-HpCDF-13C	% recovery	74	61.80 ± 9.58	42 - 90
	1,2,3,4,7,8,9-HpCDF-13C	% recovery	74	56.15 ± 10.73	34 - 93
	2,3,7,8-TCDD-37Cl <sub>4</sub>	% recovery	74	71.96 ± 14.50	27 - 106
	Lab control spikes - unlabeled	2,3,7,8-TCDD	% recovery	6	93.17 ± 5.67
1,2,3,7,8-PeCDD		% recovery	6	87.33 ± 4.80	82 - 94
1,2,3,4,7,8-HxCDD		% recovery	6	97.67 ± 3.93	92 - 102
1,2,3,6,7,8-HxCDD		% recovery	6	94.50 ± 8.98	81 - 105
1,2,3,7,8,9-HxCDD		% recovery	6	89.17 ± 8.95	75 - 99
1,2,3,4,6,7,8-HpCDD		% recovery	6	90.50 ± 4.64	82 - 95
OCDD		% recovery	6	102.3 ± 7.06	94 - 110
2,3,7,8-TCDF		% recovery	6	89.67 ± 3.98	85 - 95
1,2,3,7,8-PeCDF		% recovery	6	99.33 ± 7.03	93 - 110
2,3,4,7,8-PeCDF		% recovery	6	94.50 ± 7.18	86 - 103
1,2,3,4,7,8-HxCDF		% recovery	6	94.00 ± 7.29	84 - 103
1,2,3,6,7,8-HxCDF		% recovery	6	97.33 ± 6.02	88 - 106
2,3,4,6,7,8-HxCDF		% recovery	6	97.83 ± 5.94	92 - 106
1,2,3,7,8,9-HxCDF		% recovery	6	95.17 ± 7.99	86 - 105
1,2,3,4,6,7,8-HpCDF		% recovery	6	97.17 ± 6.79	88 - 106
1,2,3,4,7,8,9-HxCDF		% recovery	6	105.8 ± 3.76	101 - 110
OCDF		% recovery	6	100.2 ± 8.18	88 - 111
Lab control spikes - radio-labeled	2,3,7,8-TCDD-13C	% recovery	6	62.83 ± 23.42	31 - 90
	1,2,3,7,8-PeCDD-13C	% recovery	6	82.50 ± 18.32	62 - 105
	1,2,3,4,7,8-HxCDD-13C	% recovery	6	88.50 ± 8.98	79 - 97
	1,2,3,6,7,8-HxCDD-13C	% recovery	6	85.17 ± 11.34	73 - 100
	1,2,3,4,6,7,8-HpCDD-13C	% recovery	6	83.33 ± 10.21	73 - 100
	OCDD-13C	% recovery	6	75.83 ± 14.01	53 - 88
	2,3,7,8-TCDF-13C	% recovery	6	60.50 ± 23.11	30 - 89
	1,2,3,7,8-PeCDF-13C	% recovery	6	68.00 ± 17.57	47 - 92
	2,3,4,7,8-PeCDF-13C	% recovery	6	72.50 ± 15.11	57 - 92
	1,2,3,4,7,8-HxCDF-13C	% recovery	6	71.33 ± 9.61	55 - 83
	1,2,3,6,7,8-HxCDF-13C	% recovery	6	71.83 ± 11.67	56 - 87
	2,3,4,6,7,8-HxCDF-13C	% recovery	6	74.17 ± 10.11	65 - 91
	1,2,3,7,8,9-HxCDF-13C	% recovery	6	86.00 ± 12.35	70 - 99
	1,2,3,4,6,7,8-HpCDF-13C	% recovery	6	77.50 ± 9.14	63 - 87
	1,2,3,4,7,8,9-HxCDF-13C	% recovery	6	67.50 ± 7.56	55 - 76

	2,3,7,8-TCDD-37Cl <sub>4</sub>	% recovery	6	68.67 ± 25.06	37 - 104
Spiked recovery samples	2,3,7,8-TCDD	[RPD]	2	7.3	4.3 – 10.3
	1,2,3,7,8-PeCDD	[RPD]	2	1.2	1.2 – 1.2
	1,2,3,4,7,8-HxCDD	[RPD]	2	3.1	2.2 – 4.0
	1,2,3,6,7,8-HxCDD	[RPD]	2	9.55	1.1 – 18
	1,2,3,7,8,9-HxCDD	[RPD]	2	13.7	8.1 – 19.3
	1,2,3,4,6,7,8-HpCDD	[RPD]	2	5.75	2.2 – 9.3
	OCDD	[RPD]	2	6.9	3.1 – 10.7
	2,3,7,8-TCDF	[RPD]	2	1.7	1.1 – 2.3
	1,2,3,7,8-PeCDF	[RPD]	2	2.1	1.1 – 3.1
	2,3,4,7,8-PeCDF	[RPD]	2	4.95	4.5 – 5.4
	1,2,3,4,7,8-HxCDF	[RPD]	2	4.0	1.1 – 6.9
	1,2,3,6,7,8-HxCDF	[RPD]	2	5.9	2.1 – 9.7
	2,3,4,6,7,8-HxCDF	[RPD]	2	3.7	1.1 – 6.3
	1,2,3,7,8,9-HxCDF	[RPD]	2	5.0	2.2 – 7.8
	1,2,3,4,6,7,8-HpCDF	[RPD]	2	5.9	2.1 – 9.7
	1,2,3,4,7,8,9-HxCDF	[RPD]	2	2.35	1.0 – 3.7
	OCDF	[RPD]	2	10.3	4.9 – 15.7

Quality Control Measure	Analyte	Unit	n	Original concentrations		Background subtracted	
				Mean	Min. – Max.	Mean	Min. – Max.
Spiked matrix samples	2,3,7,8-TCDD	[RPD]	2	7.75	7.7 – 7.8	8.25	8.0 – 8.5
	1,2,3,7,8-PeCDD	[RPD]	2	6.35	5.1 – 7.6	6.65	5.5 – 7.8
	1,2,3,4,7,8-HxCDD	[RPD]	2	10.3	10.1 – 10.5	10.5	10.4 – 10.6
	1,2,3,6,7,8-HxCDD	[RPD]	2	4.95	0.6 – 9.3	5.0	0.3 – 9.7
	1,2,3,7,8,9-HxCDD	[RPD]	2	7.4	5.4 – 9.4	7.65	5.7 – 9.6
	1,2,3,4,6,7,8-HpCDD	[RPD]	2	5.25	4.3 – 6.2	5.9	5.2 – 6.6
	OCDD	[RPD]	2	6.05	4.0 – 8.1	6.4	4.4 – 8.4
	2,3,7,8-TCDF	[RPD]	2	4.2	1.9 – 6.5	9.8	5.6 – 14.0
	1,2,3,7,8-PeCDF	[RPD]	2	5.2	3.0 – 7.4	5.85	3.9 – 7.8
	2,3,4,7,8-PeCDF	[RPD]	2	6.6	3.9 – 9.3	7.7	5.3 – 10.1
	1,2,3,4,7,8-HxCDF	[RPD]	2	6.05	4.7 – 7.4	6.2	4.8 – 7.6
	1,2,3,6,7,8-HxCDF	[RPD]	2	7.15	5.5 – 8.8	7.5	6.0 – 9.0
	2,3,4,6,7,8-HxCDF	[RPD]	2	7.55	5.3 – 9.8	7.75	5.5 – 10.0
	1,2,3,7,8,9-HxCDF	[RPD]	2	8.0	4.2 – 11.8	8.0	4.2 – 11.8
	1,2,3,4,6,7,8-HpCDF	[RPD]	2	6.55	5.8 – 7.3	6.6	5.9 – 7.3
	1,2,3,4,7,8,9-HxCDF	[RPD]	2	5.25	3.1 – 7.4	5.25	3.1 – 7.4
	OCDF	[RPD]	2	6.85	6.6 – 7.1	6.85	6.6 – 7.1

