Size and Seasonal Variations of PCBs in Chinook Salmon *(Oncorhynchus tshawytscha)* Fillets from Lake Michigan near Ludington, Michigan, U.S.

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TABLE OF CONTENTS

EXECUTIVE SUMMARY

Eighty-one chinook salmon in three size classes were collected from the Ludington area of Lake Michigan over the course of the fishing season. A single fillet was removed from each fish, skinned, and trimmed. The entire trimmed fillet was homogenized and a sample of the homogenate was analyzed for total PCB concentration and lipid content.

Total concentrations of PCBs ranged from 0.14 to 2.1 ug/g (parts per million, ppm), wet weight, in the trimmed fillets. Only one fillet of the 81 sampled exceeded the current FDA action limit of 2.0 ppm for total PCBs. The mean of concentrations of total PCBs for the 81 fish was 0.94 ug/g with a standard deviation of 0.43.

Concentrations of total PCB in trimmed fillets did not vary significantly among samples collected in May, July and September when concentrations were normalized for the variation in fish size.

Concentrations of total PCB varied significantly among the three size classes: Less than 24 in., between 24 and 32 in., and greater than 32 in. Mean total PCB concentrations for the small, medium and large fish were 0.50 ug/g, 1.00 ug/g and 1.21 ug/g, respectively. Regression of total PCB concentration in fillet against length and weight of the whole fish explained 48% of the variability in PCB concentration. The range of expected concentrations of total PCB for a fish of a known size was 1.3

ppm based on the 95% confidence intervals for individual observations in this regression.

Lipid content of the trimmed fillets was not correlated with fish length or with PCB content of the fillets. PCB concentrations on a lipid basis ranged from 5.3 to 595 ug/g lipid. Lipid-based PCB concentrations could not be predicted from fish length.

INTRODUCTION

Polychlorinated biphenyls (PCBs) are a group of chlorinated hydrocarbons developed for commercial use as electrical transformer insulation fluids, extreme pressure oils and greases, hydraulic fluids, fire retardants and plasticizers [1]. PCBs are extremely stable and inert compounds and, as a result, have accumulated to significant levels in fishes collected from most aquatic environments, including the Great Lakes [2]. Allowable concentrations of these compounds in the flesh of fishes has been set at 2 mg/kg (ppm) by the Food and Drug Administration because of the potential toxicity and carcinogenecity of PCBs [3]. In the early 1970s, numerous studies recorded concentrations of PCBs in Great Lakes salmonids far in excess of this guideline [4]. Strict regulatory controls on the use of PCBs by industry were instituted in the early 1970s. In recent years PCB concentrations in many Great Lakes salmonids have declined to the point where concentrations of PCBs in most fishes are below the federal guideline of 2 ppm [5]. However, certain fishes, most notably lake trout, brown trout and large chinook salmon, continue to exhibit unacceptably great concentrations of PCBs [6].

Studies by the U.S. Fish and Wildlife Service and the Wisconsin Department of Natural Resources have shown significant spatial variation in concentrations of PCBs in salmonid fishes collected from different areas of Lake Michigan [7,8]. Therefore, residue concentrations in fishes

collected from specific locations in Lake Michigan quite probably are not representative of those present in fishes from other areas of the lake. It is impossible for regulatory agencies to sample adequate numbers of fishes for PCB analysis from each site of interest on Lake Michigan.

Several studies have noted that PCB concentrations show a positive correlation with size (or age) of several Great Lakes salmonid species [8,9]. Thus, various fish consumption advisories have been formulated based on fish size. In the past, these advisories have beeen issued for Lake Michigan lake trout and chinook salmon. The fish lengths recommended in these advisories can vary from year to year [9] as the results of monitoring surveys change. Also, because there appear to be site-specific differences in concentrations of PCBs Lake Michigan salmonids, it is quite reasonable to assume that residue/length relationships will vary from location to location.

Significant variations in concentrations of PCBs in Lake Michigan chinook salmon from season to season have been previously reported [8]. These variations could have been related to seasonal changes in diet and growth, or they may be associated with maturation [10,11]. It is not known if these variations are consistent throughout Lake Michigan salmon.

In Michigan and other states consumption advisories are supplemented with recommendations for preparation of fillets which include removing skin, belly and dorsal fat, and the

lateral line [12,13,14]. State surveys and national monitoring studies have traditionally used skin-on fillets in their analyses protocols. Trimming of fillets has been shown to reduce contaminant burdens [15].

This study was designed to determine concentrations of PCBs in trimmed, skin-off fillets of chinook salmon caught at a single locality. Additionally, the experiment was designed to evaluate variations in PCB concentration due to the size of the salmon and season in which they were caught.

EXPERIMENTAL DESIGN

Salmon from each of three size classes were collected during each of three collection periods (Table 1). Eighty-one fish were collected in all. The three size classes were the following: Less than 24 in., between 24 in. and 32 in., and greater than 32 in. The three collection periods were 5/7/88-5/18/88, 7/15/88-7/17/88, and 9/17/88-9/27/88. All fish were collected from Lake Michigan within a 5 mile radius of Ludington, MI, with the exception of 17 fish in the September collection period which were taken from the Michigan Department of Natural Resources (MDNR) weir on the Little Manistee River, which is approximately 30 mi. north of Ludington.

Table 1. Numbers of chinook salmon collected in each size class during each season. The number of each sex of fish making up the totals are given in parentheses.

Fish Collected

# Fish (F,M)	May	July	September	Totals:
< 24"	3 (1,2)	9 (4,5)	9 (1,8)	21 (6,15)
24" - 32"	16 (7,9)	9 (2,7)	9 (2,7)	34 (11,23)
> 32"	8 (5,3)	9 (7,2)	9 (5,4)	26 (17,9)
Totals:	27 (13,14)	27 (13,14)	27 (8,19)	81 (34,47)

METHODS

Collection

All fish were caught on standard tackle from charter boats except for those collected from the Little Manistee River. Fish taken at the weir were judged to be recent arrivals by their general condition. All fish were kept on ice until processing. Total fish lengths were measured and weights were rounded to the nearest quarter pound. Charterboat captains or a Sea Grant extension agent filleted one side of each fish. Skin, belly and dorsal fat, and lateral line were removed. Fillets were rinsed with water. Each trimmed fillet was wrapped in aluminum foil and identified with tag. The remainder of the fish was labelled with a spaghetti tag and packaged in a labelled plastic bag. Fish carcasses and fillets were transported on ice to the Pesticide Research Center at Michigan State University where they were placed in freezers at -10 F.

Sample Preparation

Foil-wrapped fillets were partially thawed before being homogenized in a Hobart (Model 8181, Troy, Ohio) meat grinder. Fillet homogenates were thoroughly mixed and then weighed into clean, solvent-rinsed, wide-mouth, glass sample jars with Teflon-lined lids (Model 03-320-7A, Fisher Scientific). An internal standard, 2.85 ug of 2,4,6-trichlorobiphenyl (2,4,6-T_3CB), was added to 10 g subsamples of fillet homogenate. This PCB congener was selected because it does not occur in commercial Aroclors nor has it been

detected in environmental samples. The samples were then stored at -10 F until analysis.

Archived samples for each fish include a carcass intact except for one fillet, homogenized fillet, and spiked homogenized fillet. Thus, analyses could be performed on skin-on, untrimmed fillet or with a different analysis protocol for comparison to the results of this study.

Extraction

Samples were ground in an Omni-mixer (Sorvall, Inc., Norwalk, Conn.) with four times their weight of anhydrous sodium sulfate (J.T. Baker Chemical Co., Phillipsburg, NJ). The resulting dry powder was extracted with 200 ml of dichloromethane at a flow of 3-5 ml/min in a 2 cm i.d. glass column. The volume of solvent in the extracts was reduced with a Rotovapor 110 (Buchi, Flawil, Switzerland) at ambient temperature and the volume was adjusted to exactly 8.0 ml with 1:1 (v/v) mixture of cyclohexane and dichloromethane.

Aliquants of the extract were pipetted onto tared, aluminum weighing boats for gravimetric lipid determination.

Five ml of the remaining extract was loaded onto an automated gel permeation chromatography (GPC) apparatus (ABC, Inc., Columbia, MO). Interfering compounds including lipids were removed on a GPC column of 60 g of SX-3 Bio-Beads (Bio-Rad Laboratories, Richmond, CA) with a 1:1 cyclohexane and dichoromethane mobile phase at 5 ml/min. A 110 ml fraction was collected after a 150 ml dump cycle. The volume of solvent of the collected fraction was reduced as above and

the volume was adjusted to 1 ml with hexane.

Interfering compounds were removed from the 1 ml extract with a combined acidic silica gel/silica gel column composed of 1 cm of sodium sulfate over 1 g of 40% H_2SO_4/silica gel (w/w), 5 g silica gel and 1 cm sodium sulfate in a 1 cm i.d. reservoir column. Silica gel (70-230 mesh, Sigma Chemical Co., St. Louis, MO) was activated at 130 C for 12 h and cooled to ambient temperature before use. Sample extracts were loaded onto the columns after the columns were rinsed with 20 ml hexane. PCBs were eluted with 50 ml of 0.5% benzene in hexane. The volume of the PCB extract was reduced as above, exchanged with hexane, and brought to exactly 1.00 ml with hexane for quantification.

All solvents used in the analyses were pesticide or HPLC grade obtained from Fisher or Mallinkrodt.

Gas Chromatography

Concentrations of PCBs in extracts were determined with a Perkin Elmer Model 8500 gas chromatograph (GC). The GC was equipped with a ^{63}Ni detector at 320 C, a split/splitless injector at 240 C, and a 30m x 0.25mm i.d. DB-5 column (J&W Scientific). The oven was temperature programmed from 120 C (1 min. hold) to 260 C at 2 C/min. with a final hold of 5 minutes. Helium at 20.0 psig was used as the carrier gas and nitrogen at 55 psig was used as the make-up gas. The split was adjusted to either 10:1 or 20:1 in order to maintain the signal in the linear range of the detector.

Quantitation

Chromatographic peak areas were integrated by the Perkin Elmer 8500. Run reports were transported and translated to ASCII files on an IBM PC compatible computer where they were formatted for input to COMSTAR, a PCB quantitation program [16]. The eighty-four PCB peaks used to quantitate total PCB concentration and Aroclor composition were identified by retention times relative to the internal standard. Retention indices were confirmed by matching all sample chromatograms to a chromatogram of a 1:1:1:1 mixture of Aroclors 1242, 1248, 1254 and 1260 on a light table.

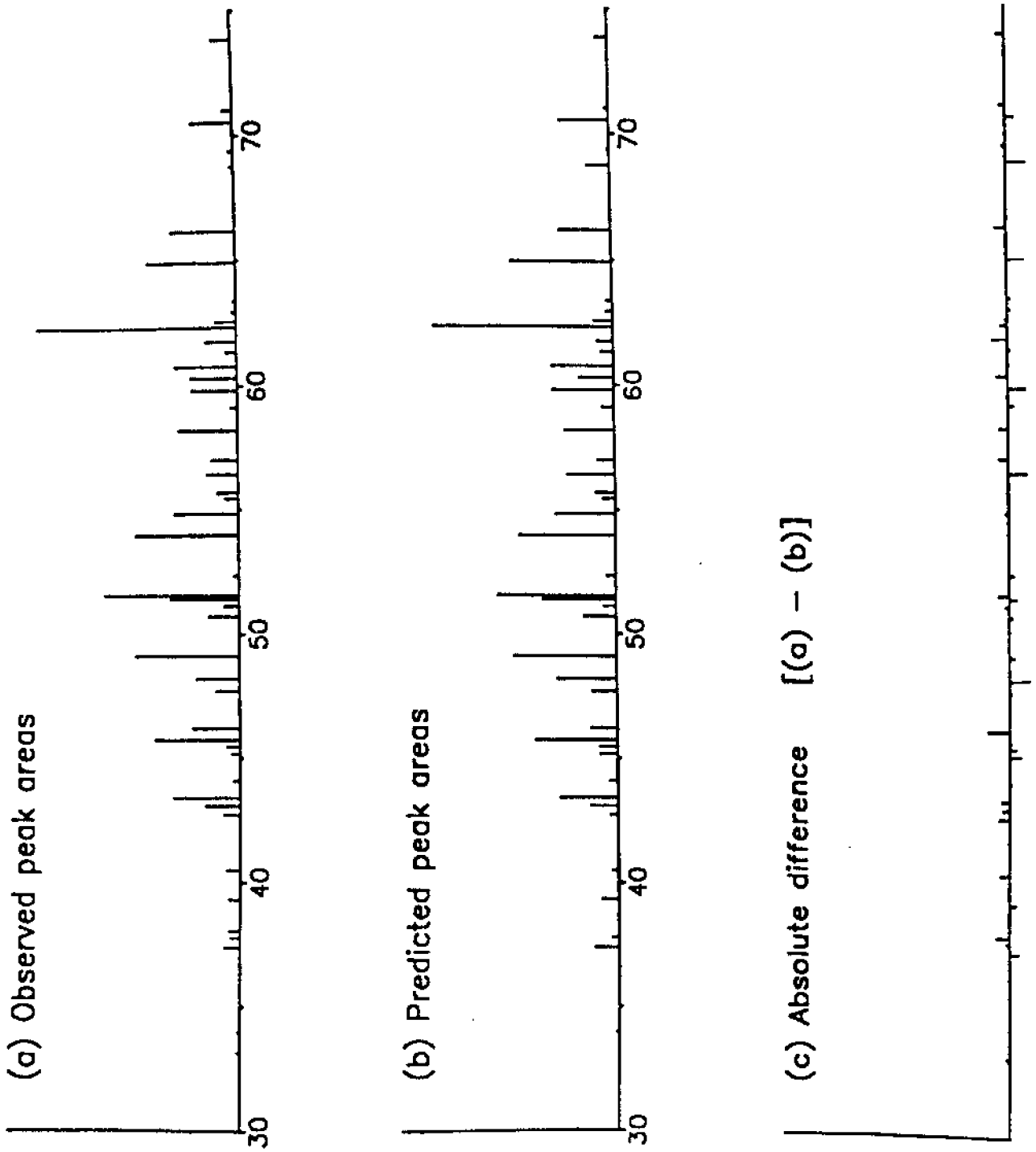
COMSTAR quantitates total PCBs and determines the best combination of commercial Aroclors to represent the congener pattern observed in the samples. The algorithm iteratively regresses sample peak areas on those of Aroclor standards and performs outlier detection and elimination and tests of significance [16]. Outlier peaks, which are identified as weathered or contaminated peaks by COMSTAR, are not used in the quantitation procedure. COMSTAR has previously been shown to perform better than both SIMCA and congener specific PCB analysis in the determination of total PCB and Aroclor composition, except in the presence of toxaphene [16]. Toxaphene was removed from all fillet samples by the acidic silica gel/silica gel column. Further characterization of COMSTAR was performed as a part of this study and is presented in the Quality Assurance section of this report.

The parameters of COMSTAR were optimized for the fillet

samples. Aroclors 1242, 1248, 1254 and 1260 (Monsanto Corp., St. Louis, MO) were used to develop the calibration table for COMSTAR. The program used the Aroclor 1242 pattern to account for early eluting peaks in less than five percent of the samples. When Aroclor 1242 was removed from the calibration table, these peaks were predicted from the Aroclor 1248 pattern with no significant change in total PCB concentration and with an improvement in overall fit of the regression. Removal of 1242 from the calibration table did not affect quantitation of samples for which 1242 was not initially detected. The PCB patterns in all samples were represented by a mixture of Aroclors 1248, 1254 and 1260 and are reported as such. All coefficients of determination (R^2) were greater than 0.937 for the regression of predicted on actual peak areas. The mean value of R^2 was 0.9718. Confidence limits for outlier rejection were set between 95% and 99% to produce optimum fits of the regression. PCB peak areas predicted by COMSTAR for a fillet sample are compared to the observed peak areas in Figure 1. The plotted residuals are evenly distributed above and below the zero axis and along the length of the chromatogram, which indicates that there was no systematic bias in the quantitation.

All peak areas were normalized to the peak area of the internal standard in COMSTAR, thus all reported concentrations are corrected for recovery through the extraction and injection procedures.

Figure 1. Comparison of a reconstructed PCB chromatogram for a sample and the reconstructed PCB chromatogram generated by COMSTAR for that sample. The absolute difference between the two chromatograms (c) is displayed on the same scale as the observed and predicted chromatograms.



Data Analysis

All statistical analyses were performed using SAS Version 6.02 for Personal Computers [17] on an AT-compatible microcomputer (Model EX-1700C, Everex Systems, Inc., Freemont, CA). All samples were included in all statistical analyses of total concentration of PCBs since all measured concentrations exceeded the quantifiable limit by at least one order of magnitude. The level of significance that was used for all statistical tests was $p \leq 0.05$.

QUALITY CONTROL AND QUALITY ASSURANCE

Samples were extracted in groups of 6 to 12. A procedural blank was extracted with each group. Every tenth sample was extracted in duplicate from separate jars of fillet homogenate. Standards prepared from commercial Aroclors were extracted to determine extraction efficiencies, reportable limits, and performance of the quantitation procedure.

No Aroclors were detected in any of the twelve procedural blanks. An Aroclor standard of 125 ng/ml was detected and quantitated under the same conditions as the fillet extracts. This concentration corresponds to a concentration in the fillets of 0.0125 ug/g (ppm) wet weight which is less than 10% of the lowest concentration observed in the fillet samples.

The extraction and quantitation was shown to be reproducible by replicate extractions of an Aroclor standard

mixture (Table A.1) and by duplicate extractions of eight randomly selected fillet homogenates (Table A.2). A safflower oil blank was spiked with Aroclor standards such that the concentration of a one ml final extract was similar to an average fillet extract: 1.93 ug/ml Aroclor 1248, 4.55 ug/ml Aroclor 1254 and 2.10 ug/ml Aroclor 1260 for a total PCB concentration of 8.49 ug/ml. The coefficient of variation (CV, the standard deviation expressed as a percentage of the mean) for the concentration of total PCBs for three extractions was 2.4% (Table A.1). The CVs for the duplicate analyses of actual fillets were slightly greater than those for Aroclor standards which may indicate a small amount of variation among subsamples of a fillet homogenate. The median CV for the eight duplicate PCB analyses was 7.3% and the mean was 6.8% (Table A.2).

All reported concentrations are corrected individually for extraction recovery and injection variation with the use of the internal standard, 2,4,6-T_3CB. The Aroclor standard mixture described above was extracted in triplicate to verify the appropriateness of this internal standard. The calculated percent recovery of this standard mixture was 110.8% based on nominal concentration and 107.4% based on quantitation before and after extraction (Table A.1). Recovery of individual peaks averaged 112.1% with an average standard deviation for individual peaks of 3.4%. The recoveries of the three Aroclors present in the mix were 115.4%, 105.0%, and 119.7% for Aroclors 1248, 1254 and 1260,

respectively, based on nominal concentrations (Table A.1). Percent recovery of the internal standard, which indicates uncorrected recovery of samples, was 90.2 $\pm$ 24.1 ($\bar{x}$ + s) based on external standard calibration curves (Table A.3).

The quantitation of Aroclor standards was linear (R^2 = 0.999, n=12) over a range of 0.125 to 40 ug/ml total PCB. This range corresponds to a range of concentrations in samples from 0.0125 to 4.0 ug/g (Figure A.1). The actual range of concentrations observed in samples was 0.14 to 2.09 ug/g. Therefore, all samples were quantitated within the linear response range of the quantitation system.

Accuracy and precision of the quantitation was evaluated with six Aroclor standard mixes which were representative of the range of Aroclor concentrations and proportions observed in the fillet samples. One mix was injected five times to determine the precision of the injection and quantitation. The average CV for the three individual Aroclors was 2.6% (Table A.4). Relative differences in observed and nominal concentrations were determined for the six mixes to evaluate the accuracy of the quantitation (Table A.5). Relative differences averaged over the six standard mixes were 4.2%, -7.3%, 3.9% and 1.5% for Aroclors 1248, 1254 and 1260 and total PCB, respectively. These values are all within one standard deviation of 0.0% difference between nominal and observed concentrations.

The lipid determination was also evaluated for repeatability (precision) and accuracy. Standard solutions

of safflower oil were prepared at concentrations spanning the range of those determined in samples and analyzed in triplicate by the same method as the samples. The lipid determination was found to be highly repeatable as evidenced by the small standard deviations and CVs for the analyses (Table A.6). The recovery of lipid was consistent over the range corresponding to 2% to 25% lipid in the samples. This range includes 64% of the fillets. Recovery for these standards was less than 100% but it is unknown if these results indicated the presence of volatile components in the safflower oil (which may or may not occur with fish oils) or a procedural error (e.g. inaccurate, although precise, pipetting) which would have affected fillet samples as well. Four extractions were made of solutions containing safflower oil at 2.75% (Table A.7). The observed recoveries were precise (CV = 2.6%) and averaged 64%, indicating a loss of oil mass in the $Na_2SO_4/MeCl_2$ extraction step in addition to the procedural loss noted above.

The accuracy of the lipid determination in actual fillet samples could not be evaluated because the true lipid concentration was not known in any fillet samples nor was fish oil standard available. Results are within the expected range of those observed in salmonid flesh samples, however [4,8]. The precision obtained in lipid determinations was evaluated within and among extracts of fillets. Lipid determinations within extracts were precise with a mean CV of 9.4% for nine extracts, each of which was analyzed in

triplicate (Table A.7). Lipid determinations for duplicate extractions of fillet homogenates are presented in Table A.2. The median CV for the eight duplicate lipid analysis was 8.3% and the mean CV was 17.2%.

RESULTS

Grand Means

The means and ranges of the parameters measured provide general information on chinook salmon from the Ludington area (Table 2). The eighty-one fish sampled ranged in length from 15.75" to 37.75" and from 1.5 to 17.5 lbs in weight. The following equation describes the length-weight relationship of the chinook salmon sampled (Figure 2):

$$WEIGHT = 0.755 * LENGTH - 12.467 \quad (R^2=0.9076)$$

when WEIGHT was measured in pounds and LENGTH in inches. The average lipid content of the fillet homogenates was 3.1%. Total PCB concentrations in the fillets averaged 0.94 ug/g, wet weight, and ranged from 0.14 to 2.10 ug/g, wet weight. Unless otherwise noted all concentrations are reported on a wet weight basis. The pattern of PCBs observed in the fillets could be described as a mixture of Aroclors 1248, 1254 and 1260. The ratio of the average concentrations of the Aroclors was 1.0:2.4:1.2, but this ratio was not constant across the range of total PCB concentrations or fish size class (Figure 3). The contribution of Aroclor 1248 (with less chlorinated congeners) increased in relation to that of 1254 and 1260 as total PCB concentration increased in the samples.

Table 2. Descriptive Statistics for All Variables Measured

Variable	N	Minimum	Maximum	Mean	Variance	Std Dev	Std Err	CV
LENGTH	81	15.750	37.750	28.150	31.822	5.641	0.627	20.04
WEIGHT	81	1.500	17.500	8.788	19.989	4.471	0.497	50.87
LIPID	81	0.102	18.375	3.071	7.188	2.681	0.298	87.30
TOTPCB	81	0.140	2.095	0.937	0.185	0.430	0.048	45.93
PCBLIP	81	5.327	595.437	68.944	9740.58	98.694	10.966	143.15
1248	81	0.000	0.717	0.203	0.022	0.148	0.016	72.83
1254	81	0.081	1.265	0.483	0.051	0.226	0.025	46.84
1260	81	0.039	0.604	0.251	0.012	0.108	0.012	42.98

Variable	Units
LENGTH	inches of whole fish, nose to tail
WEIGHT	pounds of whole fish
LIPID	% by weight in trimmed fillet
TOTPCB	ug/g wet weight in trimmed fillet
PCBLIP	ug/g lipid in trimmed fillet
1248	ug/g wet weight in trimmed fillet
1254	ug/g wet weight in trimmed fillet
1260	ug/g wet weight in trimmed fillet

Figure 2. Linear regression of fish weight as a function of fish length. Dashed lines are the 95% confidence intervals for individual observations.

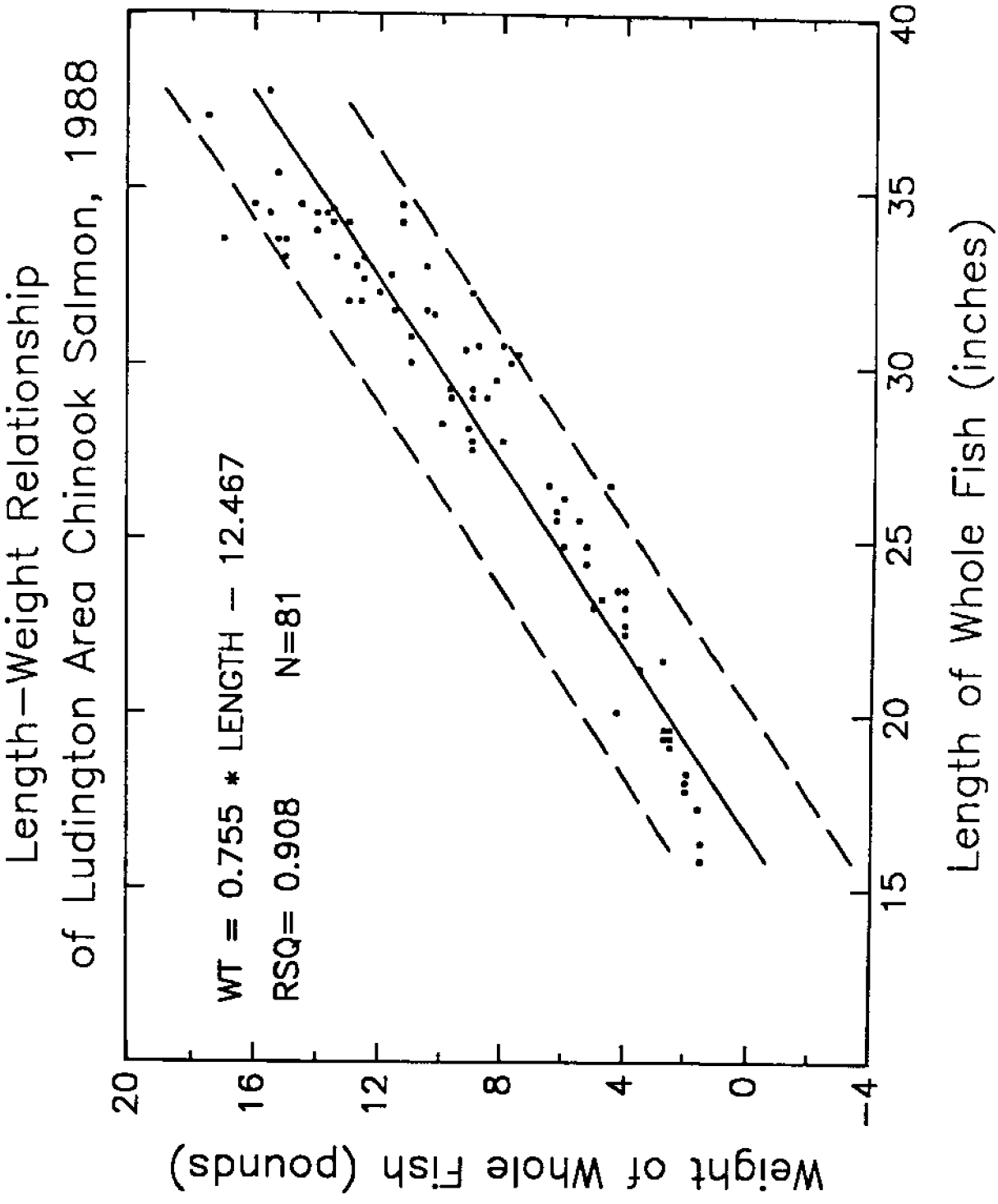
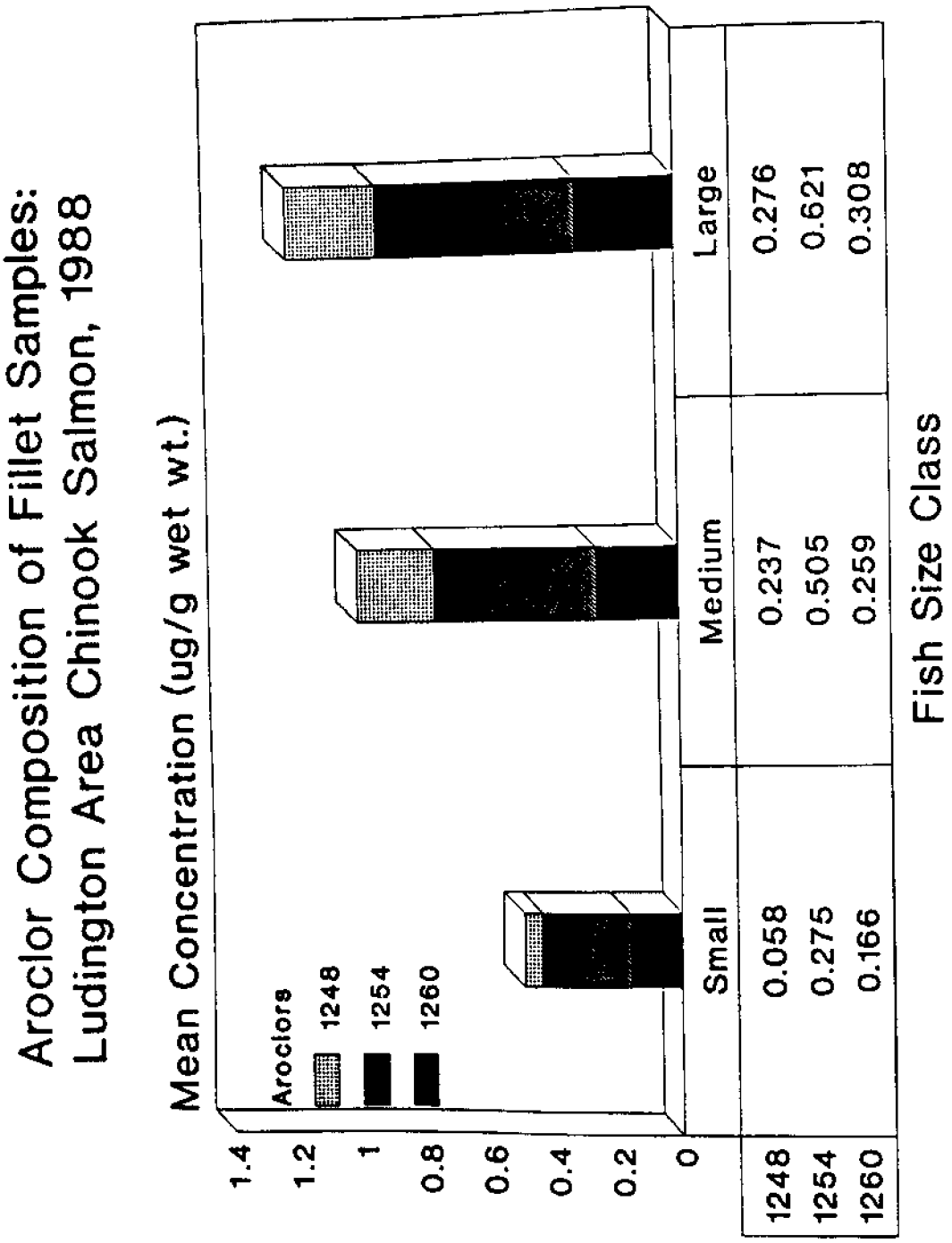


Figure 3. Mean Aroclor composition of fillets in each of the three size classes of chinook salmon. The mean concentrations of Aroclors 1248, 1254 and 1260 within each size class are listed in the data table in ug/g wet weight.



PCB Concentrations by Season, Size Class and Sex

Concentrations of total PCB were compared for fish of different size classes and from each of the three collection periods (Table 3 and Figure 4). Very little seasonal variation in PCB concentration was observed (Figure 5). Fish sampled in the spring had a mean PCB concentration of 1.07 ug/g (SD = 0.37) which was significantly greater than the concentration found in the summer fish, 0.81 ug/g (SD = 0.42). The concentration of PCB of fish collected in the fall was 0.92 ug/g (SD = 0.47) which was not significantly different from either the spring or summer fish (Tukey's Studentized Range Test). Mean length and weight were also less in the summer relative to the spring and fall (Table 4). Although the observed seasonal differences in length and weight were not statistically significant (Student-Newman-Keuls (SNK) and Tukey's multiple range tests), the variations in PCB concentration disappeared when concentrations were normalized to either weight or length (Figures 6 and 7). The differences in normalized PCB concentrations were not statistically significant (SNK and Tukey's multiple range tests).

Concentrations of PCBs varied by size class of fish, however (Figure 8). The mean PCB concentrations for each of the three size classes were 0.50, 1.00 and 1.21 ug/g for fish less than 24", between 24" and 32", and greater than 32" in total length, respectively. The concentrations of PCBs in small fish were significantly different from each of the other

Table 3. Concentrations of total PCBs in ug/g wet weight for trimmed fillets of fish collected in three time periods in each of three size classes.

Total PCB Concentration (ug/g wet wt.)

mean (std. dev.)	May	July	September	mean (std. dev.)
< 24"	0.82 (0.34)	0.37 (0.15)	0.53 (0.26)	0.50 (0.27)
24" - 32"	0.99 (0.31)	0.95 (0.25)	1.06 (0.33)	1.00 (0.29)
> 32"	1.33 (0.39)	1.12 (0.36)	1.18 (0.52)	1.21 (0.42)
mean (std. dev.)	1.07 (0.37)	0.81 (0.42)	0.92 (0.47)	0.94 (0.43)

Figure 4. Means of concentrations of total PCBs in trimmed fillets of fish collected in three time periods in each of three size classes.

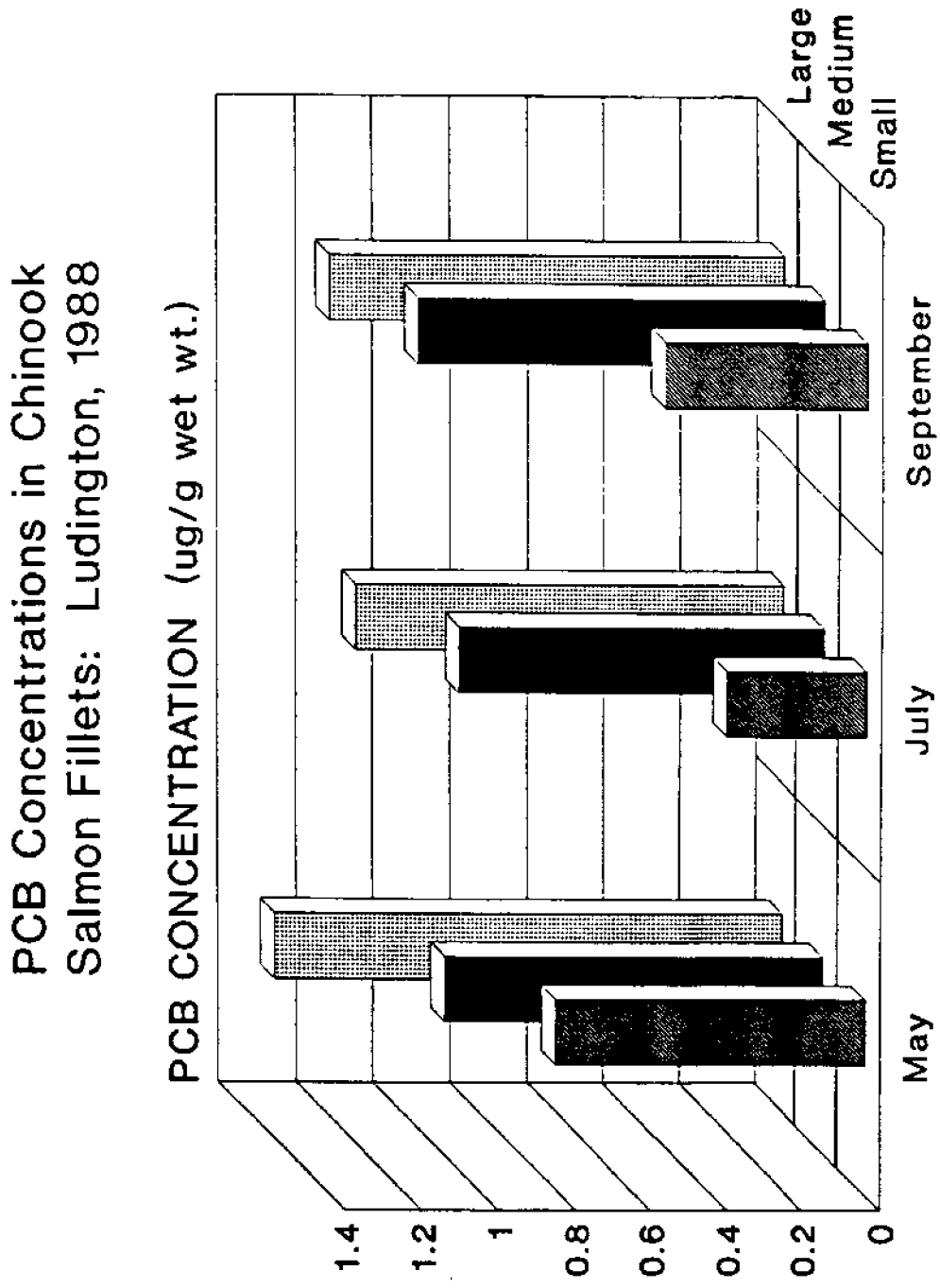
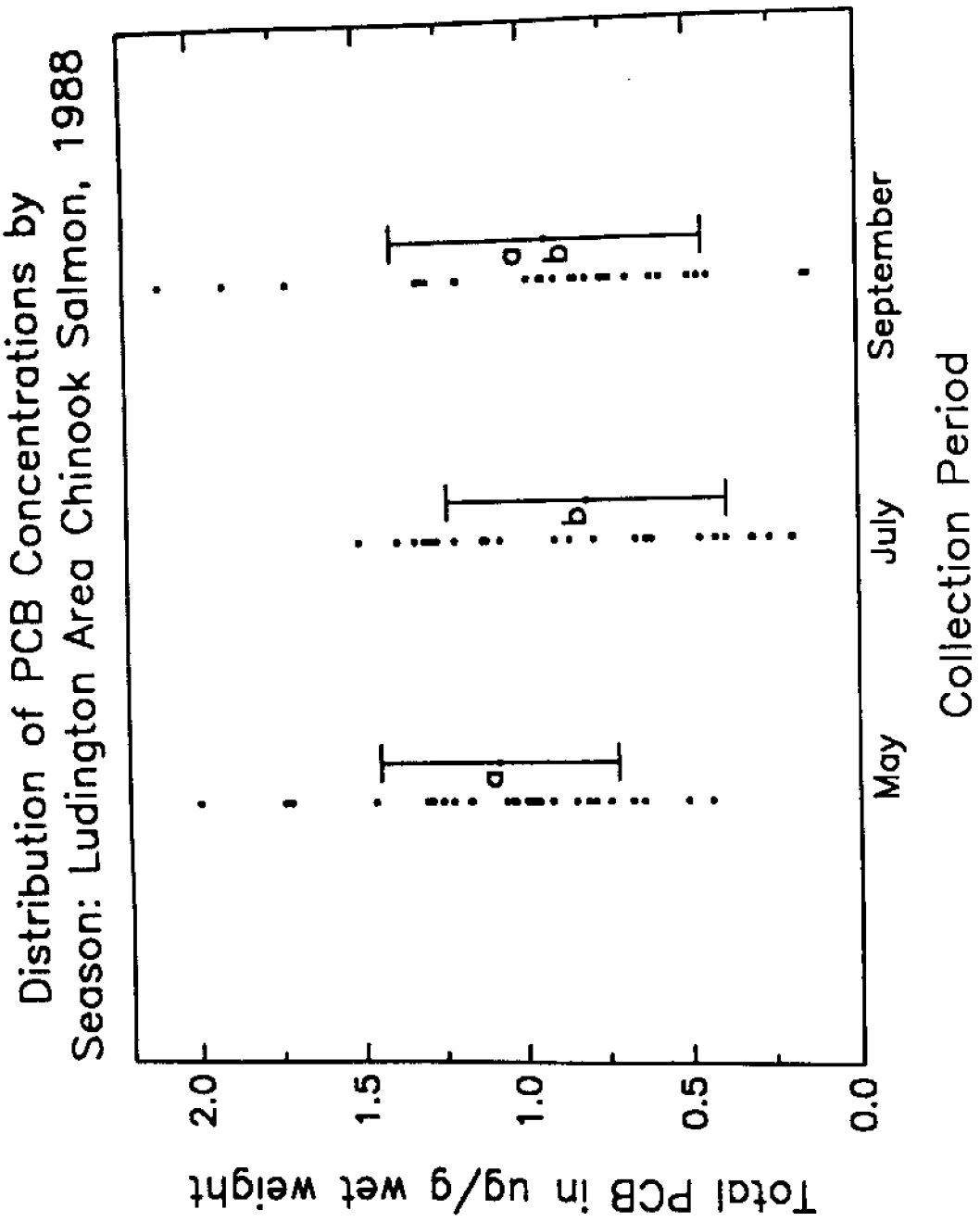


Figure 5. Concentrations of total PCBs in individual fillets. Observations are grouped by collection period.



Note: Bars indicate mean and standard deviation. Means labelled with the same letter are not significantly different at the 0.05 level.

Table 4. Fish lengths, measured from nose to end of tail, for chinook salmon collected in three time periods in each of three size classes.

Fish Lengths (inches)

	May	July	September	mean (std. dev.)
< 24"	23.2 (0.3)	17.9 (1.3)	21.4 (1.6)	20.2 (2.5)
24" - 32"	28.8 (2.5)	28.6 (1.9)	28.6 (2.4)	28.7 (2.3)
> 32"	33.6 (1.0)	33.5 (0.7)	34.5 (1.8)	33.9 (1.3)
mean (std. dev.)	29.6 (3.8)	26.7 (6.8)	28.2 (5.8)	28.1 (5.6)

Figure 6. Means of weight-normalized concentrations of PCBs. Season averages are not significantly different ($p \leq 0.05$).

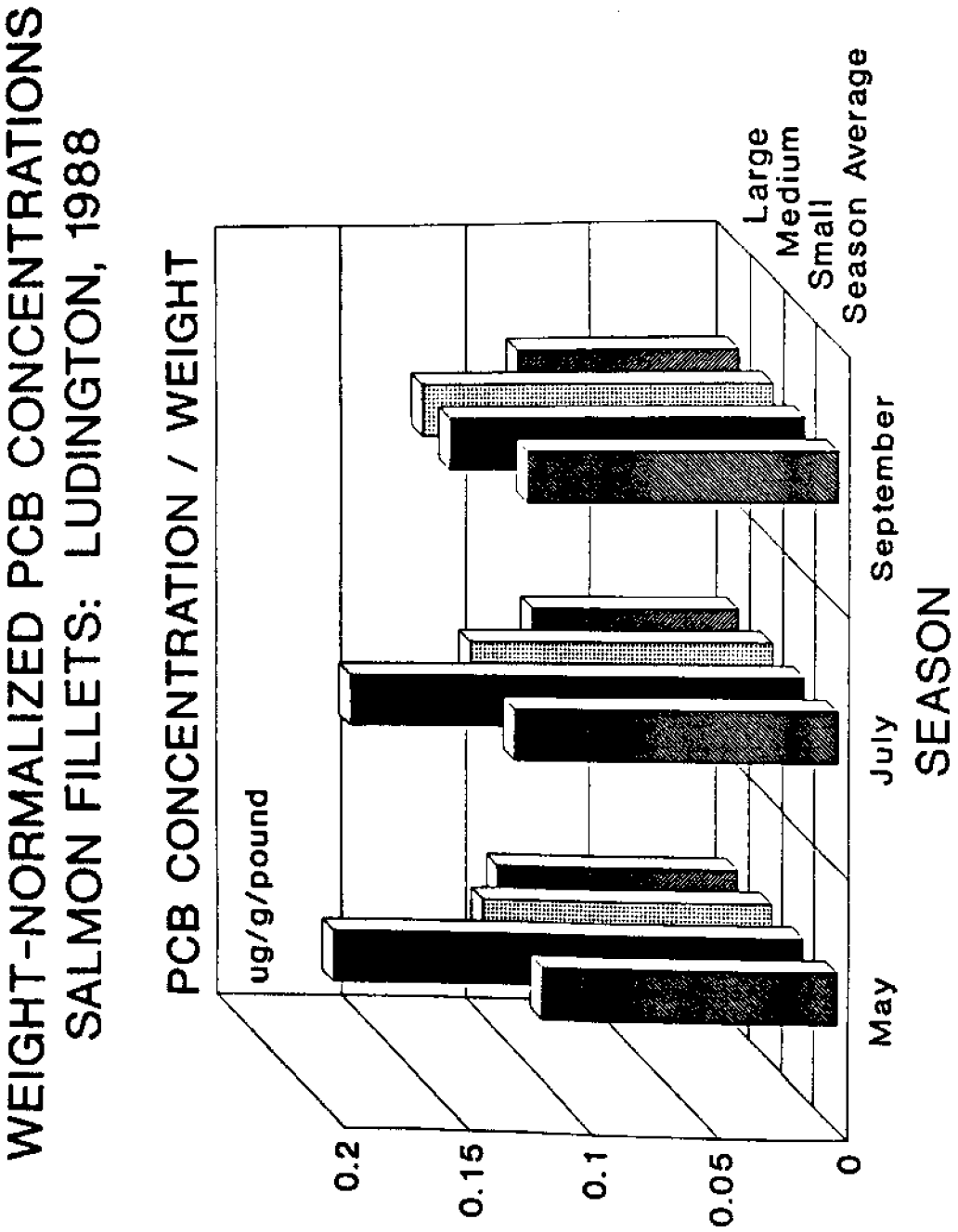


Figure 7. Means of length-normalized concentrations of PCBs. Season averages are not significantly different ($p \leq 0.05$).

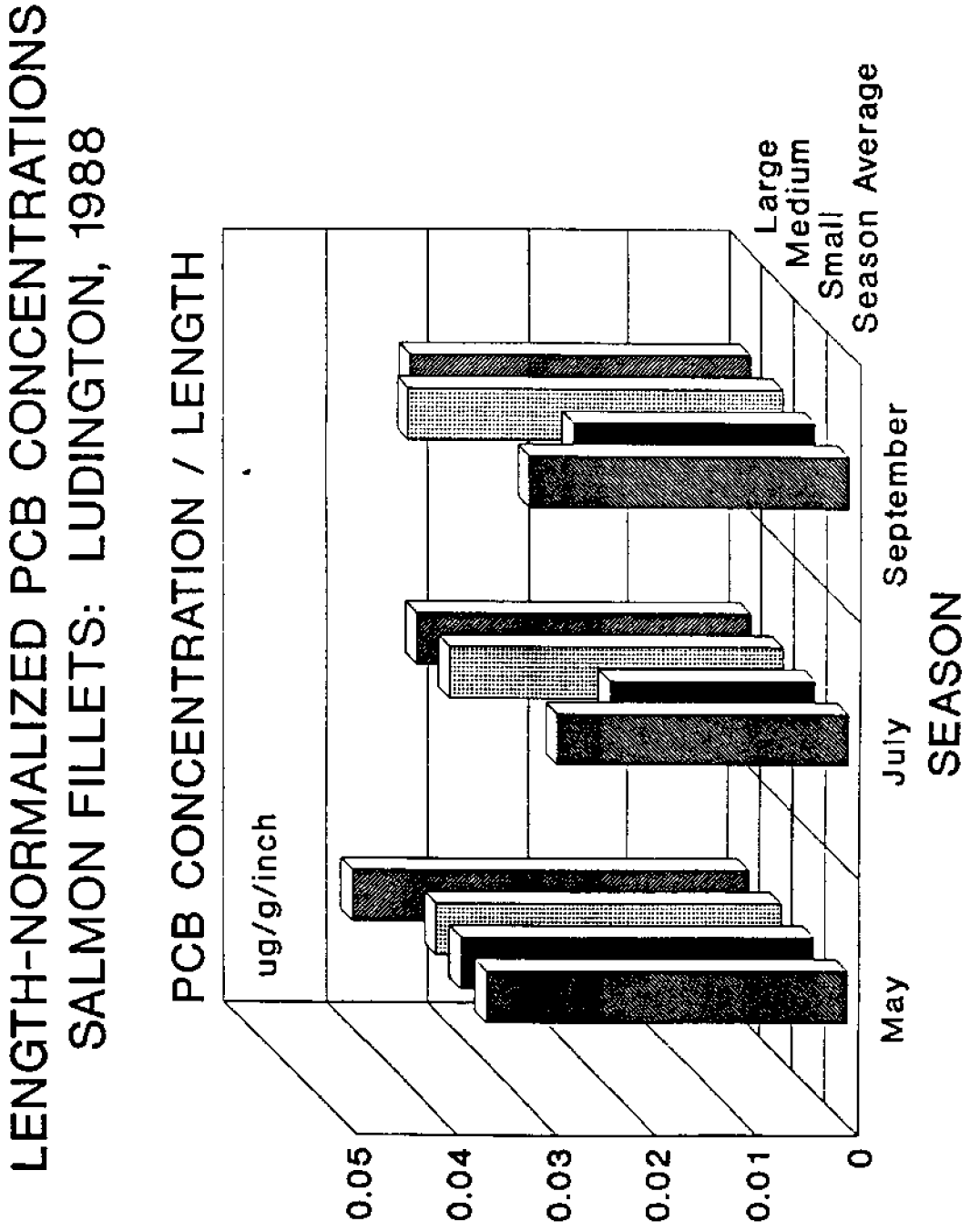
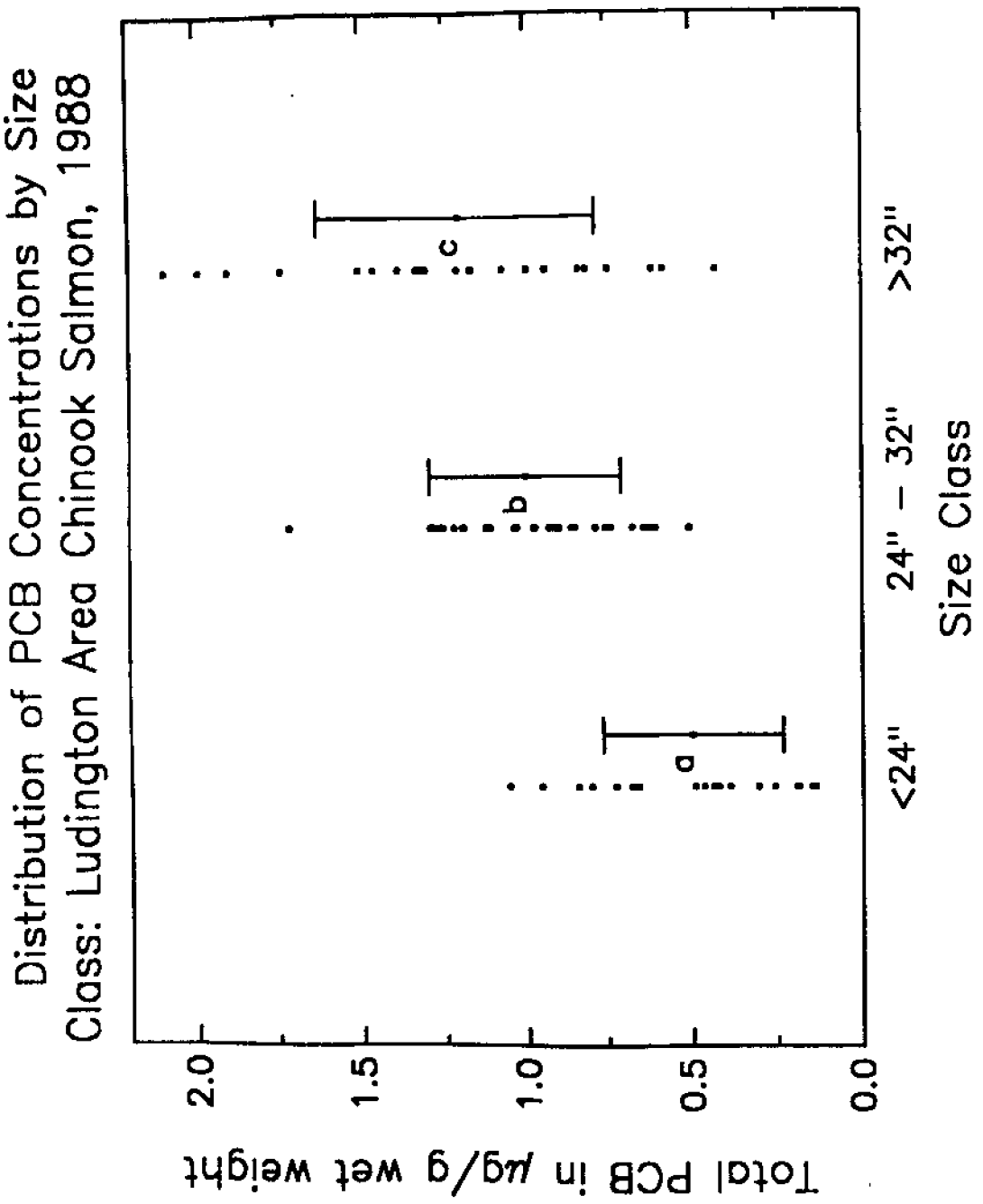


Figure 8. Concentrations of total PCBs in individual fillets. Observations are grouped by fish size class.



Note: Bars indicate mean and standard deviation. Means labelled with the same letter are not significantly different at the 0.05 level.

size classes in all statistical tests performed. PCB concentrations in medium and large fish classes were found to be significantly different by the LSD T-test, the SNK test and Duncan's Multiple Range test but not by Tukey's test.

The mean concentrations of PCBs in fillets from female and male fish were 1.04 and 0.86 ug/g, respectively, and significantly differed from one another (SNK and Tukey's tests). Female fish were also significantly larger than male fish, however. When PCB concentrations were normalized for either weight or length, the differences between PCB concentrations in the two sexes of salmon were no longer statistically significant.

PCB Concentration as a Function of Fish Length and Weight

Length and weight were more predictive of PCB concentration than date collected or lipid content. Length and weight were highly correlated themselves (Figure 2) and, as expected, they predicted PCB concentration similarly. Variations in fish weight accounted for approximately 48% of the variablility in PCB concentration while the proportion explained by length was 45%. Using length and weight together to predict PCB concentration did not increase predictive power because length and weight were intercorrelated. The R^2 for PCB concentration as a function of both length and weight was 0.48. Fillets of Ludington area chinook salmon were predicted to contain 2.00 ug/g (ppm) total PCBs at a fish length of 37.5 in. and a weight of 16.4 lbs.

The 95% confidence intervals for individual values in the

above regressions (Figures 9 and 10) indicate the range of PCB concentrations within which 95% of fish of a given length or weight will be found. For example, a fish which is 30" long would be expected to have a concentration of PCBs in the fillet of 1.03 ug/g, but may have a fillet PCB concentration between 0.39 and 1.68 ug/g, based on the 95% confidence intervals generated by this data. Confidence intervals can also be used to estimate the size range of a fish containing a given concentration of PCBs. For instance, 95% of the fish containing 2.00 ppm total PCB would measure between 28.7" and 46.2" and weigh between 9.7 and 23.2 pounds.

PCB Concentration as a Function of Lipid Content

PCB content was not correlated with lipid content in the trimmed fillets (Figure 11). The 95% confidence intervals for the regression of total PCB on fillet lipid content suggest that lipid values cannot be used to predict PCB concentrations for this type of sample. A fillet with a 5% lipid content, for example, could have a PCB concentration nearly anywhere within the range of PCB concentrations observed in the data set. Furthermore, lipid content and fish length were not related: The R^2 was 0.000. No trend in lipid content of the trimmed fillets was observed over the range of fish lengths (Figure 12). It is not surprising, therefore, that lipid-normalized PCB concentrations do not correlate as well as wet weight based concentrations with length (Figure 13).

Figure 9. Linear regression of concentration of PCBs as a function of fish weight. The dashed lines are the 95% confidence intervals for individual observations.

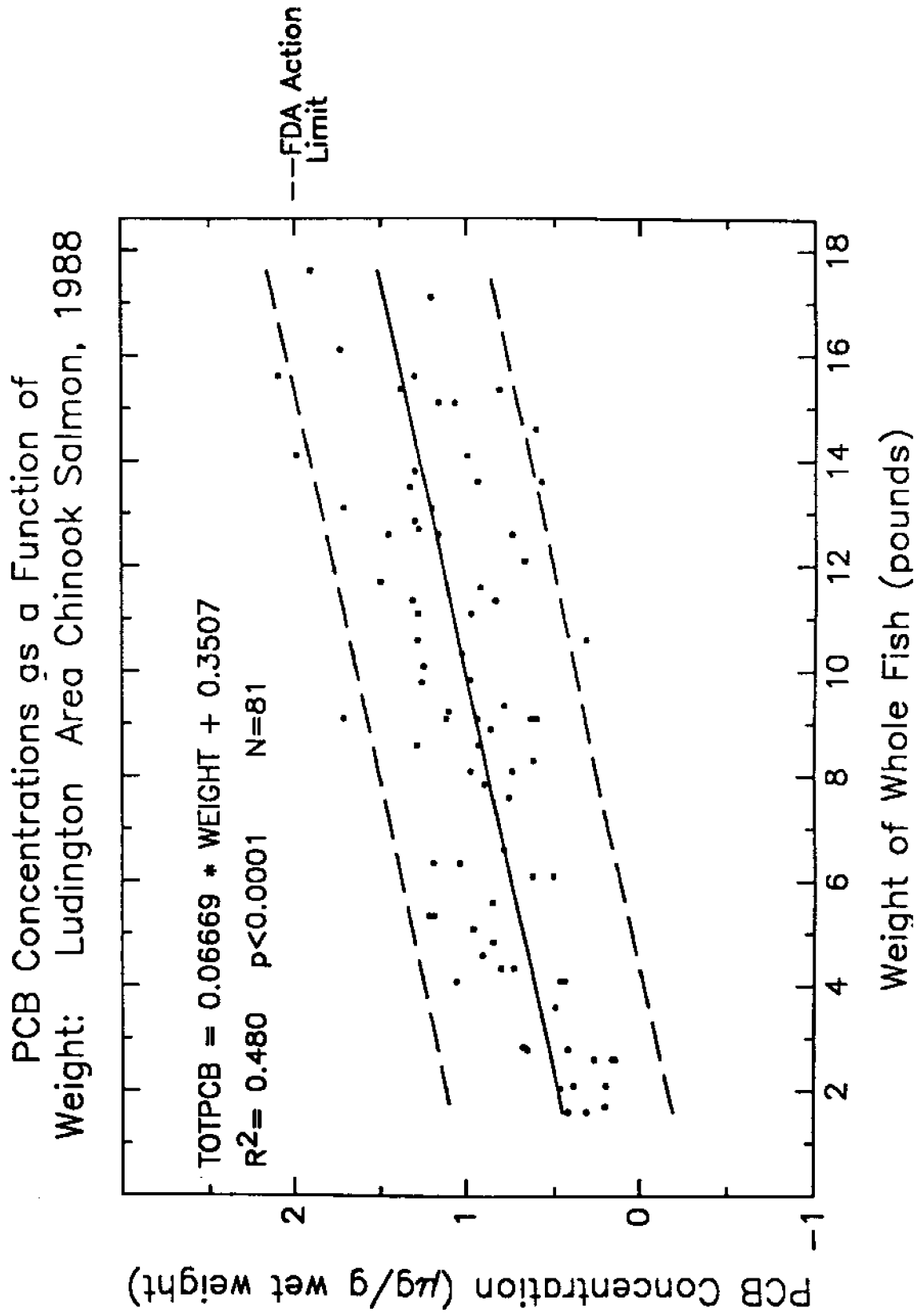


Figure 10. Linear regression of concentration of PCBs as a function of fish length. The dashed lines are the 95% confidence intervals for individual observations.

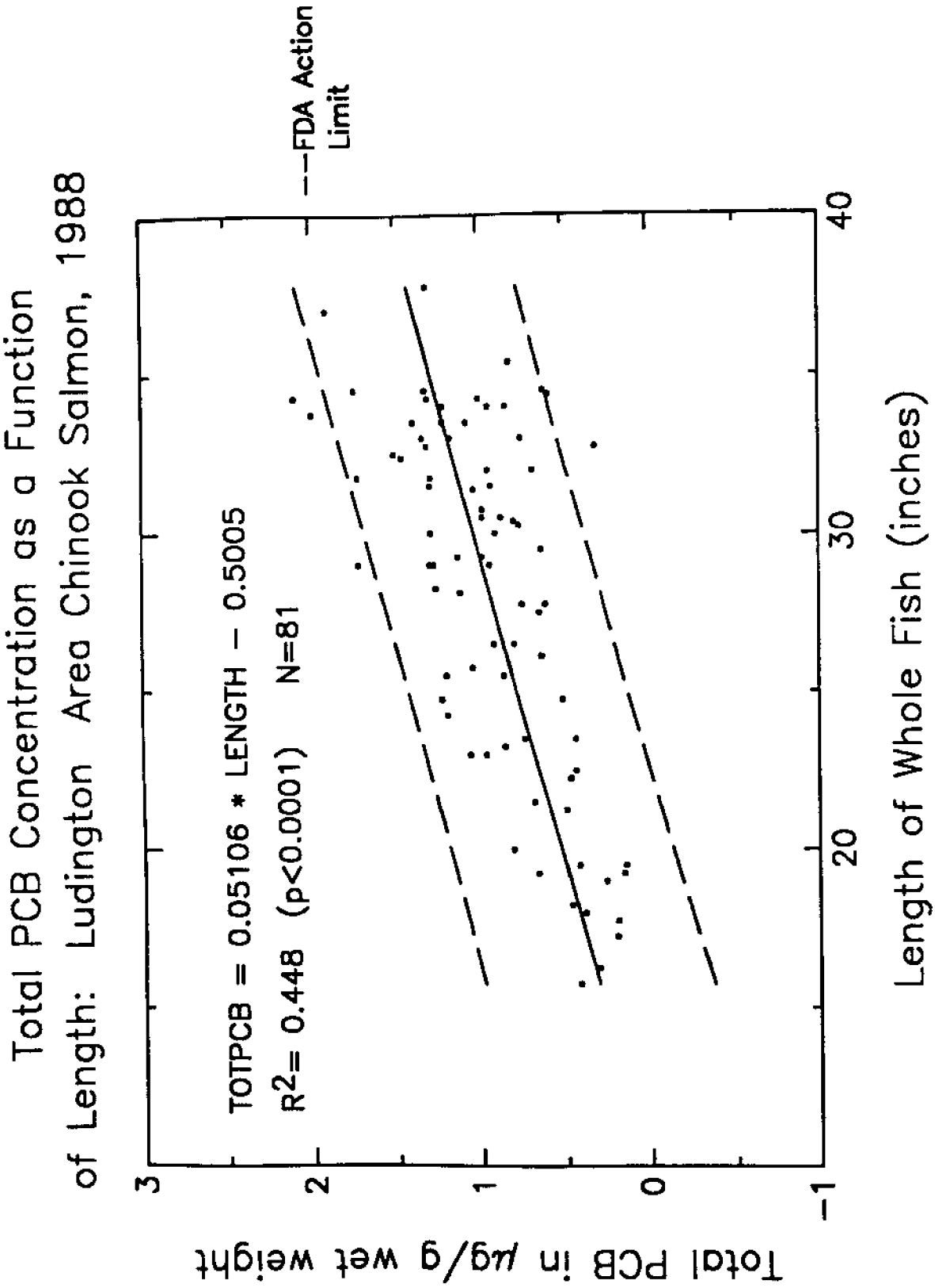


Figure 11. Linear regression of concentration of PCBs as a function of fillet lipid content. The dashed lines are the 95% confidence intervals for individual observations.

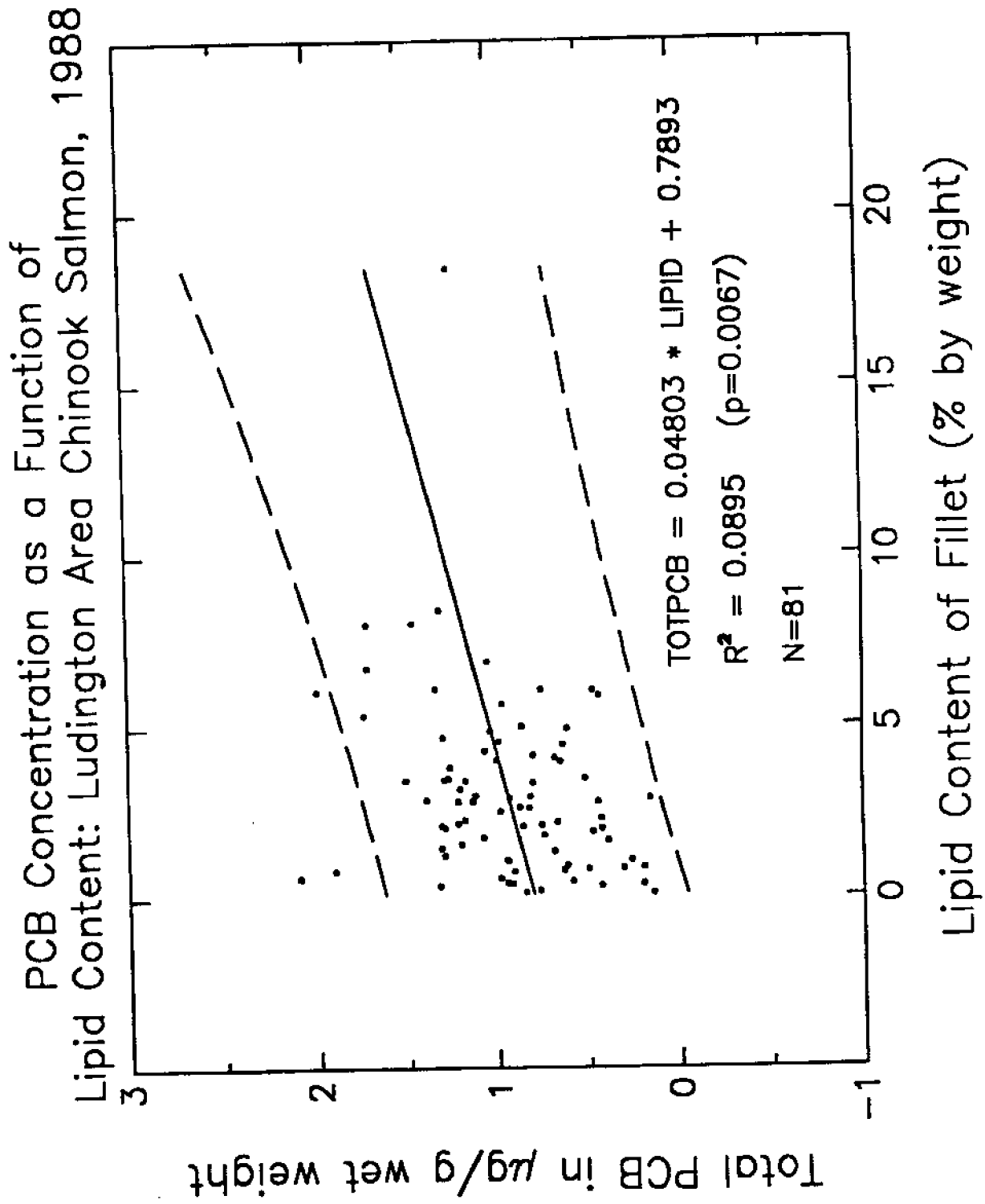


Figure 12. Linear regression of fillet lipid content as a function of fish length. The dashed lines are the 95% confidence intervals for individual observations.

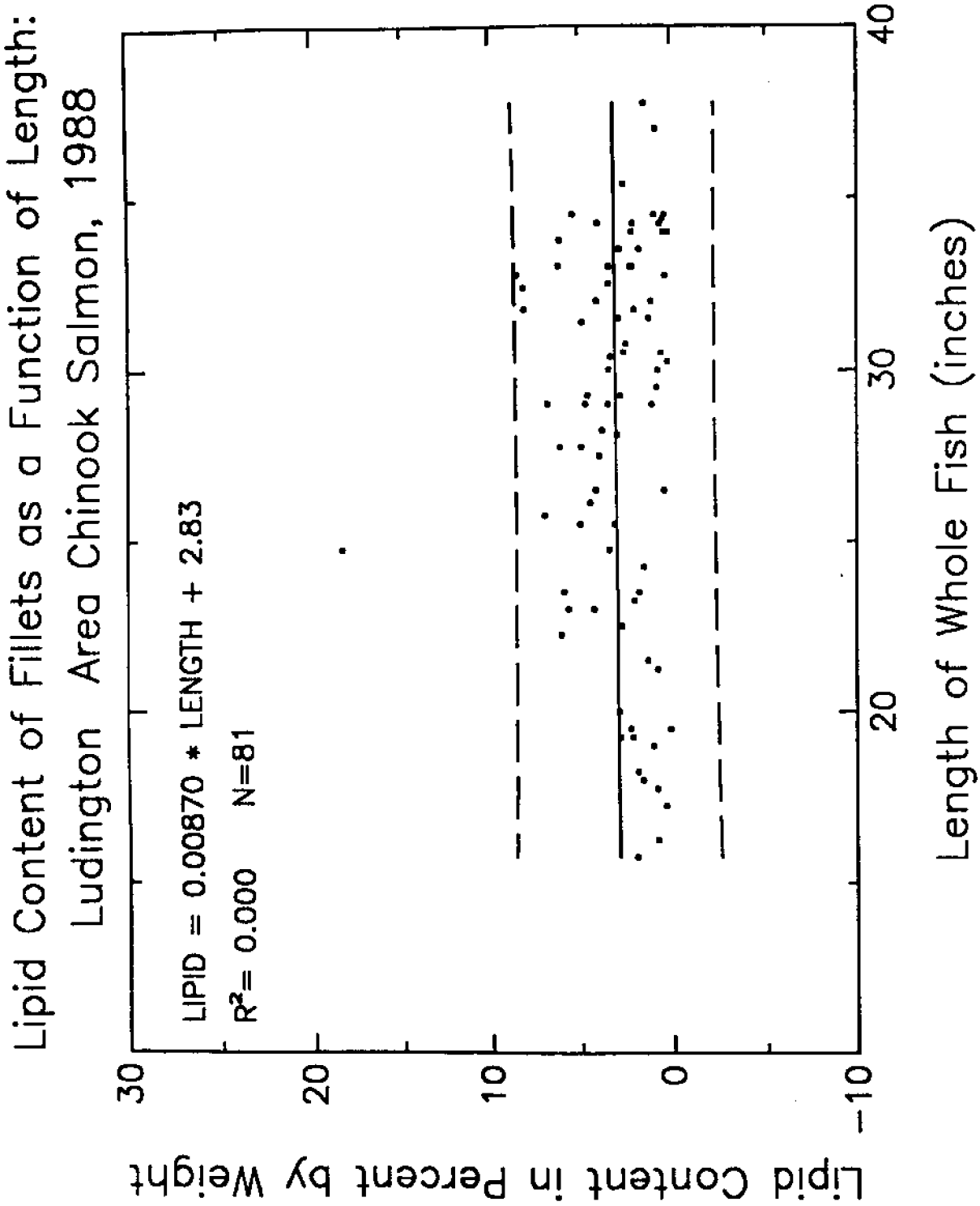
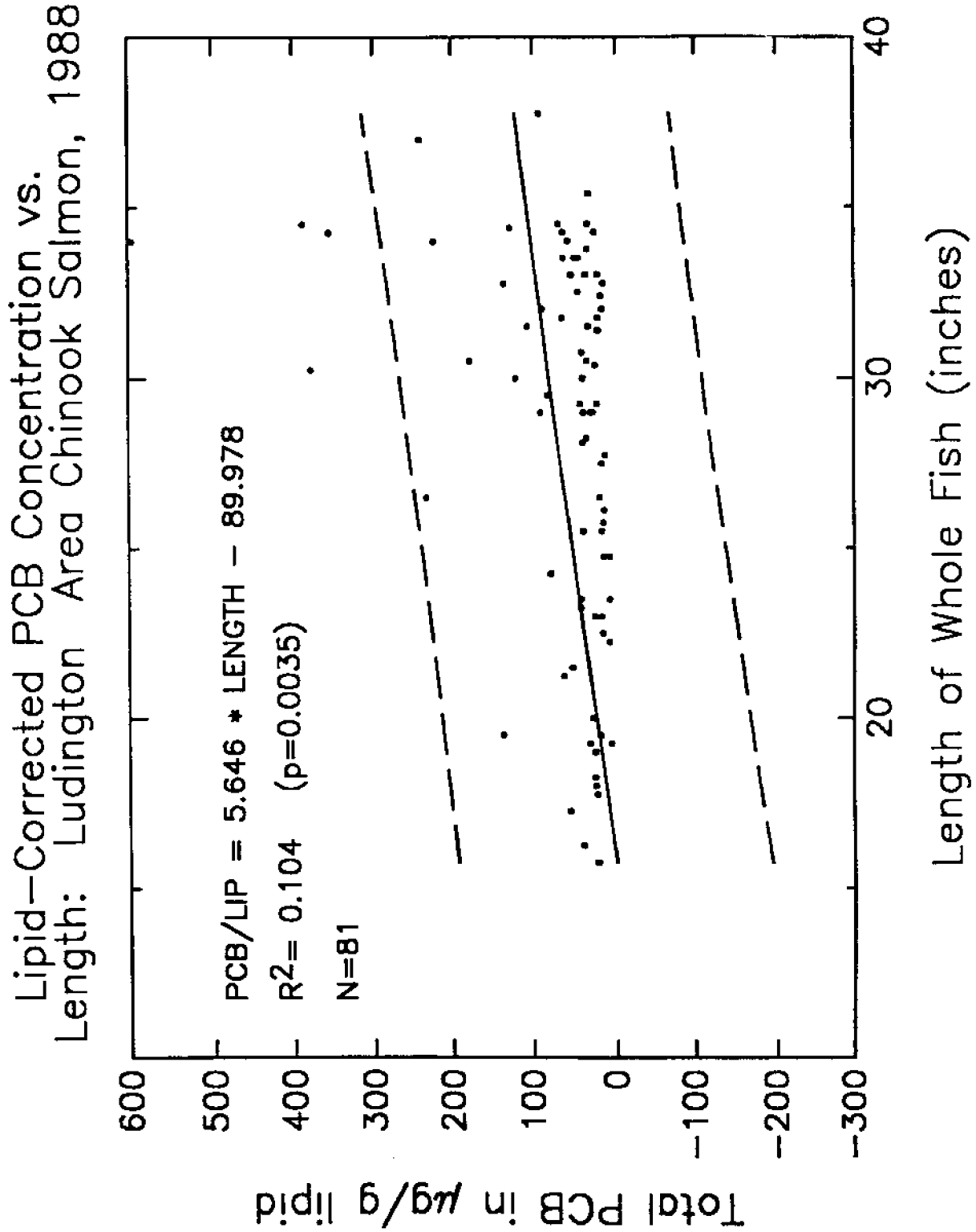


Figure 13. Linear regression of concentration of PCBs expressed as ug/g lipid as a function of fish length. The dashed lines are the 95% confidence intervals for individual observations.



DISCUSSION

In general, the mean concentration of total PCBs found in trimmed fillets from the Ludington area of Lake Michigan, 0.94 ug/g, is less than concentrations measured in whole fish and skin-on fillets of Great Lakes salmonids. Concentrations of total PCBs in whole lake trout from near Saugatuck, MI, decreased from 23 ppm in 1974 to 4.6 ppm in 1981 and 5.6 ppm in 1982 [18,19]. Mean PCB concentrations of skin-on fillets of chinook salmon from the northern and southern zones of western Lake Michigan were 1.45 ppm and 1.10 ppm, respectively, in 1985 [8]. Skin-on fillets from the 1984 run of coho salmon in the Platte River (MI) contained approximately 0.5 ppm of total PCBs [20].

The relationship between concentration of PCB and fish length determined in this study is slightly different from those found for chinook salmon in western Lake Michigan. A study of skin-on fillets of chinook salmon from the Wisconsin waters of Lake Michigan demonstrated the following relationships for fish caught north and south of Sheboygan County, WS, which is at approximately the same latitude as Ludington, MI: PCB = 0.12*LENGTH - 1.63 (R^2=0.68) and PCB = 0.10*LENGTH - 1.20 (R^2=0.65) [8]. The coefficients of determination and slopes of these relationships indicate a stronger and more positive relationship between length and PCB concentration for the skin-on fillets of chinook salmon from western Lake Michigan in 1985 than for the trimmed fillets of chinook salmon from the Ludington, MI, area in 1988.

The seasonal differences in concentration of PCBs in western Lake Michigan chinook salmon observed in 1985 [8] were only partially corroborated by this study. The concentrations of PCBs in fillets of Ludington area chinook salmon caught in the summer were less than in those caught in the spring and fall, as was the case for the western Lake Michigan fillets [8]. However, this difference was no longer statistically significant for the trimmed fillets when concentrations were normalized to fish length and disappeared altogether when concentrations of PCBs were normalized to fish weight.

This investigation was not designed to measure PCB reduction by trimming, but to accurately measure PCB content of trimmed fillets. Previous studies have shown that trimming of skin and other fatty tissues from fillets can reduce the concentration of Aroclor 1254 by 40-60% in various salmonid species [15]. Thus, the PCB concentrations presented in this report are approximately one-half of what would have been measured in untrimmed, skin-on fillets. If the concentrations of total PCBs in the individual fillets are multiplied by a factor of two, 33 (41%) would exceed the FDA action limit of 2 ppm instead of only one fillet in the 81 analyzed. Of these 33 fish whose fillets might have exceeded the guidelines had the fillets not been trimmed, one was less than 24 in. long, two more were less than 25 in. long, and 13 were between 25 in. and 32 in. in total length.

The results of this study can be used in the exposure section of a risk assessment for human consumption of Ludington

chinook salmon (Figure A.2). The measures of variance derived in this study indicate probable confidence intervals for the estimates of PCB concentrations in raw, trimmed fillet samples. These estimates can then be combined with fish consumption data to derive estimates of human exposure. A complete risk assessment is beyond the scope of this report, however.

The results of this study have limited applicability. The experiment was designed to produce information appropriate to questions of human exposure. Consequently, the results are less applicable to questions concerning fish health and dynamics of PCBs in the Great Lakes food web. For these two types of investigations, analyses of whole fish or skin-on fillets which can be directly compared to other available data would be more appropriate. Caution should be exercised in making generalizations about PCB and lipid dynamics, in particular, from the results of this study since the fillets were trimmed. The amount of lipid remaining in the trimmed fillet may not be related to the lipid content of the whole fish and probably is not. Furthermore, concentrations of total PCBs do not relate as well to biological effects in fish as do concentrations of mono- and non-ortho PCB congeners [22]. Ongoing and future research will address these issues.

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APPENDIX

Figure A.1. Quantitation of an Aroclor standard over a range of concentrations.

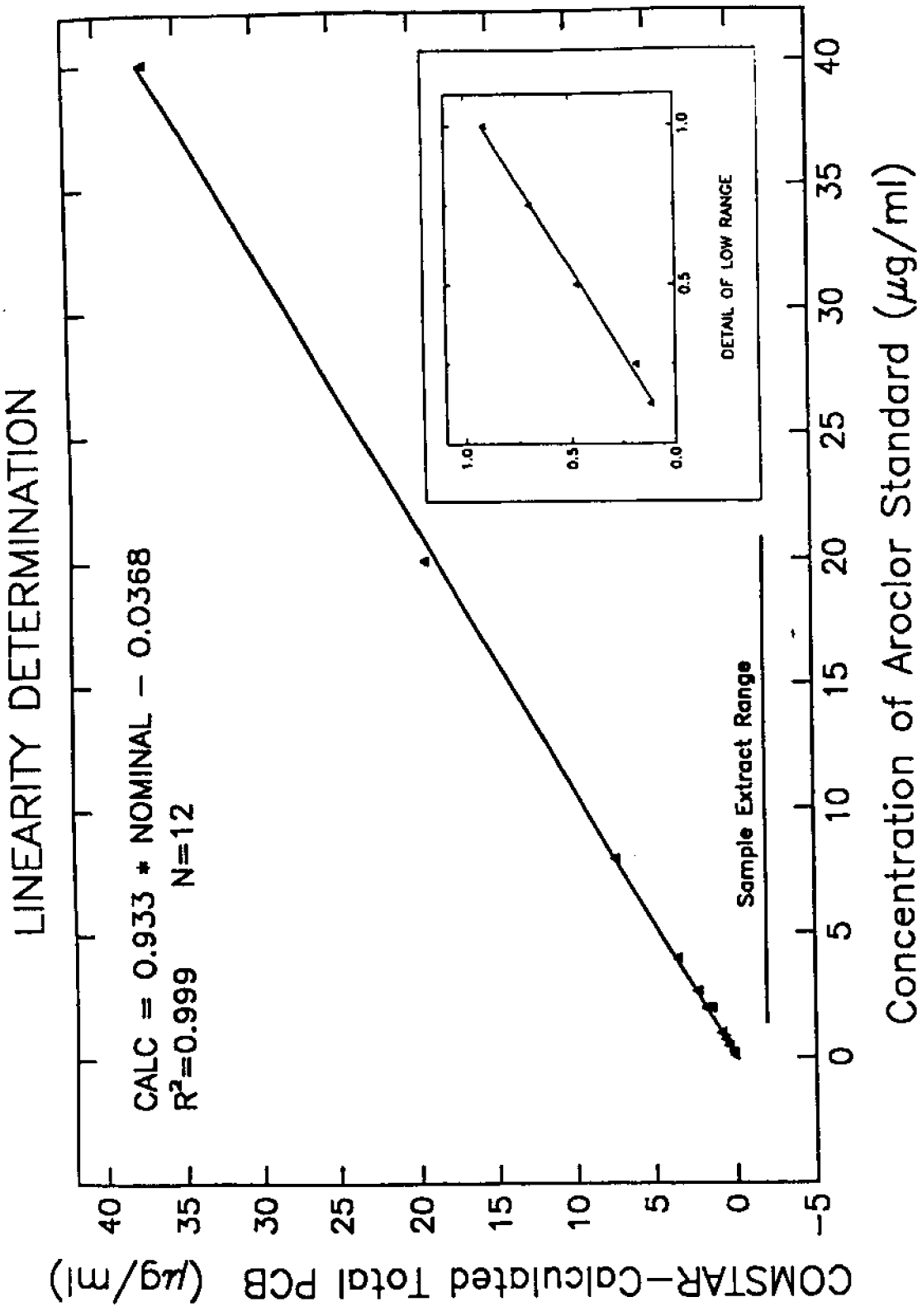

Figure A.2. Diagram of the relationship of risk assessment, risk characterization, and risk management adapted from the USEPA [21].

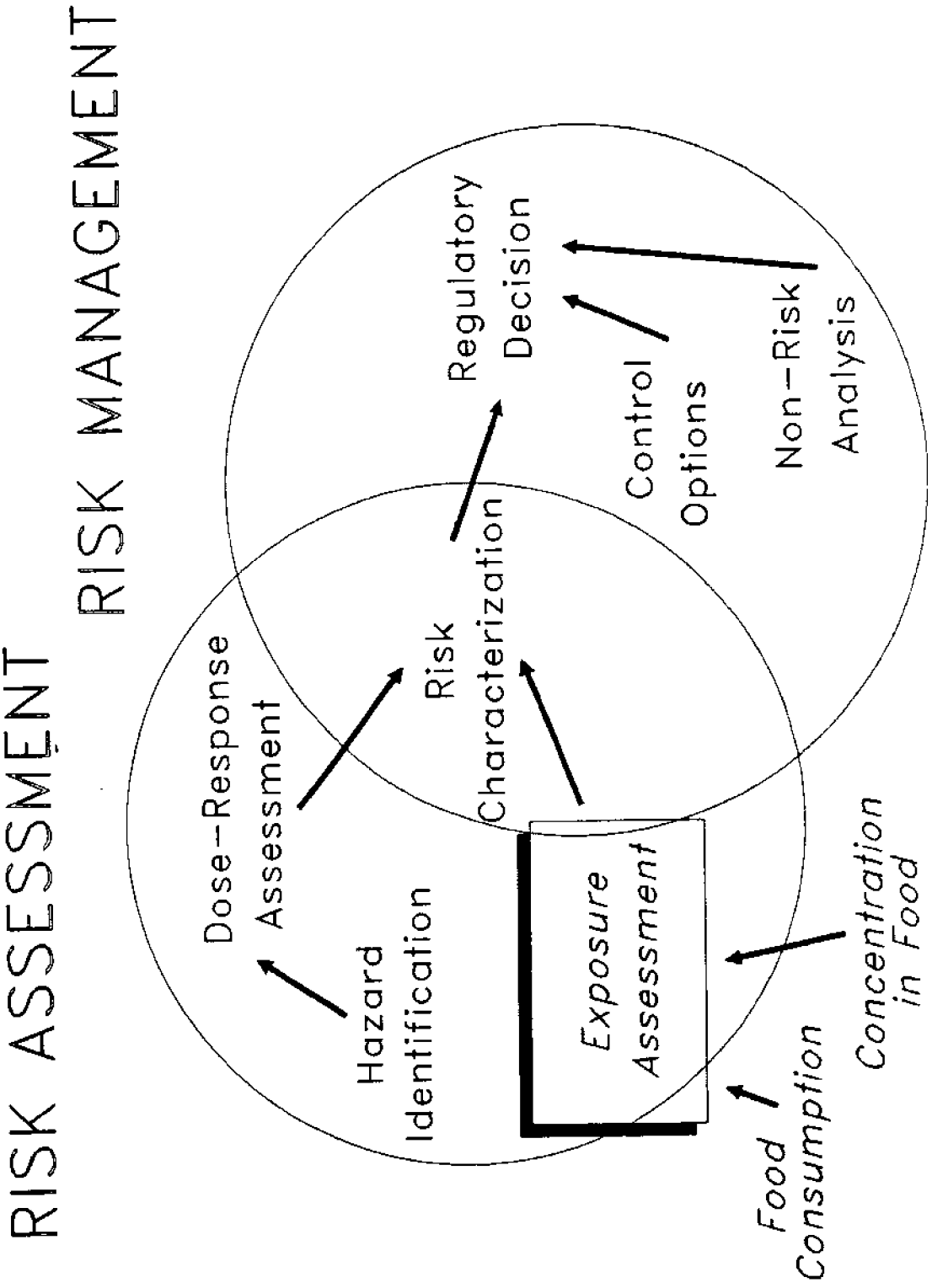

Table A.1. Accuracy and Precision of Extraction and Quantitation of an Aroclor Standard.

Extract	1248 (ppm)	1254 (ppm)	1260 (ppm)	Total (ppm)	Recovery Relative to Nominal (%) 1248	1254	1260	Total	Recovery Relative to Observed Total
Nominal	1.93	4.55	2.01	8.49	-	-	-	-	-
Observed	2.10	4.48	2.18	8.76	108.7	98.5	108.5	103.2	-
Extract A	2.11	4.91	2.40	9.42	109.8	107.8	119.3	110.9	107.5
Extract B	2.36	4.80	2.48	9.63	122.1	105.4	123.3	113.4	109.9
Extract C	2.21	4.63	2.34	9.18	114.4	101.7	116.5	108.1	104.8
			Extract Mean:		115.4	105.0	119.7	110.8	107.4
			Extract Std. Dev.:		6.2	3.1	3.4	2.7	2.6
			Extract C.V.:		5.4	2.9	2.9	2.4	2.4

Table A.2. Duplicate Analyses of Fillet Homogenates

By Extract:

Fish	Date[1]	Length (in.)	% Lipid	Total PCB[2]	PCB/ Lipid[3]	Aroclor 1248[2]	Aroclor 1254[2]	Aroclo 1260[2]
wf112428	1	32.00	3.83	0.657	17.15	0.177	0.322	0.157
wf112428	1	32.00	4.30	0.688	15.99	0.192	0.321	0.174
wf112433	1	24.75	23.77	1.189	5.00	0.468	0.538	0.183
wf112433	1	24.75	12.98	1.256	9.67	0.483	0.593	0.180
wf112478	2	32.75	0.44	0.448	101.93	0.000	0.268	0.180
wf112478	2	32.75	0.18	0.399	221.44	0.058	0.197	0.144
wf112492	1	24.75	3.68	0.540	14.68	0.179	0.245	0.116
wf112492	1	24.75	3.24	0.474	14.62	0.155	0.217	0.101
wf115926	2	33.50	3.02	1.486	49.21	0.224	0.845	0.417
wf115926	2	33.50	2.68	1.291	48.16	0.209	0.718	0.364
wf115934	2	18.25	1.92	0.449	23.37	0.000	0.284	0.165
wf115934	2	18.25	1.86	0.482	25.91	0.048	0.253	0.181
wf115948	2	19.00	1.09	0.272	24.91	0.038	0.152	0.081
wf115948	2	19.00	1.02	0.245	24.02	0.054	0.120	0.071
wf115991	2	17.25	0.37	0.204	55.11	0.022	0.112	0.071
wf115991	2	17.25	0.35	0.184	52.56	0.016	0.104	0.064

By Fish and Variable:

Fish	Variable	Mean	Std.Dev.	CV	Mean CV	Median CV
wf112428	LIPID	4.07	0.33	8.18		
wf112433	LIPID	18.38	7.63	41.52		
wf112478	LIPID	0.31	0.18	59.31		
wf112492	LIPID	3.46	0.31	8.99	17.2	8.3
wf115926	LIPID	2.85	0.24	8.44		
wf115934	LIPID	1.89	0.04	2.24		
wf115948	LIPID	1.06	0.05	4.69		
wf115991	LIPID	0.36	0.01	3.93		
wf112428	TOTPCB	0.672	0.022	3.24		
wf112433	TOTPCB	1.222	0.047	3.84		
wf112478	TOTPCB	0.424	0.035	8.33		
wf112492	TOTPCB	0.507	0.047	9.31	6.8	7.3
wf115926	TOTPCB	1.388	0.138	9.96		
wf115934	TOTPCB	0.465	0.023	5.04		
wf115948	TOTPCB	0.258	0.019	7.26		
wf115991	TOTPCB	0.194	0.014	7.27		
wf112428	PCB/LIP	16.57	0.82	4.94		
wf112433	PCB/LIP	7.34	3.30	45.01		
wf112478	PCB/LIP	161.68	84.51	52.27		
wf112492	PCB/LIP	14.65	0.05	0.32	14.7	4.1
wf115926	PCB/LIP	48.68	0.74	1.53		
wf115934	PCB/LIP	24.64	1.79	7.28		

Table A.2. (continued)

Fish	Variable	Mean	Std.Dev.	CV	Mean CV	Median CV
wf115948	PCB/LIP	24.47	0.63	2.57		
wf115991	PCB/LIP	53.83	1.80	3.34		
wf112428	1248	0.185	0.010	5.67		
wf112433	1248	0.475	0.010	2.18		
wf112478	1248	0.029	0.041	141.42		
wf112492	1248	0.167	0.017	10.14	44.0	16.0
wf115926	1248	0.216	0.011	4.97		
wf115934	1248	0.024	0.034	141.42		
wf115948	1248	0.046	0.011	24.41		
wf115991	1248	0.019	0.004	21.92		
wf112428	1254	0.322	0.001	0.20		
wf112433	1254	0.566	0.039	6.87		
wf112478	1254	0.233	0.050	21.49		
wf112492	1254	0.231	0.020	8.55	9.8	8.3
wf115926	1254	0.782	0.090	11.55		
wf115934	1254	0.269	0.022	8.01		
wf115948	1254	0.136	0.023	16.80		
wf115991	1254	0.108	0.006	5.12		
wf112428	1260	0.166	0.012	7.21		
wf112433	1260	0.182	0.002	1.22		
wf112478	1260	0.162	0.026	16.10		
wf112492	1260	0.109	0.010	9.62	8.3	8.3
wf115926	1260	0.390	0.037	9.54		
wf115934	1260	0.173	0.011	6.51		
wf115948	1260	0.076	0.007	9.49		
wf115991	1260	0.067	0.004	6.64		

[1]Values for Date signify the following: 1=May, 2=July, 3=September

[2]Concentrations are in ug/g wet weight in trimmed fillets.

[3]Concentrations are in ug/g lipid in trimmed fillets.

Table A.3. Complete Data for Individual Fish and Corresponding Fillets[1]

Fish	Date[2]	Size[3]	Sex[4]	Length (in.)	Weight (lb.)	Total PCB[5]	Aroclor 1248[5]	Aroclor 1254[5]	Aroclor 1260[5]	Percent Recovery[6]	Percent Lipid	PCB/ Lipid[7]
wf112427	1	3	2	23.00	5.00	0.959	0.151	0.536	0.272	69.64	5.64	17.01
wf112439	1	3	1	23.00	4.00	1.058	0.213	0.554	0.291	79.90	4.28	24.72
wf112440	1	3	2	23.50	4.00	0.434	0.069	0.231	0.134	87.58	5.87	7.39
wf112492	1	2	2	24.75	6.00	0.507	0.167	0.231	0.109	71.03	3.46	14.65
wf112433	1	2	1	24.75	5.25	1.222	0.475	0.566	0.182	82.75	18.38	7.34
wf112434	1	2	2	25.50	5.50	0.849	0.185	0.442	0.222	89.56	4.99	17.01
wf112488	1	2	2	25.75	6.25	1.038	0.264	0.537	0.237	67.68	6.88	15.09
wf112469	1	2	2	27.50	9.00	0.639	0.112	0.342	0.185	80.31	3.96	16.14
wf112484	1	2	2	27.75	8.00	0.740	0.149	0.388	0.203	84.04	6.05	12.23
wf112432	1	2	2	28.25	10.00	1.254	0.453	0.514	0.286	75.79	3.80	32.99
wf112462	1	2	2	29.00	8.50	1.292	0.337	0.630	0.325	105.69	4.67	27.67
wf112483	1	2	1	29.25	9.75	0.978	0.324	0.444	0.210	83.96	4.55	21.50
wf112491	1	2	1	30.00	11.00	1.286	0.259	0.657	0.370	77.28	3.44	37.39
wf112486	1	2	1	30.38	9.25	0.787	0.183	0.395	0.209	97.80	3.34	23.57
wf112477	1	2	2	30.75	11.00	0.973	0.127	0.541	0.305	98.59	2.49	39.09
wf112464	1	2	2	31.38	10.25	1.029	0.316	0.542	0.170	88.04	4.86	21.17
wf112485	1	2	1	31.50	11.50	0.920	0.207	0.468	0.246	74.58	2.89	31.85
wf112463	1	2	1	31.75	13.00	1.717	0.717	0.748	0.252	87.06	7.99	21.49
wf112428	1	2	1	32.00	12.00	0.672	0.185	0.322	0.166	84.45	4.07	16.57
wf112429	1	1	1	32.38	12.50	1.461	0.501	0.668	0.292	89.85	8.02	18.21
wf112487	1	1	1	32.75	12.75	1.306	0.203	0.758	0.344	78.66	8.39	15.56
wf112480	1	1	1	33.00	12.50	1.170	0.337	0.553	0.281	85.83	2.24	52.24
wf112467	1	1	1	33.00	15.00	1.166	0.267	0.558	0.341	68.71	3.41	34.20
wf112461	1	1	1	33.75	14.00	1.992	0.394	1.096	0.502	68.05	6.04	32.97
wf112479	1	1	2	34.25	14.00	0.995	0.274	0.471	0.250	86.33	4.00	24.87
wf112466	1	1	2	34.50	16.00	1.737	0.630	0.723	0.384	85.84	5.34	32.52
wf112465	1	1	2	35.38	15.25	0.810	0.207	0.375	0.228	73.02	2.60	31.16
wf115938	2	3	1	15.75	1.50	0.419	0.053	0.228	0.138	84.99	1.98	21.14
wf115981	2	3	2	16.25	1.50	0.305	0.000	0.168	0.137	88.69	0.82	37.23
wf115991	2	3	2	17.25	1.60	0.194	0.019	0.108	0.067	105.90	0.36	53.83
wf115949	2	3	1	17.75	2.00	0.189	0.018	0.104	0.067	117.79	0.85	22.23
wf115980	2	3	2	18.00	2.00	0.386	0.044	0.207	0.135	181.24	1.62	23.84
wf115934	2	3	2	18.25	1.95	0.465	0.024	0.269	0.173	108.09	1.89	24.64

Table A.3. (continued)

Fish	Date[2]	Size[3]	Sex[4]	Length (in.)	Weight (lb.)	Total PCB[5]	Aroclor 1248[5]	Aroclor 1254[5]	Aroclor 1260[5]	Percent Recovery[6]	Percent Lipid	PCB/ Lipid[7]
wf115948	2	3	1	19.00	2.50	0.258	0.046	0.136	0.076	87.34	1.06	24.47
wf115969	2	3	1	19.25	2.70	0.657	0.102	0.352	0.203	94.75	2.18	30.15
wf115987	2	3	2	19.50	2.70	0.420	0.000	0.265	0.155	144.21	2.28	18.41
wf115944	2	2	2	26.12	6.00	0.627	0.129	0.328	0.170	118.13	4.45	14.10
wf115994	2	2	2	26.50	6.50	0.786	0.308	0.309	0.169	93.08	4.14	19.00
wf115999	2	2	1	26.50	4.50	0.908	0.162	0.534	0.211	85.73	0.39	232.73
wf115972	2	2	2	28.12	9.14	1.110	0.290	0.517	0.304	105.37	2.97	37.39
wf115946	2	2	2	29.00	9.70	1.264	0.444	0.591	0.229	97.46	3.47	36.41
wf116000	2	2	2	29.25	9.00	1.125	0.244	0.522	0.359	122.17	2.79	40.32
wf115983	2	2	2	29.50	8.20	0.625	0.113	0.323	0.190	70.49	0.78	80.15
wf115995	2	2	2	30.50	8.80	0.862	0.186	0.396	0.280	102.22	2.62	32.91
wf115929	2	2	1	31.75	12.60	1.284	0.276	0.681	0.327	20.14	2.02	63.57
wf115988	2	1	2	32.50	11.60	1.505	0.386	0.754	0.365	59.09	3.43	43.87
wf112478	2	1	1	32.75	10.50	0.424	0.029	0.233	0.162	81.72	0.31	161.68
wf115940	2	1	1	33.00	13.40	1.334	0.399	0.603	0.331	137.67	6.10	21.87
wf112474	2	1	1	33.50	15.00	1.070	0.223	0.609	0.237	88.87	1.73	61.84
wf115927	2	1	2	33.50	17.00	1.210	0.386	0.560	0.264	112.96	2.79	43.36
wf115926	2	1	1	33.50	15.25	1.388	0.216	0.782	0.390	79.13	2.85	48.68
wf115996	2	1	1	34.00	13.00	1.210	0.363	0.509	0.338	71.55	2.15	56.28
wf115985	2	1	1	34.25	13.70	1.303	0.319	0.659	0.325	16.52	2.09	62.36
wf115986	2	1	1	34.50	14.50	0.610	0.168	0.258	0.184	70.09	0.90	67.74
wf102399	3	3	2	19.25	2.50	0.152	0.031	0.081	0.040	79.49	2.86	5.33
wf102397	3	3	2	19.50	2.50	0.140	0.017	0.084	0.039	82.07	0.10	137.31
wf102376	3	3	2	20.00	4.25	0.802	0.069	0.443	0.290	84.30	2.94	27.28
wf102385	3	3	2	21.25	3.50	0.493	0.000	0.297	0.195	85.66	0.80	61.27
wf112417	3	3	2	21.50	2.75	0.677	0.138	0.319	0.220	72.89	1.33	50.91
wf111955	3	3	1	22.25	4.00	0.465	0.086	0.273	0.106	77.54	6.03	7.71
wf102400	3	3	2	22.50	4.00	0.436	0.030	0.247	0.159	85.30	2.76	15.80
wf115925	3	3	2	23.25	4.75	0.845	0.000	0.503	0.342	121.53	2.06	41.01
wf115997	3	3	2	23.50	4.25	0.728	0.110	0.364	0.254	132.73	1.80	40.45
wf115974	3	2	2	24.25	5.25	1.192	0.137	0.605	0.451	125.44	1.54	77.40
wf115957	3	2	2	25.50	6.25	1.197	0.159	0.652	0.386	73.97	3.17	37.75
wf111929	3	2	2	27.75	9.00	0.605	0.102	0.335	0.168	81.70	4.92	12.30
wf115943	3	2	2	29.00	8.50	0.932	0.206	0.505	0.221	65.33	1.05	88.78

Table A.3. (continued)

Fish	Date[2]	Size[3]	Sex[4]	Length (in.)	Weight (lb.)	Total PCB[5]	Aroclor 1248[5]	Aroclor 1254[5]	Aroclor 1260[5]	Percent Recovery[6]	Percent Lipid	PCB/ Lipid[7]
wf112415	3	2	1	29.00	9.00	1.715	0.224	0.967	0.524	91.94	6.71	25.56
wf115958	3	2	2	30.00	7.75	0.898	0.134	0.517	0.247	112.69	0.74	121.31
wf115970	3	2	1	30.25	7.50	0.756	0.105	0.376	0.275	102.61	0.20	377.81
wf115956	3	2	2	30.50	8.00	0.976	0.180	0.545	0.251	57.44	0.55	177.50
wf115953	3	2	2	31.50	10.50	1.288	0.196	0.706	0.386	105.43	1.21	106.46
wf115930	3	1	2	32.00	9.00	0.939	0.240	0.433	0.266	100.96	1.07	87.79
wf115998	3	1	1	33.00	12.50	0.741	0.054	0.481	0.207	106.82	2.10	35.29
wf115964	3	1	1	34.00	13.50	0.938	0.149	0.530	0.259	137.74	0.42	223.41
wf115936	3	1	2	34.00	11.25	0.834	0.161	0.457	0.215	98.58	0.14	595.44
wf115973	3	1	1	34.25	15.50	2.095	0.225	1.265	0.604	74.34	0.59	355.02
wf115962	3	1	1	34.38	13.50	0.579	0.113	0.302	0.164	105.82	0.45	128.63
wf115942	3	1	1	34.50	11.25	1.318	0.360	0.692	0.267	87.89	0.34	387.67
wf115951	3	1	2	37.00	17.50	1.905	0.451	1.005	0.449	63.90	0.79	241.16
wf115952	3	1	2	37.75	15.50	1.306	0.122	0.813	0.371	122.49	1.43	91.34
Averages:				28.15	8.79	0.937	0.203	0.483	0.251	90.17	3.07	68.94
Std.Dev.:				5.61	4.44	0.428	0.147	0.225	0.107	24.09	2.66	98.08

[1]Mean values are listed for fillets analyzed in duplicate. See Table A.2 for more information on duplicate analyses.

[2]Values for Date signify the following: 1=May, 2=July, 3=September.

[3]Values for Size indicate the following: 1= <24", 2= >24" and <32", 3= >32".

[4]Values for Sex indicate the following: 1= female, 2=male.

[5]Concentrations are in ug/g wet weight in trimmed fillets.

[6]Concentrations listed in this table have already been corrected for recovery.

[7]Concentrations are in ug/g lipid in trimmed fillets.

Table A.4. Injection and Quantitation Precision Using an Aroclor Mix Standard.

Injection	1248 (ppm)	1254 (ppm)	1260 (ppm)	Total (ppm)
A	2.09	4.46	2.14	8.69
B	2.08	4.42	2.21	8.72
C	2.01	4.39	2.12	8.52
D	2.21	4.52	2.20	8.93
E	2.09	4.61	2.24	8.94
Mean:	2.10	4.48	2.18	8.76
Std. dev:	0.07	0.09	0.05	0.18
C.V.:	3.50	1.91	2.38	2.04
Nominal Concentration:	1.93	4.55	2.01	8.49
Percent Difference[1]:	8.66	-1.50	8.49	3.17

[1]Percent Difference = $\frac{\text{(Obser. Mean - Nominal Conc.)}}{\text{Nominal Concentration}} * 100\%$

Table A.5. Quantitation of Aroclor Mix Standards.

Mix	Aroclor	Nominal Concentration	Measured Concentration	Percent Difference
Mix 2	1248	1.93	2.10	8.66
	1254	4.55	4.48	-1.50
	1260	2.01	2.18	8.49
	Total	8.49	8.76	3.17
Mix 3	1248	3.19	3.82	19.75
	1254	6.58	6.54	-0.64
	1260	3.24	3.79	16.89
	Total	13.03	14.15	8.56
Mix 4	1248	1.47	1.28	-12.66
	1254	3.89	3.01	-22.69
	1260	2.01	1.64	-18.58
	Total	7.37	5.93	-19.57
Mix 5	1248	0.21	0.23	8.45
	1254	1.11	1.17	5.72
	1260	0.68	0.82	20.66
	Total	2.02	2.22	10.09
Mix 6	1248	1.62	1.78	9.81
	1254	1.87	1.74	-6.98
	1260	3.87	3.86	-0.30
	Total	7.36	7.38	0.23
Mix 7	1248	6.34	5.79	-8.69
	1254	7.27	5.98	-17.76
	1260	3.87	3.73	-3.51
	Total	17.48	15.50	-11.32

Summary:

	Percent Difference			
	1248	1254	1260	Total
Mean:	4.22	-7.31	3.94	-1.47
SD:	12.34	10.90	14.48	11.69

Table A.6. Accuracy and Precision of Lipid Determination for Safflower Oil.

Equivalent Percent Lipid	Safflower Oil Concentration[1]	Observed Concentration	Percent Recovery	Mean	Std. Dev.	CV
0.1%	1.275	1.125	88.2	117.6	35.3	30.1
		2.000	156.9			
		1.375	107.8			
2.0%	25.013	18.875	75.5	72.3	3.3	4.5
		17.250	69.0			
		18.125	72.5			
5.0%	62.431	46.750	74.9	72.8	2.9	4.0
		43.375	69.5			
		46.250	74.1			
8.0%	100.094	75.500	75.4	72.2	2.9	4.0
		70.125	70.1			
		71.125	71.1			
25.0%	312.477	227.250	72.7	72.2	0.5	0.7
		224.000	71.7			
		225.875	72.3			

[1]This concentration has been calculated to contain the same mass of oil in 80 ul as is contained in 80 ul of an extract from a 10 g sample which contains lipid at the listed equivalent percent. Concentration is in mg/ml.

Table A.7. Precision and Accuracy of Lipid Determination for Safflower Oil Through $Na_2SO_4/MeCl_2$ Extraction.

Nominal Percent Lipid	Observed Percent Lipid	Percent Recovery	Mean	Std. Dev.	CV
4.2	2.75	65.5	63.8	1.7	2.6
4.2	2.72	64.8			
4.2	2.64	62.9			
4.2	2.60	61.9			