

12-1991

Residues of Mirex and Photomirex in Eggs and Fillets of Lake Ontario Coho and Chinook Salmon

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RESIDUES OF MIREX AND PHOTOMIREX IN EGGS
AND FILLETS OF LAKE ONTARIO COHO
AND CHINOOK SALMON

A Thesis

Presented to the Faculty of the Department of Biological Sciences
of the State University of New York College at Brockport
in Partial Fulfillment for the Degree of
Master of Science

by
Michael D. Murray
December, 1991

ABSTRACT: Coho (Oncorhynchus kisutch) and chinook (O. tshawytscha) salmon collected from Lake Ontario during the fall of 1986 were analyzed by gas chromatography for mirex and photomirex residues. Mirex in fillet tissue (n=24) ranged from 0.015 to 0.41 mg/kg (mean = 0.19 mg/kg). Photomirex concentrations ranged from nondetectable (in two samples) to 0.18 mg/kg (mean = 0.080 mg/kg). Mirex in egg samples (n = 5) ranged from nondetectable to 0.21 mg/kg (mean = 0.10 mg/kg), while photomirex ranged from nondetectable to 0.38 mg/kg (mean = 0.11 mg/kg). Analysis of variance revealed no statistical difference ($P > 0.05$) between the value of 0.19 mg/kg reported here and either the 0.22 mg/kg reported by Insalaco et al. (1982) for salmon collected in 1977, or the mean residue of 0.18 mg/kg observed by Makarewicz (1985 unpublished data) for salmon collected in 1982. However, analysis of covariance revealed a decrease of 26% ($P < 0.01$) in mirex levels in 1982 and 1986 relative to 1977 levels after removal of the confounding variable weight. The data suggest the need for more rigorous statistical analyses than are typically applied in studies attempting to elucidate trends in mirex contamination in Lake Ontario.

ACKNOWLEDGMENTS

I would like to thank my major advisor, Dr. J. Makarewicz, for his various contributions toward the completion of this study, as well as toward my academic career at Brockport. No less important than the instruction and guidance he provided along the way was the encouragement he offered during moments of self-doubt. His counsel was ever a quickening motivational influence, and his patience well appreciated.

I am also grateful to Dr. J. Haynes for serving on my graduate committee, and for lending his expertise and advice during the progression of this project. I also wish to thank Dr. J. Buttner for serving as an advisor on the committee. His insightful comments and suggestions were invaluable in shaping this study.

I would also like to thank Mr. Ted Lewis for sharing his knowledge and experience with me during the course of my research. I wish to express my gratitude as well to Mr. Steve Iveson, Mr. Scott Seibold, Mr. Glen Gerber and Dr. Haynes for their assistance in the sampling portion of this study.

I wish also to thank my family for their loving support, especially my wife, whose faith in me exceeded my own.

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INTRODUCTION

Levels and distribution patterns of mirex in Lake Ontario have been closely monitored since Kaiser (1974) first detected its residues in fish samples taken from the Bay of Quinte, Ontario, Canada. In 1976, the Ontario Ministry of the Environment (OME) reported mirex contamination in nearly all fish species in Lake Ontario. Approximately one-half of the species monitored exceeded the United States Food and Drug Administration (U.S. FDA) consumption guideline of 0.1 ug/g mirex (Kaiser 1978). Subsequent sediment analyses by the International Joint Commission (IJC) and their Reference Group on Pollution from Land Use Activities (PLUARG) revealed a pattern of two discernible zones of mirex contamination: one extending eastward from the Niagara River (the Niagara Anomaly), the other showing a similar eastward trend off the Oswego River (the Oswego Anomaly) (Kaiser 1978). These zones have been attributed to two point sources: the Hooker Chemical Corporation on the Niagara River, and the Armstrong Cork Company on the Oswego River, respectively (Holdrinet et al. 1978).

Mirex, a chlorocarbon insecticide, is characterized by its ecological stability; it resists both environmental and metabolic degradation, especially under aerobic conditions (World Health Organization 1984). Breakdown occurs mainly via photolytic dechlorination, resulting in several mono - and dihydro derivatives, including the considerably more toxic 8- monohydromirex, or photomirex (Norstrom et al. 1980). Mirex bioaccumulates and

biomagnifies in aquatic systems, with concentrations dependent upon trophic relationships (Environmental Studies Board 1978). Since it is lipophilic, mirex is readily stored in fatty tissues (Scrudato and DelPrete 1982), particularly in the liver and brain, where it shows a high potential for chronic toxicity (Ivie et al. 1974).

Acute sensitivity of crustaceans to mirex, particularly among juvenile stages, has been reported in a number of studies (Bookhout et al. 1972; Ludke et al. 1971; Markin et al. 1972). Lue et al. (1978) reported biomagnification and sublethal toxicity of mirex in hydra after ingestion of zooplankton, which had concentrated the contaminant from suspended particulates and from solution.

While data on mirex toxicity to fish are limited, it appears to be much less toxic than to crustaceans (Stickel et al. 1973). Mirex also does not appear to be acutely toxic to birds, although moderate chronic toxicity and delayed mortality in grackles has been reported (Stickel et al. 1973).

Daly et al. (1989) reported increased reactivity to adverse events in rats fed Lake Ontario salmon, relative to rats fed Pacific Ocean salmon or a regular chow diet. While mirex could not be singled out as the causative agent among other compounds present in Lake Ontario salmon, the exposure method used in this study represents a view held by a number of researchers; that is, toxicological effects of chemicals may best be evaluated in the combinations that occur naturally, and that have undergone envir-

onmental modifications.

There have been no reports of toxicological effects of mirex in man, but its carcinogenicity in rats (Ulland et al. 1977) and in mice (Innes et al. 1969) indicate its potential hazard.

The persistence and lipophilicity of mirex hold implications beyond the immediate food web. According to Kaiser (1978), migrating gulls contaminated within the Lake Ontario system probably contribute significantly to mirex residues in the biota of the other Great Lakes, although levels remain low overall outside the Lake Ontario system.

While peak influx of mirex from the Niagara and Oswego rivers occurred during the 1960s (Durham and Oliver 1983; Whittle and Fitzsimons 1983), both tributaries remain significant sources of mirex to Lake Ontario due to leaching from storage sites (Halfon 1987; Scudato and DelPrete 1982; Thomas 1983). In addition, it has been reported that salmonids have contaminated Lake Ontario tributaries with mirex via their spawning migrations (Lewis and Makarewicz 1988; Scudato and McDowell 1989). According to these studies, as salmonid carcasses and eggs decompose, mirex is made available to tributaries utilized for spawning, both via direct release as well as through ingestion and subsequent transport by stream organisms. Recycling of mirex back to Lake Ontario must also be considered; Lewis and Makarewicz (1988) estimated this potential as 26 gm/yr. Halfon (1984) reported that even with cessation of mirex loadings to Lake Ontario, exposure of biota would remain significant for the foreseeable future; coverage of

contaminated by "clean" sediments could require up to 600 years.

Additional data on photomirex, the major degradation product of mirex, is also needed, especially in light of its greater toxicity. An apparent rise in photomirex to mirex ratios in Lake Ontario biota around 1980 was noted by several authors (Insalaco et al. 1980; NYSDEC 1981).

Reports of the New York State Department of Environmental Conservation (NYSDEC) suggested a peak in mirex levels during the late 1970s, followed by a decreasing trend in subsequent years (Norstrom & Hallet 1980; Armstrong and Sloan 1980). If a trend exists, however, the determination of its significance requires additional data as well as a somewhat more rigorous statistical approach than is available in the NYSDEC reports. Insalaco et al. (1982) reported fillet tissue concentrations of mirex in Lake Ontario coho and chinook salmon. Based on these data, and those of the present study, I report here a decline in mirex levels in Lake Ontario salmon since 1977.

METHODS

Coho (Oncorhynchus kisutch) and chinook (O. tshawytscha) salmon were collected by electroshocking from Sandy Creek and Eighteen-Mile Creek, tributaries of Lake Ontario, during the autumn 1986 spawning run. A total of 24 fish were sampled according to the following design: 12 coho, 4 each from weight intervals < 1.8, 1.8-3.6, and > 3.6 kg (corresponding to intervals of < 4, 4-8, and > 8 lbs, respectively); 12 chinook, 4 each from intervals < 4.5; 4.5-9, and > 9 kg (corresponding to intervals of < 10, 10-20, and > 20 lbs, respectively). Total length (cm) and weight (kg) were recorded for each fish before packing in ice and subsequent transport to the laboratory. Fish age was determined by scale analysis (Nielsen and Johnson 1983). The sex of each fish was recorded. A standard fillet from each fish and eggs from each female were taken for analysis. Each sample was wrapped in aluminum foil and frozen immediately, until analysis could be performed.

Tissue samples were ground in a food processor and frozen until analysis could be performed. After thawing, a 5-gram aliquot of each egg and tissue sample was mixed with 20 grams of anhydrous sodium sulfate and homogenized in a Virtis tissue homogenizer. The sample was then extracted overnight with 75 ml of methylene chloride/hexane (1:4 v/v) in a Soxhlet extraction apparatus for a minimum of 200 cycles. Excess lipid was removed using a standard Florisil column. Interfering PCBs were removed by

nitration after Insalaco et al. (1980), followed by a final mini-column Florisil cleanup prior to gas chromatographic analysis. Sample lipid content was determined by Soxhlet extraction followed by evaporation under nitrogen to constant weight, and expressed as percent extractable residue.

Mirex and photomirex residues were analyzed on a Hewlett-Packard 5750B Research gas chromatograph equipped with a high-temperature Ni-63 pulsed electron capture detector. Peaks were integrated using polar planimetry. Chromatographic runs were isothermal; temperatures at the injection port, column oven and detector were 225, 195 and 225 degrees C, respectively. The chromatographic column was a 1.8m X 2mm glass column packed with 3.8% UCW-982 on 80/100 Chromosorb W.H.P. The carrier gas was argon/methane (95%/5%) with a flow rate of 50 ml/min. Mirex/photomirex standards (0.1 mg/ml) were analyzed with each set of samples. Routine quality control/quality assurance procedures included analysis of reagent blanks, replicate sample analyses (Appendix I), internal standard additions (Appendix II), and spiked recovery analyses. All reagents were pesticide grade.

RESULTS

In order to assess the accuracy and precision of the analytical techniques used during this study, replicate analyses (n=4) were performed on a fillet tissue sample previously analyzed in our laboratory. No significant difference ($P > 0.05$) was found between the two analyses for either mirex or photomirex concentrations (Appendix I). Spiked recovery analyses were performed after Insalaco et al. (1980); efficiencies exceeded 90% for both mirex and photomirex. To validate further the presence of mirex and photomirex in the samples, internal standard additions containing each compound were given to three samples in which both mirex and photomirex residues had been detected. If the peaks observed in each sample were indeed mirex and photomirex, then the integrity of the peaks should not change after the standard additions, and recoveries should be quantitative. Resultant recoveries, as shown in Appendix II, provide confirmatory evidence of the presence of mirex and photomirex in the samples. The slight but consistent pattern of "excess" recovery for each compound may reflect a small increase in response in the presence of sample matrix; the standard solution used for quantitation was injected prior to the recovery samples themselves, i.e., before any coating of the column by the sample matrix had occurred.

Representative standard and sample chromatograms are shown in Figures 1 and 2, respectively.

Since analysis of variance revealed no significant differences ($P > 0.05$) in mirex or photomirex concentrations

between species (Table 1), the data for all fish were pooled for statistical analysis. The mean tissue concentrations for mirex and photomirex were 0.19 and 0.080 mg/kg, respectively. Mirex was detected in all samples and ranged from 0.015 to 0.41 mg/kg. Photomirex ranged from undetected (in two samples) to 0.18 mg/kg. Only five egg samples were available for analysis among the fish sampled; mean values for mirex and photomirex were 0.10 and 0.11 mg/kg, respectively (Table 1).

Fish weights ranged from 0.34 to 11.1 kg (mean = 4.25 kg); the values for length ranged from 37.3 to 100.3 cm (mean = 69.0 cm). Percent lipid ranged from 0.35 to 9.05 (mean = 3.46%). These data are presented in Appendix III.

Fish were assigned ages of 1+, 2+, or 3+, based on scale analyses. Of the twenty-four analyzed, eight, seven and nine, respectively, fell into each age class (Appendix III).

The variation in the mirex residue data was evaluated via multiple linear regression analysis using the RS/1 statistical package (BBN Software Products Corp.). The regression model was optimized through stepwise construction, allowing comparison of the effects of the independent variables in terms of F-values, coefficients, etc. The variables assessed with the model were species, age, length, weight and % lipid; the associated data are shown in Appendix III. The best model was provided by a simple linear regression of mirex concentration vs. fish weight, which explained 62% of the variation in the data ($P < 0.01$). The other variables, whether considered singly or in the various possible

combinations, failed to improve the model due either to the lack of a significant effect (e.g. % lipid), or the high degree of interrelation with certain variables (e.g. age and weight). Regression data for both mirex and photomirex are shown in Table 2, and regression lines in Figures 3 and 4, respectively.

DISCUSSION

Historical Trends

Insalaco et al. (1982) reported a mean mirex concentration of 0.22 mg/kg in fillet tissues for coho and chinook salmon in Lake Ontario. Although fish were collected during both autumn and spring (1977-78) seasons, the respective mean mirex concentrations, with sexes combined, were identical. Similarly, unpublished data collected from 1982 salmon averaged 0.18 mg/kg (Makarewicz, personal communication). These concentration means are comparable to that of 0.19 mg/kg obtained in the present study. An analysis of variance revealed no significant difference ($P > 0.05$) between 1977, 1982 and 1986 mirex concentrations in fillet tissues. My results thus concur with NYSDEC reports of no change in mirex concentrations in salmon since the late 1970's. However, these comparisons are simply the mean fillet concentrations for the respective years, and are not adjusted for any differences in variables which may contribute to the variation in the data. An analysis of covariance (ANCOVA) was performed on the mirex concentration data reported for 1977 (Insalaco et al. 1982), 1982, (Makarewicz personal communication) and in the present study, with means adjusted for the covariate weight. A 26% decline in the mean weight-adjusted mirex concentration from 1977 to 1982 and 1986 was revealed by ANCOVA (Tables 3 and 4). There was no significant difference in the mean weight-adjusted mirex concentrations from 1982 to 1986 (Table 5, Figure 5).

A lesser (20%), but significant decline (20%) from 1977 to 1982 was also observed using mirex body burden as the dependent variable (Table 4, Figure 6). A decrease of 19% from 1977 to 1986, however, was not significant (Table 3). These results reflect the trend of fish weight increase from 1977 to 1986 (Table 6), and further indicate the need to correct for fish weight variability between populations when monitoring mirex residues.

Lake Ontario Photomirex/Mirex Ratios

Reports of increasing photomirex to mirex ratios around 1980, such as those cited earlier, are perhaps not surprising in light of concurrent reports of decreasing mirex levels (Norstrom and Hallett 1980; Armstrong and Sloan 1980), assuming availability of mirex for photolytic degradation during that period. However, Norstrom and Hallett (1980) reported a stable photomirex/mirex ratio (0.3 to 0.4) in Lake Ontario biota for that period, and suggested that sequestration of mirex within the food web resulted in a reduced rate of photolytic breakdown. Kaiser (1978) considered it unlikely that photodecomposition into photomirex occurred once mirex entered the lake system. The process has been observed to accelerate, however, in the presence of organic matter such as humic acids (Mudambi and Hassett 1986) and aliphatic amines (Alley et al. 1974).

The ratio of photomirex to mirex apparently is relatively constant within species (Kaiser 1978), as well as among species for a given system (Norstrom et al. 1980; McDowell et al. 1986). While Lake Ontario values for this parameter fluctuate somewhat

from year to year, current and recent reports indicate a range of approximately 0.4 to 0.6 (McDowell et al. 1986; Sloan 1987), somewhat higher than the overall range for 1976 values. Sloan (1987) reported a mean ratio of 0.43 for 1984, compared to that of 0.38 calculated for seven fish species in 1983. In the present study the mean photomirex/mirex ratio for all fish was 0.42, a value in close agreement with the above. It seems apparent, therefore, that the photomirex/mirex ratio has stabilized since the initial increase around the 1980 period.

Correlation of Mirex and Lipid Levels

The depletion of lipid reserves that occurs in spawning salmonids apparently does not reduce lipophilic contaminant levels in the fish; Lieb et al. (1974) reported a doubling of PCB concentrations in rainbow trout lipid following starvation-induced depletion of lipid levels from 8.52% to 4.65% of body weight. The overall tissue concentrations and body burdens of PCBs, however, remained essentially the same. Gruger et al. (1975) reported a similar failure of fat depletion to increase elimination of PCBs in rainbow trout. Mirex residues in spawning salmon also appear to be reconcentrated in the remaining lipid as a result of fat depletion (Skinner personal communication).

This "reconcentration effect" may be the major factor contributing to the lack of correlation between percent lipid and mirex concentrations observed in this study. A positive relationship between percent lipid, fish size, and halocarbon residues has been reported by a number of authors (Norstrom 1978;

Insalaco et al. 1982; Monod and Kech 1982). During the spawning run, however, the relationship between size and fat content apparently changes, due to fat depletion in the more mature, spawning fish, as was seen in this study. For example, the mean lipid content for the third age group in chinook (2.11%) represents a decrease of 59% from that of the second age group (5.20%). In coho, the second age group averaged 4.13% lipid, whereas the mean for the 3+ age group was only 2.70%, or 35% lower. Sloan (1987) reported a mean lipid content of 2.02% for the 2+ age group of spawning chinook salmon, compared to 1.32% for the 3+ age group (35% lower).

Residues in Eggs

Interpretation of the egg residue data from the present study, however, is limited by both the small sample size (n=5), and the variability as indicated by the relatively large standard deviations (Table 1). Wide variation in organochlorine residues in egg samples was also observed by Sloan (1987) and Niimi (1983). According to the latter study, both percent lipid in the fish as well as the percent of total lipid deposited in the eggs were significantly correlated with contaminant transfer to eggs. No pattern was discernible in the present study, however. Similarly, Scrudato and McDowell (1989) found no significant correlation between egg lipid content and mirex concentration in standard fillets of spawning salmonids in Lake Ontario. The effect of spawning kinetics on contaminant tissue concentrations depends upon relative levels between eggs and tissues (Niimi 1983).

In this study, the means for total mirex (mirex + photomirex) in eggs and tissues, respectively, were virtually identical. Therefore, tissue concentrations should not be expected to change significantly upon egg deposition, since residue losses would be proportional to weight losses. Scudato and McDowell (1989) also reported no significant differences in mirex concentrations between standard fillets and eggs in migrating salmonids in Lake Ontario.

The disproportionate male to female ratio may have been an effect of the season, as well as method, of sample collection undertaken in this study. It is possible that the more aggressively territorial spawning males were less likely than females to avoid the electroshocking boat; territoriality was suggested (Nielsen and Johnson 1983) to induce such behavior in fish. Such factors may be worth consideration for future contaminant monitoring efforts, particularly those involving the analysis of egg samples.

While mirex continues to be supplied to the lake ecosystem, overall discharge to the lake has been significantly reduced since peak discharges during the 1960s. By 1981, for example, mirex loadings from the Niagara River had dropped to approximately 5% of 1960-62 values (Lewis and Makarewicz 1988).

The fundamental reference point for continuing mirex monitoring efforts by both Canadian and U.S. laboratories is the FDA action level of 0.1 ug/g for Lake Ontario fish (Kaiser 1978). The data reported here show that levels are still well in excess of

this guideline, with no significant change in mirex concentrations since 1977.

The covariate analysis, however, allows somewhat more insight into mirex dynamics within the lake ecosystem. Removal of the confounding variable weight revealed a significant ($P < 0.01$) decline of the contaminant in salmon from 1977 to 1982, a change too subtle to be detected via direct comparison of concentration means from the respective years.

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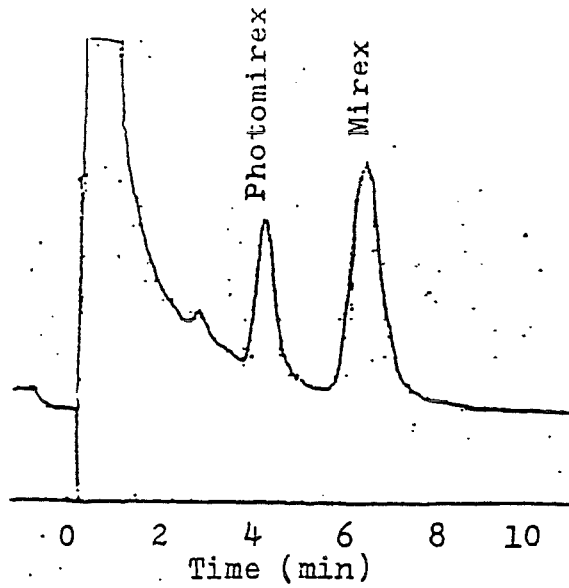


Figure 1. Representative chromatogram of standard mixture. Chromatographic conditions are given in text.

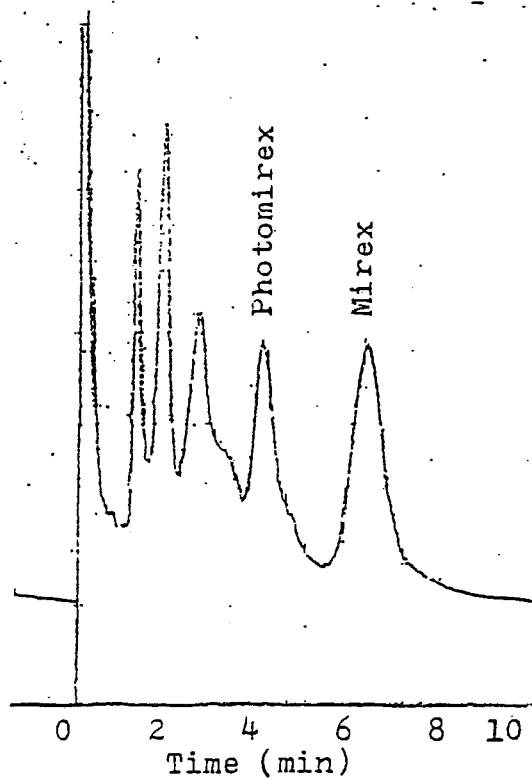


Figure 2. Representative chromatogram of sample extract. Chromatographic conditions are given in text.

Table 1. Means and ranges for contaminant concentration, weight, length and percent lipid. Numbers in parentheses are standard errors.

| | Mirex Conc. (mg/kg) | Photomirex Conc. (mg/kg) | Weight (kg) | Length (cm) | % Lipid |
|----------------------|------------------------|-----------------------------|----------------|----------------|-------------|
| Coho (n=12) | 0.16 (0.024) | 0.071 (0.014) | 2.7 (0.46) | 63.3 (4.19) | 3.2 (0.46) |
| Range | 0.015-0.32 | *ND-0.18 | 0.34-5.3 | 37.3-80.0 | 1.4-6.5 |
| Chinook (n=12) | 0.22 (0.032) | 0.088 (0.013) | 5.9 (1.03) | 75.1 (5.28) | 3.7 (0.72) |
| Range | 0.067-0.41 | 0.031-0.18 | 1.8-11.1 | 52.8-100.3 | 0.35-9.1 |
| Egg samples (n=5) | 0.10 (0.034) | 0.11 (0.063) | N/A | N/A | 11.0 (0.89) |
| Range | *ND-0.21 | *ND-0.38 | N/A | N/A | 7.3-13.4 |

* Not detected.

Table 2. Results of linear regression of contaminant concentration (dependent variable) vs. fish weight (independent variable); n=24.

| | Correlation Coefficient (r) | Coefficient of Determination (r-squared) | F-Value | Predictive Equation |
|------------|-----------------------------------|--|---------|------------------------|
| Mirex | 0.78 | 0.62 | 35.29** | 0.026(Weight) + 0.076 |
| Photomirex | 0.61 | 0.37 | 13.17** | 0.0092(Weight) + 0.039 |

** Significant at P < 0.01.

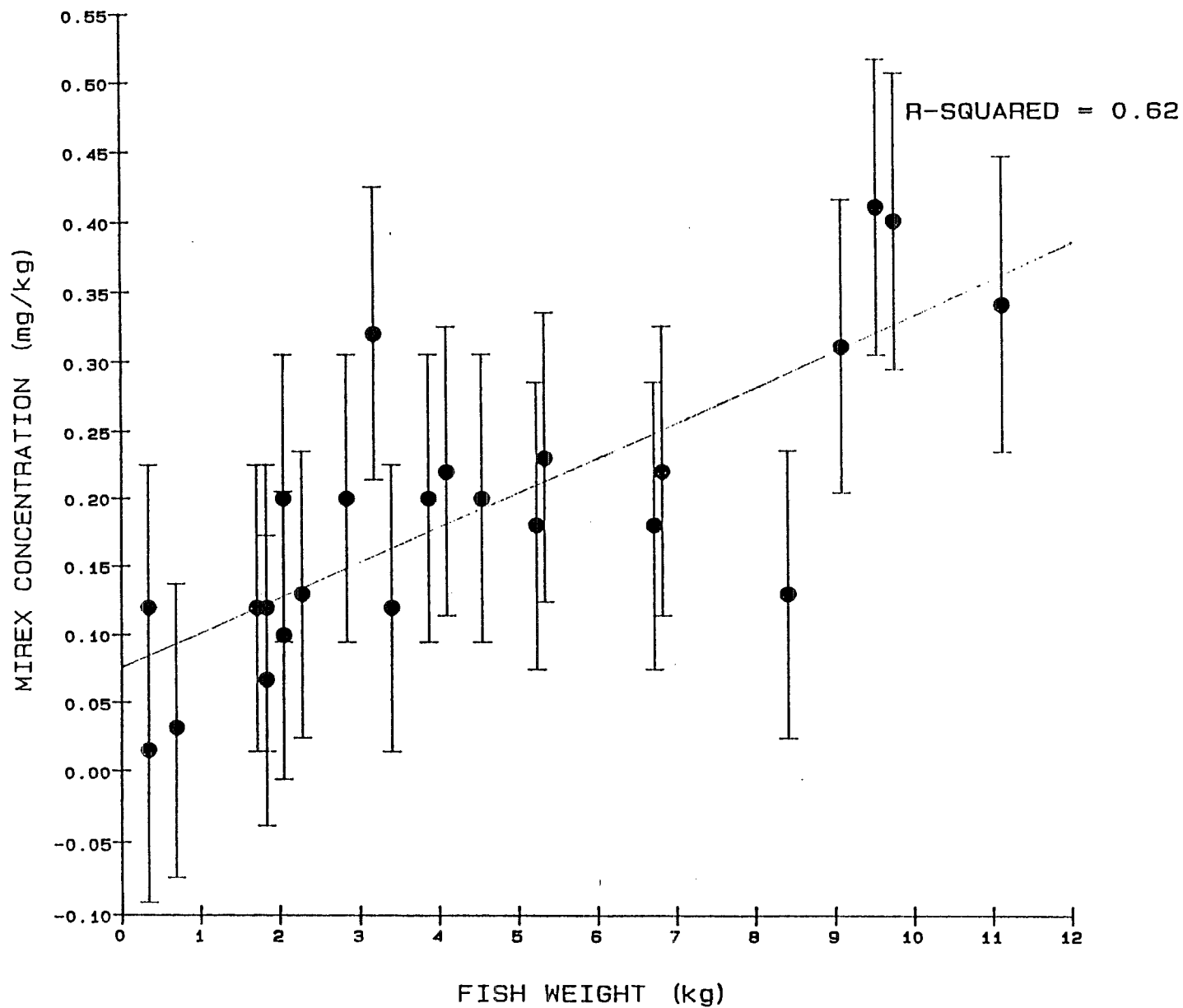


Figure 3. Regression of mirex concentration and fish weight for 1986 collections. Error bars for individual data points are shown.

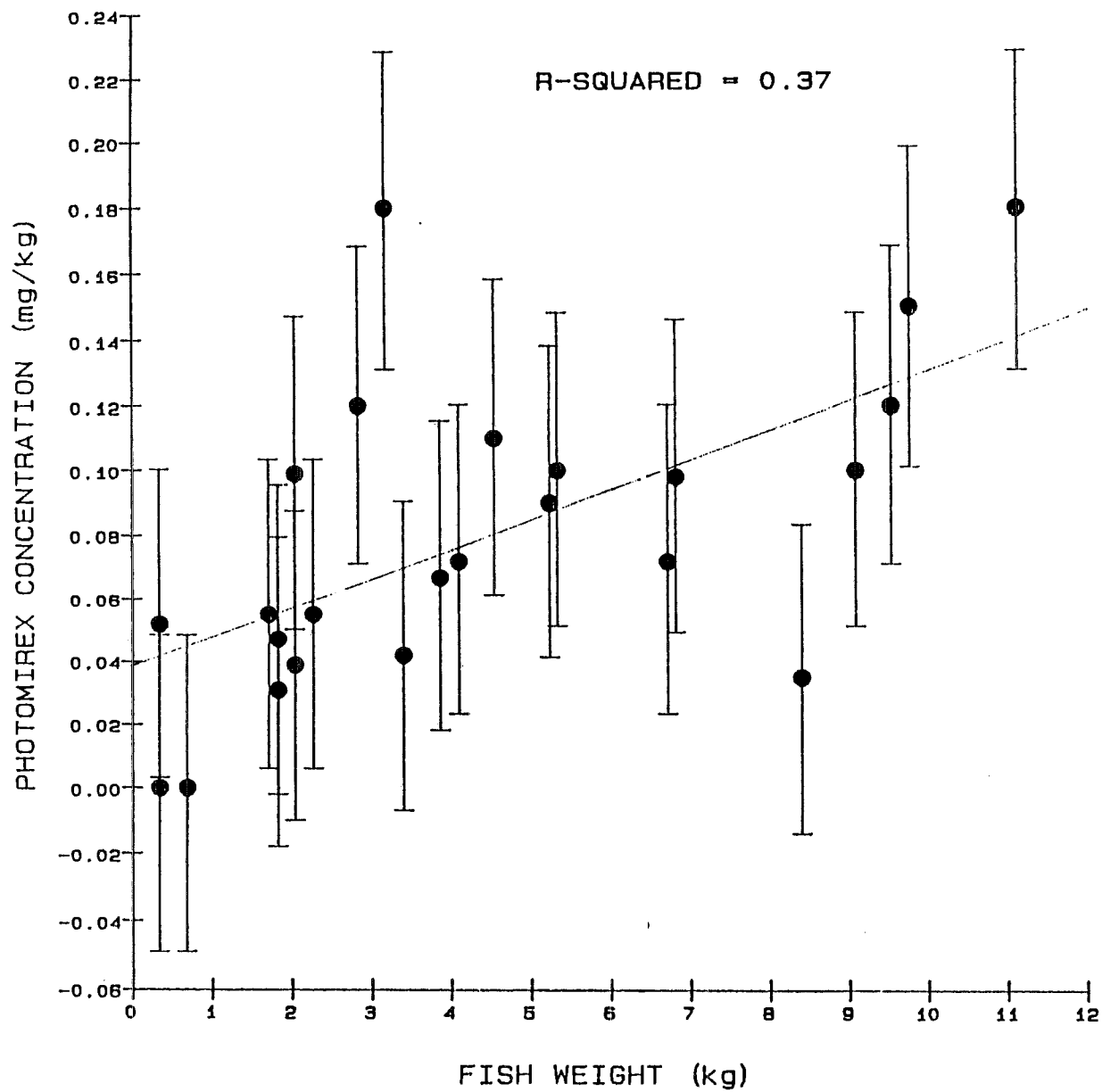


Figure 4. Regression of photomirex concentration and fish weight for 1986 collections. Error bars for individual data points are shown.

Table 3. Results of analysis of covariance of mirex residue data for 1977 and 1986 analyses. Slopes compared are those of regression lines for respective years of the relationship between (a) fish weight and mirex concentration (mg/kg) and (b) fish weight and mirex body burden (mg).

| | 1977 (n=24) | 1986 (n=23) | F-Value | Significance Level |
|--------------------------------|----------------|----------------|---------|-----------------------|
| Adjusted mean (mg/kg mirex) | 0.24 | 0.17 | 13.52 | 0.00063 |
| Slope | 0.0287 | 0.0256 | 0.200 | 0.66 (NS) |
| Adjusted mean (mg mirex) | 1.08 | 0.88 | 3.01 | 0.090 (NS) |
| Slope | 0.3568 | 0.3508 | 0.019 | 0.89 (NS) |

NS = Not Significant

Table 4. Results of analysis of covariance of mirex residue data for 1977 and 1982 analyses. Slopes compared are those of regression lines for respective years of the relationship between (a) fish weight and mirex concentration (mg/kg) and (b) fish weight and mirex body burden (mg).

| | 1977 (n=24) | 1982 (n=24) | F-Value | Significance Level |
|--------------------------------|----------------|----------------|---------|-----------------------|
| Adjusted mean (mg/kg mirex) | 0.23 | 0.17 | 11.70 | 0.0013 |
| Slope | 0.0287 | 0.0271 | 0.051 | 0.82 (NS) |
| Adjusted mean (mg mirex) | 0.94 | 0.75 | 6.25 | 0.016 |
| Slope | 0.3568 | 0.3205 | 1.21 | 0.28 (NS) |

NS = Not significant

Table 5. Results of analysis of covariance of mirex residue data for 1982 and 1986 analyses. Slopes compared are those of regression lines for respective years of the relationship between (a) fish weight and mirex concentration (mg/kg) and (b) fish weight and mirex body burden (mg).

| | 1982 (n=24) | 1986 (n=23) | F-Value | Significance Level |
|--------------------------------|----------------|----------------|---------|-----------------------|
| Adjusted mean (mg/kg mirex) | 0.19 | 0.18 | 0.300 | 0.59 (NS) |
| Slope | 0.0271 | 0.0256 | 0.047 | 0.83 (NS) |
| Adjusted mean (mg mirex) | 0.96 | 0.97 | 0.013 | 0.91 (NS) |
| Slope | 0.3205 | 0.3508 | 0.49 | 0.49 (NS) |

NS = Not Significant

Table 6. Results of analysis of variance of fish weight data for 1977, 1982 and 1986 collections. Numbers in parentheses are standard errors.

| | 1977 (n=24) | 1982 (n=24) | 1986 (n=24) | F-value | Significance Level |
|---------------------|----------------|----------------|----------------|---------|-----------------------|
| Mean weight (kg) | 3.20 (0.47) | 3.69 (0.52) | 4.45 (0.66) | 1.30 | 0.28 (NS) |

NS = Not Significant

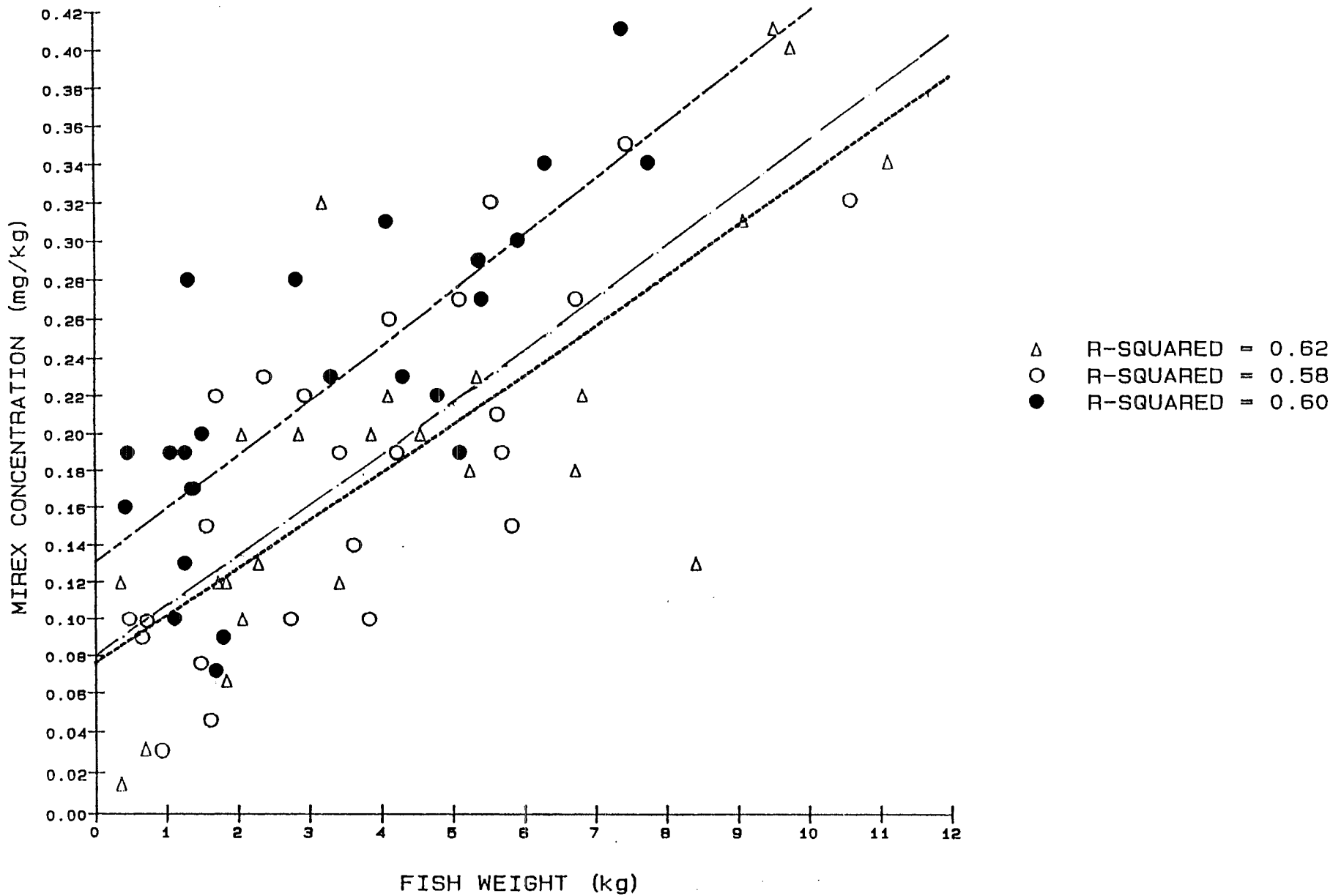


Figure 5. Comparison of the regression lines of mirex concentration and fish weight for 1977, 1982 and 1986 collections.

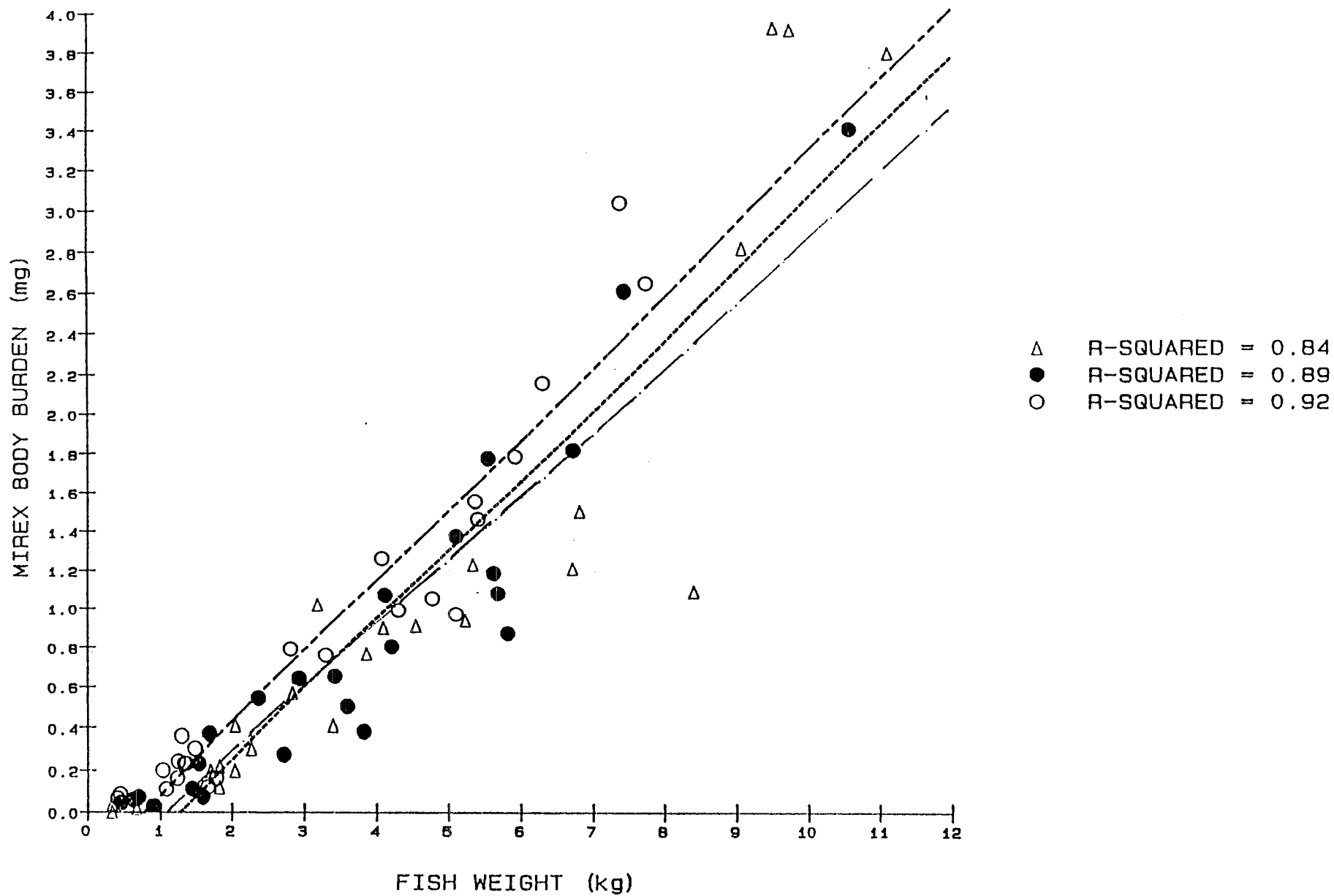


Figure 6. Comparison of the regression lines of mirex body burden and fish weight for 1977, 1982 and 1986 collections.

APPENDIX I

Evaluation of 1988 Analytical Techniques

Replicate mirex analyses (n=4) were performed on a previously analyzed (1982) salmon (Kent 1984 unpublished data). No difference ($P < 0.05$) was detected between mean fillet sample concentrations for 1982 and 1988 analyses.

| | 1982 | 1988 |
|--------------|--------|--------|
| ----- | | |
| Mean (mg/kg) | 0.083 | 0.082 |
| S.D. | 0.016 | 0.019 |
| S.E. | 0.0051 | 0.0095 |
| Replicates | 10 | 4 |

APPENDIX II

Internal Standard Spike Recoveries

Internal standard additions were applied to three samples in which both mirex and photomirex residues had been detected. Experimental design was as follows: 0.5 ml of standard mix (0.1 ug/ml each of mirex and photomirex) were added to each sample. Samples were then evaporated to dryness under nitrogen and reconstituted to 1 ml with hexane prior to analysis by gas chromatography under conditions identical to those used for the entire study.

| | Sample No. 1 | Sample No. 2 | Sample No. 3 |
|---|--------------|--------------|--------------|
| Mirex Conc. (mg/kg) (before addition) | 0.23 | 0.19 | 0.22 |
| Mirex Conc. (mg/kg) (after addition) | 0.29 | 0.24 | 0.28 |
| % Recovery | 120 | 100 | 120 |
| Photomirex Conc. (mg/kg) (before addition) | 0.085 | 0.10 | 0.098 |
| Photomirex Conc. (mg/kg) (after addition) | 0.14 | 0.16 | 0.16 |
| % Recovery | 110 | 120 | 124 |

APPENDIX III

Field and analytical data for 1986 Collections of Lake Ontario Coho and Chinook Salmon.

| SAMPLE NO. | SPECIES | SEX | AGE (yr) | LENGTH (cm) | WEIGHT (kg) | LIPID % | MIREX CONC. (mg/kg) | PHOTOMIREX CONC. (mg/kg) |
|------------|---------|-----|-------------|----------------|----------------|------------|------------------------|-----------------------------|
| K1 | COHO | M | 1+ | 37.3 | 0.34 | 6.53 | 0.12 | 0.052 |
| K2 | COHO | M | 2+ | 62.2 | 2.27 | 3.22 | 0.13 | 0.055 |
| K3 | COHO | M | 2+ | 71.5 | 3.40 | 3.86 | 0.12 | 0.042 |
| K4 | COHO | M | 2+ | 66.7 | 2.84 | 3.16 | 0.20 | 0.12 |
| K5 | COHO | F | 2+ | 68.5 | 3.18 | 6.28 | 0.32 | 0.18 |
| T6 | CHINOOK | M | 3+ | 90.2 | 8.40 | 0.35 | 0.13 | 0.035 |
| T7 | CHINOOK | M | 1+ | 52.8 | 1.82 | 6.10 | 0.067 | 0.031 |
| T8 | CHINOOK | M | 1+ | 53.0 | 1.82 | 3.41 | 0.12 | 0.047 |
| T9 | CHINOOK | M | 1+ | 55.4 | 2.04 | 5.84 | 0.10 | 0.039 |
| T10 | CHINOOK | M | 3+ | 97.2 | 9.08 | 1.09 | 0.31 | 0.10 |
| T11 | CHINOOK | M | 3+ | 90.2 | 9.53 | 2.11 | 0.41 | 0.12 |
| T12 | CHINOOK | F | 3+ | 94.0 | 9.76 | 1.95 | 0.40 | 0.15 |
| T13 | CHINOOK | F | 3+ | 100.3 | 11.12 | 2.27 | 0.34 | 0.18 |
| T14 | CHINOOK | F | 2+ | 81.3 | 6.70 | 1.75 | 0.18 | 0.072 |
| T15 | CHINOOK | M | 2+ | 72.5 | 5.22 | 4.79 | 0.18 | 0.090 |
| T16 | CHINOOK | M | 2+ | 82.6 | 6.81 | 9.05 | 0.22 | 0.098 |
| T17 | CHINOOK | M | 1+ | 54.0 | 2.04 | 3.46 | 0.20 | 0.099 |
| K18 | COHO | M | 3+ | 78.7 | 4.54 | 2.12 | 0.20 | 0.11 |
| K19 | COHO | M | 3+ | 80.0 | 5.33 | 2.96 | 0.23 | 0.10 |
| K20 | COHO | M | 3+ | 74.9 | 4.09 | 2.85 | 0.22 | 0.072 |
| K21 | COHO | M | 3+ | 75.7 | 3.86 | 2.88 | 0.20 | 0.067 |
| K22 | COHO | F | 1+ | 59.7 | 1.70 | 1.35 | 0.12 | 0.055 |
| K23 | COHO | M | 1+ | 45.0 | 0.68 | 1.69 | 0.032 | ND * |
| K24 | COHO | M | 1+ | 39.4 | 0.34 | 1.56 | 0.015 | ND * |

* Not Detected