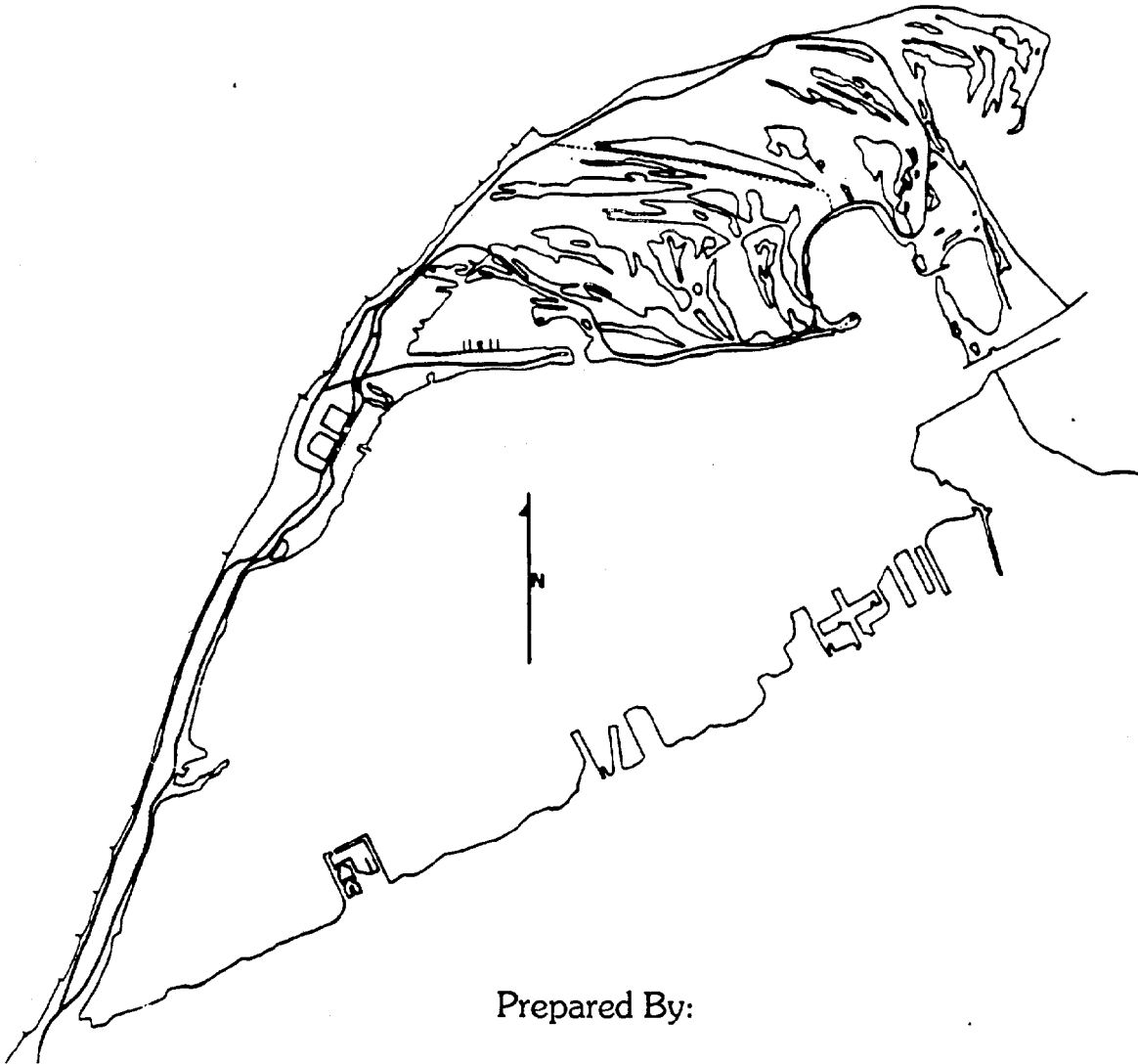


# The Lake Erie/Presque Isle Bay Fish Flesh Study 1987-1988



Prepared By:

**Erie County Department of Health  
December 1989**

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1989

**Coastal**

Funded and Coordinated through  
Dept. of Environmental Resources  
Office of Resources Management  
Div. of Water Resources Management  
Div. of Coastal Zone Management

**Zone**

**THE LAKE ERIE/PRESQUE ISLE BAY**

**FISH FLESH STUDY,**

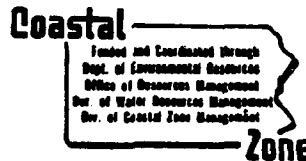
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### Abstract

Fish were collected from Presque Isle Bay and the Pennsylvania waters of Lake Erie, by the Erie County Department of Health. The fish were analyzed at the Pennsylvania Department of Environmental Resources (DER) laboratory in Harrisburg, Pennsylvania. They were tested for select organic and inorganic contaminants. The purpose of the study was to broaden the base of knowledge on fish contaminants in the local area. This study was not intended to be the final word on the subject, and any choice on whether to eat or limit consumption of fish is left up to the individual.

There is a great deal of uncertainty as to exactly what might be considered "safe" to eat. Pennsylvania issues advisories against eating certain species of fish, when there is evidence to show that contaminant levels exceed the United States Food and Drug Administration (FDA) "action level." Currently the State advises against eating carp and channel catfish from the Pennsylvania waters of Lake Erie. Recent controversy, however, has developed with respect to the adequacy of the FDA's action level in adequately protecting the health of sport fishermen who may consume more than the "average" amount of fish. An FDA market basket survey reportedly indicated that the average American consumes about five pounds of fish per year. Some sportsmen and their families may consume considerably more than the FDA estimate. Because organics, such as PCB's,

accumulate over time in the human body, there are health concerns that need to be considered. In this study we have listed the FDA's action levels as points of comparison. However, because of the rather recent questions on the safety of such action levels, we do not mean to imply that just because certain fish tested apparently don't exceed the action level that they are "safe" to eat. Such a determination is well beyond the scope of this project.

Eighteen species of fish were collected and their edible fillets were tested for fourteen organic chemicals, including PCB's. They were also tested for eleven metals, percent lipids ("fat content") and percent moisture.

Six species of fish did not reveal any amount of organic contamination above the detection level of the laboratory test. None detected (ND) should not be equated with none present, because it is possible that some would be found if lower detection methods were used.

Except for chlordane in five species, none of the eighteen species of fish analyzed by the Pennsylvania DER exceeded any of the FDA action levels. [Note in a split sample with the State of Michigan (a large lake trout), Michigan did find PCB's to be just slightly above the 2 ppm FDA action level.]

As noted above, five species of fish had values of "technical chlordane" above the FDA action level. They included carp and channel catfish (currently on Pennsylvania's advisory list), as well as lake trout,



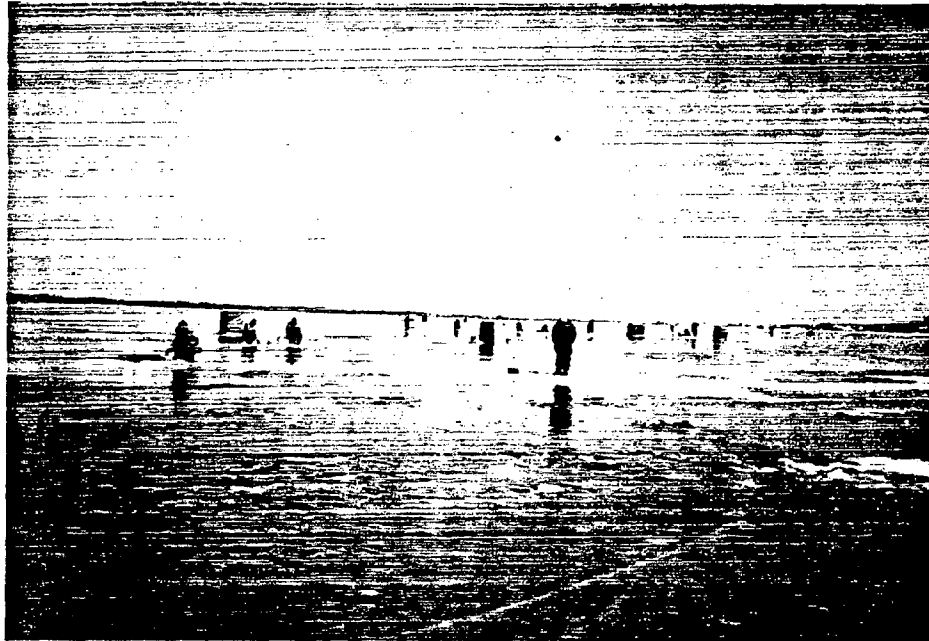
gizzard shad and a largemouth bass. A significant question arose regarding the testing procedures for chlordane used by the State of Pennsylvania, compared to methods used by some other agencies. Depending on the methods used, chlordane results can be significantly different. For example, while the DER laboratory found 0.32 ppm chlordane in a largemouth bass (the FDA action level is 0.3 ppm), the Michigan Department of Public Health found none detected in the very same bass. Other data also showed that Pennsylvania might be overestimating the chlordane results, as compared to other agencies. An intensive review by the Pennsylvania DER laboratory of the chlordane testing methodology was undertaken. It is likely that, in the future, testing protocols will become more standardized so that inter-agency comparisons will be more meaningful.

Of the eighteen species of fish tested none exceeded the FDA action level for mercury. Mercury currently is the only metal that has an established action level.

Problems relating to quality assurance were discovered regarding lead analysis. The original lead results, as noted in this report, are apparently overestimates of the true values. A limited amount of followup laboratory work revealed that some of the originally reported lead results were up to about a factor of ten higher than the followup testing results using improved methodology. Refinements in laboratory methodology were undertaken, and future test results will reflect the changes.

Additional fish collections, already underway, along with increased field and laboratory proficiency, should help clarify the above issues. For example, for our "routine" water quality work for 1989, yellow perch, channel catfish, walleye and Lake trout from Lake Erie have been scheduled and collections started.

As new data on fish from Erie County is generated, it should be available for the public. Interested parties may wish to contact the Erie County Department of Health, 606 West Second Street, Erie, Pennsylvania, 16507, to check for updates on the fish analysis.



Weekday Ice Fishermen in Presque Isle Bay  
Study Area "C" - This and other areas can  
get "very" crowded on weekends.  
(photo March 1989, R. J. Wellington)

## I. Introduction

In October 1987 the Erie County Department of Health initiated a study of fish flesh contaminants in select species of fish from the Pennsylvania section of Lake Erie and Presque Isle Bay. The study was funded in part by the County of Erie and a grant from National Oceanic and Atmospheric Administration (NOAA) through the Division of Coastal Zone Management, Commonwealth of Pennsylvania Department of Environmental Resources (see page 5).

The purpose of the study was to determine the levels of certain organic chemicals and heavy metals in the edible portion (fillets) of fish (see figures 1, 2 and 3 for general study areas, pages 6-8). The Erie area is a center of attraction for sport fishermen, as well as providing a limited commercial fishery. The area offers unique diversification in sport fisheries. Both warm water and cold water fish are abundant. It is possible to fish for lake trout and salmon in the morning and for perch or walleye in the afternoon. Not only does the open lake provide excellent fisheries, but Presque Isle Bay offers a protected harbor for fishing even when Lake Erie is too rough to fish. In addition to the summer fishing, the bay also offers a good ice fishery for panfish (see photo on previous page). Some winter catches in the bay may include an occasional rainbow trout, coho salmon or northern pike, which adds interest to the ice fishing trip. Unfortunately, this protected harbor has received considerable amounts of

pollution from the Erie area over the years.<sup>1</sup> There was enough concern over the bay that the bay has been recommended to become the 43rd Area of Concern by the Science Advisory Board and the Water Quality Board of the International Joint Commission (IJC).<sup>2</sup>

An area of concern is an area designated where there is a major problem(s) and impairment of use(s) due to pollution, e.g., closed bathing beaches, dredging restrictions, etc.

Because of the high interest in the lake and bay fisheries and the general lack of information on fish contaminants from our specific study area, it was decided that more information was needed so that the public could make better educated decisions on the advisability of eating fish from these areas.

This study, because of its limited resources, is not meant to be the final word on local fish contaminants. It is to serve to supplement existing data and provide the impetus for additional data collection efforts.

The reader is cautioned that the numbers of fish collected are very small compared to the total population in the lake and bay and may not represent the "average" fish contamination levels of a particular species. Also, fish of different ages and/or sizes may and probably do contain different concentrations of contaminants. Likewise, fish from the eastern portion of the study area, for example, may or may not contain different contaminants and levels of contaminants from those to the west near the Ohio line.

The choice of parameters analyzed for was based on some of the more commonly found contaminants, what the laboratory could run and what was affordable under the Coastal Zone Management grant. It is possible that other contaminants not tested for could be in the fish. It is also likely that some contaminants are present in concentrations below the laboratory's detection level. The term not detected (ND) should not be equated with not present.

Where Federal Food and Drug Administration (FDA) guidelines are available, we have noted them (see Appendix A). They are used by some people as a general reference point but, again, caution must be exercised. The FDA action levels were not intended by the Federal government to serve as "safety" guidelines for localized populations who may be eating many more sport fish caught than the "average" American.<sup>3,4</sup> It is likely that the FDA and U. S. Environmental Protection Agency (EPA) and/or other cooperating agencies will further refine what are considered to be acceptable "safe" risk levels. Much needs to be evaluated, especially the human health effects of multiple contaminants in fish tissue.

We believe this study does provide a meaningful start on determining contaminants in fish, but we must advise the reader that the decision on whether to eat certain fish is an individual choice. It is suggested that consumers of fish keep themselves updated on new developments and be aware that "new" chemical contaminants in fish likely will

be discovered. "Acceptable" risk levels may be adjusted as more information is available. Qualified physicians should be consulted about the advisability of consuming fish. Particularly the risk to children, pregnant women and women of child-bearing age should be considered.

Included in this report, as noted above, is a list of FDA action levels for informational purposes. It must be pointed out even if no tested-for-parameters for the specific fish exceed FDA action levels, there is no guarantee those species of fish are safe or are not safe to eat. On the other hand, literature indicates that eating fish may have beneficial effects.<sup>5,6</sup>

## II. Study Design

The Fish Flesh Contamination Study was initiated because of the public's concern over eating fish from local waters. The Pennsylvania Department of Environmental Resources (DER) Division of Coastal Zone Management (CZM) provided an 80% grant for the study. The Erie County Department of Health provided the additional 20% with both monetary resources and in kind services. The Pennsylvania DER Bureau of Laboratories provided the fish flesh analyses under contract to the County of Erie.

Fish were collected in accordance with Pennsylvania Fish Commission permits. The general collection methods included the use of gill nets set in the lake and gill and hoop nets set in the bay. Rainbow trout were collected from Trout Run by using dip nets. Fish collected in Presque Isle Bay during the winter of 1988 were captured by hook and line through the ice.

Fish were weighed, measured and processed in the field. Fish were frozen and shipped to the Pennsylvania DER Laboratory in Harrisburg, Pennsylvania for analysis (see Appendix D for protocols).



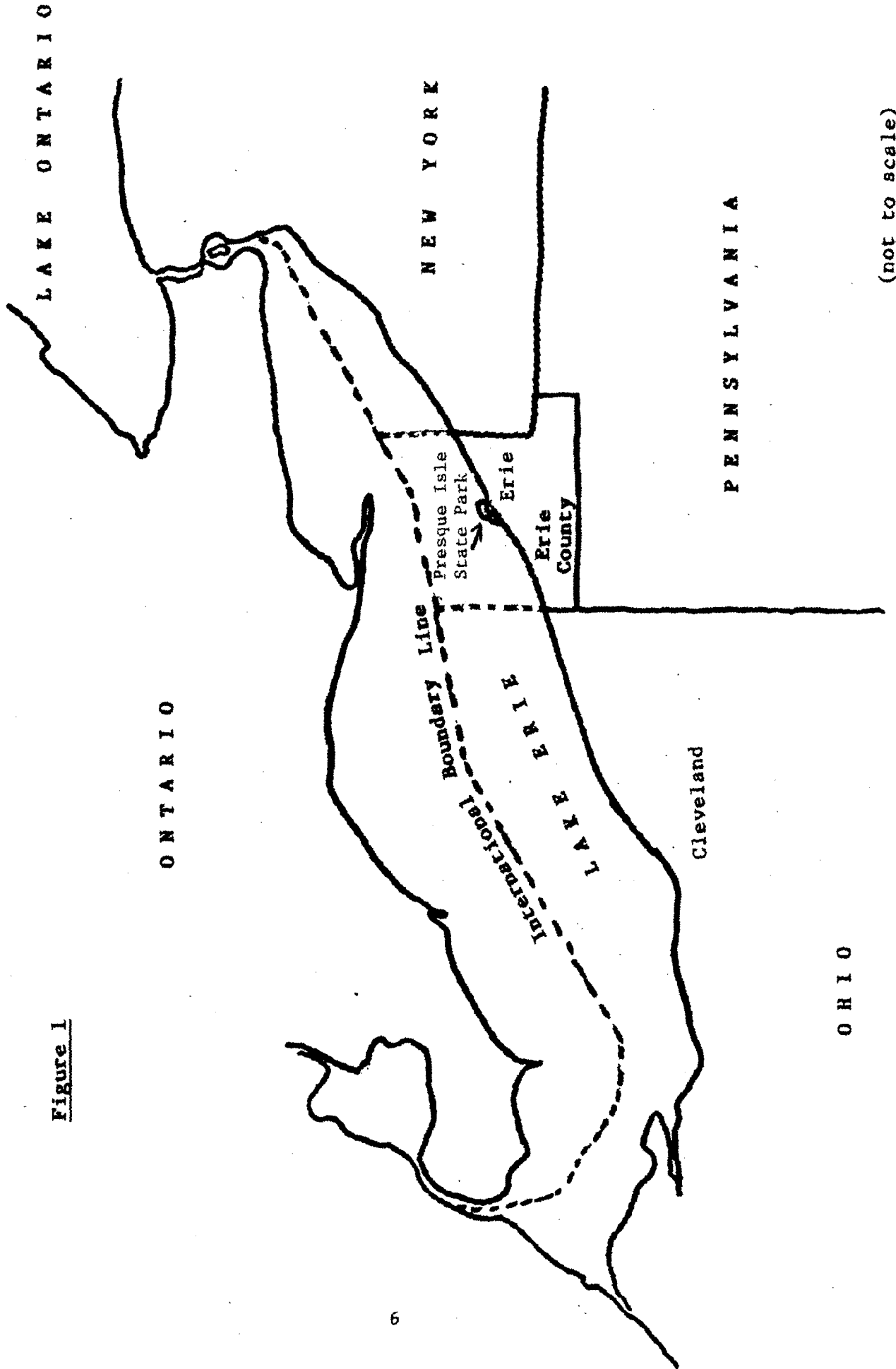


Figure 1

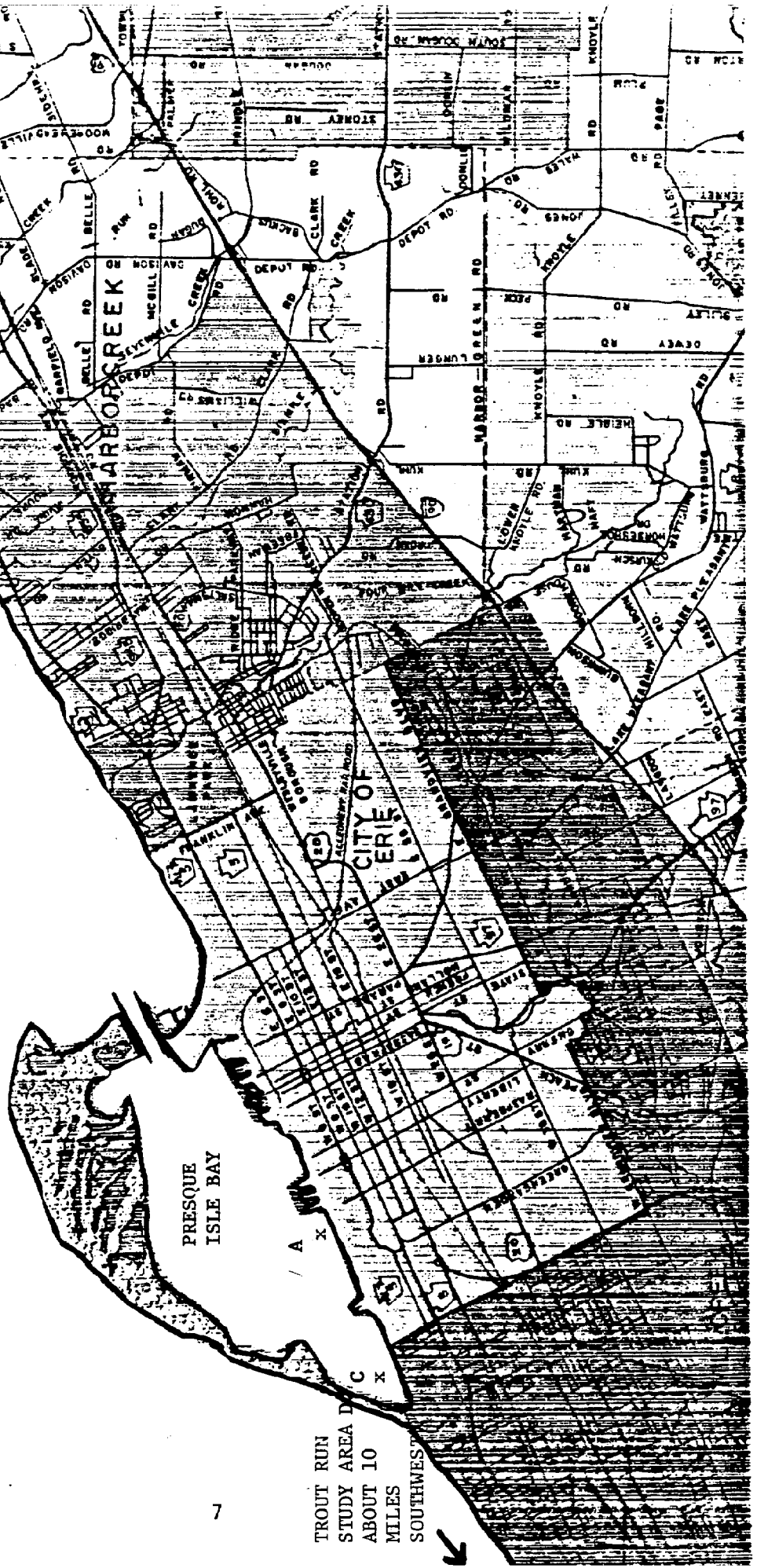
(not to scale)

L A K E E R I E

Figure 2



SHADES BEACH  
STUDY AREAS



TROUT RUN  
STUDY AREA D  
ABOUT 10  
MILES  
SOUTHWEST



### III. Quality Assurance/Quality Control (QA/QC)

Quality control assures that test results are consistent and reproducible. Duplicate samples of the same test theoretically should be very close to each other. Blank samples are samples that should not contain measurable amounts of the contaminants to be tested. They help assure that there is no outside interference or contaminants being introduced into the testing process or errors in the interpretation of results. Spike samples help determine the percent recovery of a particular element or substance in a particular test. In a spike sample, a known amount of contaminant is injected into the sample. The actual recovery of a spiked blank sample should be proportionately very close to the amount put in. If there are significant differences in concentrations one way or the other, it could be inferred that either the laboratory could not find a particular substance even if it were there, or was perhaps finding much more than was thought to be present.

Quality assurance demands that not only are the results consistent and reproducible, but that indeed they are reasonably correct and that other laboratories should be finding approximately the same levels of and types of contaminants. For some types of samples, a laboratory can obtain a certified standard, that is, a standard known to be within a certain tolerance. However, for some parameters it is difficult, if not impossible, for a supply house to certify the amounts of contamination in a commercially

available fish tissue. Because of the ubiquitous nature of some organic chemicals, it is difficult to find fish with absolutely no contaminants. Also because certain contaminants may be more concentrated in fat than in muscles and that fat is not uniformly distributed in fish, getting a "perfect homogenate" is, at best, very difficult if not impossible. In the absence of known samples for many parameters in fish, then "round robin" analysis of a particular sample by various laboratories may be the only reasonable substitute for the situation where there are no certified quantities available. "Round robins" involve having various laboratories participating in the analysis and recording their individual answers. Later the results can be prepared and a consensus may be taken and/or standard deviations determined, etc.

Initially we had hoped that there would have been more of a QA protocol prior to the running of any test for our project. However, because of monetary and time constraints, the State DER laboratory agreed upon the QA/QC program which would work in QA/QC along with routine testing of our CZM fish. The program specified for every four fish tested, there would be a spike and a duplicate sample (see Appendix J) and that the DER would obtain and check some reference samples.

An actual lake trout sample of "known" chemical contaminants was obtained for the purpose. It was provided by the U.S. Fish and Wildlife Service and labeled "85-C."

This check 85-C came as the result of many round robin tests of the fish. When our laboratory tested this sample, it appeared clear to us that there were differences in the chlordane results, that is, the State DER lab was showing considerably more chlordane than some other laboratories (see Appendix J).

Because of this difference and the fact that in the past the State lab found more apparent chlordane than other laboratories, the Erie County Department of Health formally requested that the State DER laboratory try to resolve the question as to whether they were correct with respect to the chlordane results, or at least within an acceptable window of accuracy (see Appendix J).

In discussing the chlordane test results, it was suggested by an outside agency that, as the State of Michigan had considerable experience and expertise working with chlordane and other contaminants, perhaps they could help us. The Michigan Department of Public Health CEHS, Division of Laboratory Services was contacted and they agreed to test three fish samples for us. We chose the right fillets from a composite of channel catfish (CZM 32, 0692285), a largemouth bass (CZM 18, 0692288) and a large lake trout (CZM 6A, 0692264). The Michigan Department of Health found considerably less chlordane in the same fish than did the Pennsylvania DER (see Appendix J). It was also clear that while Michigan was looking at certain isomers and components of chlordane, the Pennsylvania State DER

laboratory was looking for "technical chlordane," rather than adding the sums of selected chlordane components as Michigan was doing. It should be noted in comparing the data on DDE; both laboratories showed the single-peak component DDE test results to be virtually identical (see Appendix J). This indicated good QA/QC for DDE but pointed out the question of quality assurance regarding the multiple components of chlordane.

Subsequent to comparing the testing results of the three fish between the two laboratories, Michigan sent their extract from their right half of the Pennsylvania lake trout in question back to Pennsylvania. The Pennsylvania State DER laboratory tested Michigan's extract again and found virtually identical DDE results and found a PCB isomer; but in looking at two chlordane isomers, alpha and gamma, again Pennsylvania found more than Michigan, which raised a series of discussions concerning test procedures. The resolution of these concerns was determined to be beyond the scope of this project. EPA and others are currently working on a revised protocol for conducting chlordane testing in fish samples.

#### IV. Study Area

The study area included open Lake Erie waters of Pennsylvania east of Presque Isle State Park, Presque Isle Bay and Presque Isle State Park waters, and Trout Run, a small tributary stream which enters Lake Erie about ten miles west of the City of Erie (see figures 1, 2 and 3).

The general locations of the catches were primarily in Presque Isle Bay or Lake Erie to the east of Presque Isle State Park. The decision on where to collect fish was due, in part, to the fact that most of the major Pennsylvania discharges in the area enter the bay or Lake Erie to the east of the bay. Also, because Presque Isle State Park provides natural protection from westerly winds, it was determined to be somewhat safer for the collectors to set nets to the east of the park. Had we been able to have more samples tested, we also would have attempted to collect fish from the area near the Ohio line and in deeper offshore waters of Lake Erie.

Although most collection locations are designated by latitude and longitude, it must be pointed out that these are general locations as determined by the Loran C navigation system and/or coordinants on lake or bay charts. For example, because more than one net was sometimes set in one area, the far end of one of the nets might be a distance from the starting point. Also, when we collected fish through the ice, it was necessary to move around in the general area of a given latitude and longitude until a sufficient number of fish were collected.



Most of the fish collected in the bay (see study area A, page 8) were collected at various water depths between the mouth of Cascade Creek and the Erie Yacht Club. This area is on the south side (City of Erie side) of the bay, and the area has received considerable amounts of waste water over the years from Cascade Creek.<sup>7</sup> Because of the physical nature of the bay off the Cascade Creek sampling area including the creek mouth, weed beds, rock bars and some deeper water areas, we were able to catch a wide diversity of fish species.

Some fish were collected in the lagoons on Presque Isle State Park (see study area B, page 8). This is a popular sport fishing area. It is somewhat removed from any direct waste discharges but is subject to flows into and from the bay, depending on wind direction and water levels. It is suspected there may be fish migration between the bay and the lagoons during certain times of the year.

During the winter of 1988 yellow perch and bluegills were collected in study area C west of the Erie Yacht Club and east of the western end of the bay. These fish were collected by hook and line.

Rainbow trout were netted from Trout Run. These were Lake Erie fish that ascended the creek for spawning (see study area D, page 7).

Fish were also collected off the area of Shades Beach, which is located about eight miles east of Erie. This is a

popular spot for fishing for smallmouth bass and walleye (see study area E, page 7). Some yellow perch were collected between Shades Beach and Shorewood Beach to the east.

## V. Selection of Target Species

Fish were collected according to several criteria including their importance in the sport and/or commercial catch, their level on the food chain and their availability. It will be apparent to the reader that coho salmon, a popular sport fish, were not sampled in this study. There is an ongoing coho sampling program as part of the Great Lakes International Surveillance Plan (GLISP). Sampling coho in this study would have duplicated that program.

In general, in our CZM study larger sizes of fish were used when available, as we believed that the larger fish would be older and would have been exposed to contaminants for a longer period of time. However, IJC reported that in one study 5+ year old walleyes "contained lower PCB levels than both age 1 and age 4+ fish." The reason for this was not clear. It was suggested that it may have been due to bias because of small sample size.<sup>8</sup> Originally we had hoped to look at various sizes of fish of the same species but, because of limited time and money, we generally were not able to do this. We did collect two sizes of rainbow trout and one very large and one smaller lake trout.

We deliberately sampled terminal predator fish, such as northern pike and a muskellunge, as well as bottom feeders, such as channel catfish, carp and brown bullheads. We believed terminal predators might show the results of

biomagnification of contaminants, and bottom feeders would be in more direct contact with contaminated sediments and food chain organisms that live on or near the bottom sediments.

We also looked at yellow perch in Presque Isle Bay during different seasons of the year. This sampling was conducted because it had been suggested that body burdens of contaminants in fish may be different at different seasons, depending on factors such as feeding habits and/or pre- and post spawning conditions (see Appendix C for species collected).

## VI. Equipment, Fish Collection and Processing

Most of the fish in this study were collected using gill nets. Methods of collection are noted with other general information on the data sheets for each of the individual sample results. Besides gill nets, fish were captured with dip nets (from Trout Run), hoop nets in Presque Isle Bay and the lagoons on Presque Isle State Park and hook and line through the ice on Presque Isle Bay. Gill nets varied, but generally were 50 to 100 yards in length and were 6 feet in height. Nets were set on the bottom of the lake or bay. Net mesh sizes ranged from 1-1/4 inch square to about 2-1/2 inch square. The smallest net, 1-1/4 inch square, was a multifilament float and lead type net. All other gill nets were monofilament lead and float type nets. Some of the gill nets had the lead weights internally inserted in the lead line on the bottom of the nets. Other nets had the lead weights attached to the exterior of the bottom lines. The gill nets after use were usually stored in galvanized metal wash tubs. Nets were generally set one day and pulled early the next day.

Nets were carefully pulled by hand into the boat and loaded directly into the metal tubs. When very large fish were encountered, they were generally taken from the net and then placed in another wash tub. The procedure we generally used was to line an empty tub with clean aluminum foil, dull side up (the shiny side is treated and may not be suitable for our testing purposes), and place the large fish on the

clean foil to avoid contamination. We were careful never to buy gasoline when we had fish on our boat (to avoid the possibility of getting any gasoline on the fish). When we did purchase gasoline, we were very careful to avoid spilling gasoline or otherwise contaminating the work area.

Smaller fish were brought aboard the boat and fish and nets were placed in the metal tubs. Later, after all nets were pulled in, we picked the fish from the gill nets and placed them in a prepared tub or a clean, stainless steel bucket until they could be processed.

The same general procedures were used when we used hoop nets or fish caught on hook and line.

In selecting fish for analysis, where there were more than five fish of the same species, we tried to pick the five biggest fish. We assumed that the largest fish would likely be the most contaminated and also are the ones most likely to be kept by fishermen for consumption (see Appendix H).

Where we caught less than five fish of a particular target species, we used all the fish that were available, unless there was a very significant size difference. For example, if we captured three large fish of a given species and one very small fish, our policy was to keep the three similar size fish for analysis and discard the very small fish. Therefore, our composite would include three, rather than four, fish. We believed that radically mixing sizes of fish would provide less meaningful information.

Once the fish were collected, they were prepared as follows: lengths and weights were taken and recorded, along with date of capture, location of capture, method of capture and any other relevant information. Before this CZM project was initiated, our procedures were discussed with the Bureau of Water Quality Management of the Pennsylvania DER. We agreed to use their sampling and processing protocol (see example of DER's field data collection sheet that we used, Appendix D).

It was agreed that we would analyze standard skin on fillets. This was in keeping with work being done in other Great Lakes areas. The idea was to try to provide sample results that could be compared to similar samples in other areas. It should be noted that FDA uses skinless fillets for catfish. However, we left the skin on catfish and bullhead fillets as we believed this would be the more conservative approach. Any future studies are expected to have the skin removed from catfish samples which is in keeping with the FDA protocol and more in keeping with how most fishermen clean their catfish.

Where fish species were encountered with pelvic fins on the "standard fillet," we elected to remove these fins (see Appendix D).

Other fish were scaled and cleaned with commercially available metal fish scalers. The scalers were cleaned each time prior to use. Our protocol for the cleaning procedure involved rinsing the scaler. After being cleaned in this manner, the scaler was rinsed with pesticide grade hexane to attempt to remove any possible organic contaminants.

After the fish were scaled, they were filleted using a stainless steel knife. The knife was cleaned each time in a manner similar as described for the fish scaler. It should be pointed out that the scaler and the knife were cleaned between each composite sample. This was done to eliminate cross-contamination of fish samples.

Filleting took place on clean aluminum foil, dull side towards the fish. Aluminum foil was replaced with new, clean foil between composite fish samples. The foil was not replaced nor was the knife cleaned for individual fish within a composite.

When we started collecting fish in the fall of 1987, once the fish were filleted the fillets were scraped reasonably clean, wrapped and frozen. Later we learned that the fillets were not later washed by the laboratory, rather the fillets were ground up in a frozen or semi-frozen state and processed. This meant any slime or scales, etc., still on the fish fillets ended up in the edible fillets. To remedy this, on at least one occasion (5/4/88), some fish were rinsed in "clean" nearshore waters. The wisdom of this was later questioned and subsequently the practice was discontinued. Later, more care was given to more carefully scrape as much mucus and loose scales off the fillets as reasonably possible with the knife blade. However, there is little doubt some mucus and scales remained on at least some of the fillets.



We filleted both sides of each fish. We securely wrapped all left side fillets for each composite sample in clean aluminum foil, dull side towards the fish. The bright side of aluminum foil has a special factory coating that might interfere with test results. The foil was taped closed and labeled with species name, location, date and an identifying number (see Appendix D). The right fillets were processed in a similar manner. The packages were then placed in food grade plastic bags to further prevent the fish from contamination, prevent tearing of the aluminum foil and to retard dehydration. It was our general policy to send the left fillets to the laboratory and keep the right fillets for duplicate sample "backups." The backups proved very useful when additional testing on the composite was done.

As fish were collected and frozen, the laboratory was contacted and at appropriate times fish were shipped to the laboratory in coolers with dry ice. The laboratory was always alerted as to when they should expect to receive the fish so that they could put them in their freezers as soon as they were received.

## FIELD PROCEDURES

To eliminate variables due to procedures, it is suggested that in future studies, where possible, whole fish be shipped to the contract laboratory. This would help eliminate possible contamination in the field during fish cleaning operations. An associated problem with field preparation we encountered was rough water, making it more difficult to use measuring scales to come up with precise live fish weights. Questionable sources of "clean" water were another concern. We assumed that as the fish themselves came from the water, it "wouldn't hurt" to wash our scalers, rinse our knives and hands in the water. Our policy was not to rinse instruments on the downwind side of the boat and use the water only on the "clean" side of the boat. We followed up washings of the scalers and knives with a hexane rinse. Although we were always very careful to avoid contamination, there still is always the question of how clean was the water alongside the boat.

Rinsing knives with hexane itself presented a potential danger on the boat. Hexane is extremely flammable and needed to be carried in a clean glass bottle. Care had to be taken not to spill the hexane or break the bottle on the boat. The danger from fire is increased when such a volatile substance is carried in a glass bottle.

Other problems included waves, which could increase the probability of the worker getting cut on a knife or producing a fillet that was not as uniform as hoped for.

One of the protocols we used included taking dry ice on each and every trip. Dry ice would be needed in certain cases where there would be quite some period of time between catching the fish and when they are put in a freezer. Often we processed the fish at the end of the day. The fish were then placed on dry ice, but perhaps an hour or two after being put on the ice the fish were removed from the coolers and placed in our freezer. Fish fillets were cold when removed from coolers but were usually not frozen, as they had not been in contact with the dry ice long enough. It is believed that using dry ice is a good idea, but in some cases should be left to the discretion of the collector. For example, during the winter, according to protocol, we took coolers with dry ice out on the bay ice; however, we do not believe this was practical or necessary. Having tried this, it is recommended that fish not be processed at all in the field in the winter. If it is necessary to process the fish before sending them to the contract laboratory, they should be processed in an appropriate inside location. Difficulties we encountered were trying to hold wet fish and a sharp knife with very cold hands. In fact, it is dangerous to do so, as fingers lose control over the knife when it is very cold. Accidents are very likely. Also, if gloves are worn, even intermittently to warm hands, their cleanliness both inside and out comes to question. Also, ice may start forming on the knife. Therefore, it is obvious fish cleaning preparations during the winter should not be conducted in the field.

As noted above, not only was clean water a concern for rinsing our knives, but the question arose as to how to properly clean the slime and scales off the fish once they were filleted. This could be important because mucus can in certain instances apparently affect lead results.<sup>9</sup> Scales and slime on test fish may not necessarily represent what a fisherman might be eating. The problem is however finding suitable rinse water or a way to clean the fish. When we commenced the project in the fall of 1987, we did not rinse fish. Some scales and slime were likely unavoidably included in the sample. After learning that the laboratory did not rinse the fish, we did rinse some fish in nearshore waters on May 4, 1988. However, the practice was discontinued. During our fish collection trips, we did not take water with us. We chose not to use plastic containers because of the possibility of organic contaminants from the plastic. The general way we handled the fillets, at least towards the end of the study, was to carefully "wipe" the fillets as reasonably free of scales and slime as possible using a knife blade. However, some scales and slime likely were still left on the fillets. Again, this is another reason not to process fish in the field.

## RECOMMENDATIONS ON FIELD PROCEDURES

Ideally, it seems fish would be shipped whole to the testing laboratory and they would process fish. However, if field processing is to continue, some mechanisms for washing the fillets prior to freezing should be considered. Fish should not be processed in the field during very cold conditions.

It is recommended that edible fillets continue to be collected from both sides of the fish. The right half and left half can be frozen in separate packages. One side can be held in reserve should the original be lost or destroyed or if confirmation of a particular contaminant is needed. These samples also may have future value if the same or another laboratory wishes to use the fish to check their results or perhaps even check for additional parameters if problems are found or suspected or if detection techniques improve. It is suggested that investigators consider keeping the "other half" of the samples for a minimum of six months after any final reports are issued. This would better assure that outside concerned parties would have time to read the report. If comments were generated regarding the results, it would be possible to re-evaluate the issues by making the frozen samples available for more testing.

With respect to the particular study area, it is suggested, if funds become available, some sampling be conducted near the Ohio line and towards the international border to the north of Erie. These areas were not sampled

during this current study. Also, as laboratory capabilities improve and additional environmental contaminant concerns develop, some fish should be checked periodically to monitor present contaminant levels, as well as providing background information should contaminant concentration changes be noted.

When future studies are undertaken, it might be well to consider the advisability of checking composites of only three to four species, for example, and obtaining a rather fast "turn around time." Larger studies may tend to prolong the time between when samples are collected and when they are finally presented to the public. Consideration should be given in comparing quantity of samples versus the timely reporting of a very few samples. For example, had we tested three or four samples and found questionable chlordane and/or lead results at the start of the program in 1987, we would have been more likely to have changed field and/or laboratory methods. Likewise, it was noted that the large lake trout (#0692264) had elevated levels of chlordane and PCB's. Had we received the results in the Fall of 1987, we could have focused more attention towards collecting more lake trout in the Spring of 1988. As it was, all the fish collections were completed before we received the final laboratory results. Had this study been staged into three or four smaller reports, it is likely some of our sampling priorities would have been changed.

## LABORATORY PROCEDURES

### Discussion of Chlordane Results

Chlordane is a mixture of chemicals rather than a pure compound. According to the Handbook of Environmental Data on Organic Chemicals, technical chlordane consists of a mixture of many compounds. Technical chlordane consists of approximately 19% alpha chlordane (cis chlordane), 24% gamma chlordane (trans chlordane) and 10% heptachlor epoxide, as well as other compounds.<sup>10</sup>

Because chlordane is not a single-peak compound, identifying it is not a simple matter. Some laboratories or agencies pick some of the isomers of chlordane and add up the quantities. The Federal Food and Drug Administration's methodology addresses two methods for determining chlordane (see analytical notes, Appendix I). One sums individual components of chlordane 0.02 ppm or higher. The other, if the pattern matches technical chlordane, indicates the results should be quantitated against a technical chlordane standard.

In reviewing the quality assurance information, it was noted that in some cases DER's PCB results were lower at times than other laboratories. However, chlordane was generally a factor of up to 4 (or more) times higher than other agencies were finding in the same sample, raising the question whether the actual levels for chlordane were being overestimated.

Upon reviewing the testing procedures, the laboratory indicated it reported technical chlordane rather than summing isomers. This led to the question, would it be expected that "technical chlordane" as such would be found in fish. We speculated that the various components of technical chlordane would have different decay rates in the environment, different water solubilities, different attractions to silt and clay particles in the water, as well as biomagnification rates. It would not be expected that "technical chlordane" as such would be found in fish.

Because of these questions, we contacted several agencies, including the Michigan State Department of Health. They agreed to help us look at the situation. They agreed to look at the other sides (right fillets) of the lake trout sample (CZM 6A), largemouth bass sample (CZM 18) and the channel catfish sample (CZM 32) (see Appendix J). They found less chlordane using different testing procedures.

Acting on this and other information, it was agreed to do further research into the matter. Both laboratories cooperated and later split samples with the U.S. Environmental Protection Agency (EPA). It appeared, following rather extensive testing, that the DER was overestimating the chlordane results (see excerpts from attachments, Bruzel memo, Appendix K). However, it should be pointed out because of matters far beyond the scope of this report, that at this time we do not have a definite answer as to which method(s) will ultimately be acceptable.



Alan Bruzel points out in his memo dated July 7, 1989 that there will be a chlordane conference held in Missouri. At that time, hopefully, the issue of the correct or acceptable methodology will be resolved. Until then, the reader is cautioned that, although we can clearly see that the chlordane results of the DER are higher than others, it is difficult, if not impossible, to compare the answers. It seems likely that the quantification of isomers might be the preferred choice. If this is so, in the future we might see lower chlordane results than the DER is now reporting.

### Discussion on Lead Results

The FDA has no action level for lead in fish. However, the International Joint Commission (IJC) 1985 Annual Report, Revision of October 1986, suggests that long-term consumption for an adult should not exceed 2 mg/kg.<sup>11</sup> This lead level included both organic and inorganic lead. The IJC also suggested jurisdictions could adopt more stringent standards to protect their respective populations that might be exposed to other sources of lead. They also suggested protecting sensitive subgroups, such as children and women capable of bearing children. It was emphasized that this proposed limit should be considered tentative. When the Erie County Department of Health received the lead results in the fish, we noted two issues. One was that the duplicate samples in some cases were considerably different from the original sample. The other was that the results of some of the lead test results were considered high.

Had the rather high lead level results in our study been found in a particular area, we would have suspected a localized source of lead. However, some of the results showed rather high levels, even in the open lake. After the biologist from the Erie County Department of Health made several phone calls to other agencies, it became clear that the apparent high lead results might be related to a problem with either field preparations and/or laboratory procedures.

A document prepared by Schmitt and Finger showed that there was generally a significant difference in lead results between fish prepared in the field as compared to fish prepared under ultra-clean laboratory conditions.<sup>9</sup>

As a result of the questions, a meeting was held in Harrisburg to discuss the issue of high lead results and other sample procedures.

Regarding lead sampling, it was agreed at that meeting that the Harrisburg laboratory would attempt to secure a certified "fish tissue" lead standard and recheck their QA/QC procedures and recheck mathematics, etc. It was also agreed that the Erie County Department of Health would look into field preparations as a possible cause for the elevated lead levels. It was agreed additional testing would be done by the DER for lead. Four samples that were held frozen (the right fillets from previously tested fish) with high lead or relatively high lead were chosen to be retested.

They were as follows:

CZM #1, yellow perch, 0692260:

|                              |                       |
|------------------------------|-----------------------|
| original sample, left fillet | retest, right fillets |
| 0.758 ppm                    | after rinsing by ECDH |
|                              | 0.317 ppm             |

CZM #1, right fillets, aliquot stored at laboratory from original sample, no additional cleanup by ECDH:

|                 |             |
|-----------------|-------------|
| original sample | 1989 retest |
| 0.758 ppm       | 0.067 ppm   |
|                 | duplicate   |
|                 | 0.073 ppm   |

CZM #19, bluegills, 0692289:

|                 |             |
|-----------------|-------------|
| original sample | 1989 retest |
| 2.35 ppm        | 0.211 ppm   |

CZM #22, sunfish, 0692278:

|                 |             |
|-----------------|-------------|
| original sample | 1989 retest |
| 1.18 ppm        | 0.293 ppm   |
|                 | duplicate   |
|                 | 0.249 ppm   |

The frozen right fillets from the above fish had been taken to a private residence for processing before shipping them to Harrisburg. The fish were thawed and then vigorously washed in the City of Erie drinking water that had previously been tested and found to be of a currently acceptable lead level (<0.05 ppm). All scales and slime and any pieces of true rib bones inadvertently missed in the filleting process were washed and/or picked off the fillets. The fillets were rewashed, drained and then carefully wrapped in clean aluminum foil, put in a food-grade plastic bag and refrozen. They were then shipped to DER's Harrisburg laboratory and retested, using slightly different methodology because of additional knowledge for testing for lead in fish flesh.

Additional testing was also done on CZM #29, 0692287, which consisted of 5 walleyes (see Appendix G). As these walleye fillets were from large fish, it was decided to try to isolate possible sources of lead in the fish. The fillets were subdivided into 3 packages. All 3 packages (composites B, C and D) contained portions of all 5 fish. Hence, each package remained a composite of part of each of the 5 original fish.

Composite A was the result of the original left fillets.

In composite B the skin and scales were removed from the 5 right-half fillets. The bones imbedded in the fish were removed by cutting a wedge of flesh down both sides of the bones to remove a piece of flesh with imbedded bones. Composite B was flesh only. All detectable skin and bones were removed. The fillets were then vigorously rinsed in the City of Erie drinking water under a kitchen spigot.

Composite C consisted of the wedge of bones along with a small amount of flesh on either side of the bones. This composite was rinsed under tap water.

Composite D consisted of the residue skin, "slime" and any loose scales and tips of bones removed during the skin removal process. Skin removal consisted of using a long, sharp knife blade and holding it down against the skin. The fish fillet was placed skin down on clean aluminum foil (dull side towards the fish). A sliding downward cutting motion with the knife was then used to separate the flesh from the skin. The remaining skin and other parts were not rinsed, as it was suspected. Perhaps this fraction of the fish could be much higher in lead.

It can be seen (in Appendix G) that there is a considerable difference between the original lead result and the followup samples. It did not appear that the lead, based on the walleye sampling, was concentrated in the skin or bones in this particular case; rather, the highest lead

concentrations were found in the flesh. It will be noted, however, that overall, the concentration of lead in the 1989 testing was approximately one-tenth the original amount reported in 1988.

Based on the above information, the DER laboratory reviewed their findings and reported their findings in a memo dated August 24, 1989. The memo reads in part:

". . . Your review of your fish tissue data indicated elevated levels of lead in some samples. Because they were higher than permitted levels you were very concerned, as we were. We reviewed all our work and felt it was correct. We agreed to run more samples that (sic) you requested. These samples were submitted to us, and along with some of the original samples, we set up an analytical program in which we altered some of the original preparation procedures. In the first assay run we digested one gram of wet fish tissue. We felt this might not be a true representative sample so for the second run we used two grams of material.

"The wet digestion was performed as normal. During the first analytical procedure we diluted the digested material one to five and performed the normal AA Furnace method for lead. In the study of the data from both runs we discovered the dilution factor caused a higher result that did not show up in the undiluted sample. The diluted sample reading was very close to our MDL, and this way (sic) be the reason for the elevated results.

"We feel the results reported from our second run more nearly represent the lead levels in the fish tissue samples. We do not have enough data to make a statement on other metal parameters reported. We do feel that most of the reported data looked good and the QA results indicated that it was accurate. If any of the reported data indicated excessive levels or levels above the limits, of any of these parameters, we would be glad to analyze similar fish tissue samples.

"In conclusion, it is very difficult to get a truly representative sample of fish tissue. We try very hard, but we are dealing with such a small amount it makes it difficult. One gram of wet tissue will only yield 0.3 grams of dried

material. We do have a new technique available. We can now freeze dry larger amounts of tissue, grind it, mix it and thus get a much more representative sample to analyze. We will be using this technique for our 89 samples. . ."12

The grant that funded this study was to be terminated at the end of August 1989. Because of the lack of time, we were not able to have additional work done on lead or on the other metals. It appears that those "original" lead levels listed in this report, likely, are inflated values. As stated in the above memo, we do not know if the other metals are overestimates of the "true" values or not. The process of fish flesh analysis is an evolving process and almost certainly will be revised as more refinements are made in analytical techniques.

## VII. General Discussion

As can be seen in the species list, there is a good diversity of fish in the bay and nearby Lake Erie waters. We collected several game fish (terminal predators) from the bay. Only one gamefish from the bay, a largemouth bass, showed any contaminant (chlordan) above the FDA action level. This analysis result is in question because the Michigan Department of Public Health did not detect any in a sample from the same fish. It is interesting to note that there were no organics identified in the 35-inch muskellunge. The five-fish composite of northern pike revealed only 0.02 ppm pp' DDE (average size of these pike was 30.1 inches). No PCB's or chlordan were found in these fish.

There was no noticeable change in organic contaminants in yellow perch from the bay over a period covering October, February and June. All perch sampled were reported as none detected (ND) regarding the fourteen organics. At some future time it might be interesting to sample perch roe (eggs) to see if organics accumulate in the roe because some fishermen do eat fried perch roe. The roe might be more contaminated than the flesh.

Of the five species of fish that showed levels of organics over the FDA action level, four came from Presque Isle Bay. However, this may not be directly related to the bay water quality, but more to the type species sampled (and analytical methods used). Carp and catfish are known to be



high in at least one organic contaminant, as evidenced by the existing warning advising against eating these fish. There were no channel catfish or carp from the lake to compare the results to. Gizzard shad are a particularly oily fish; it was suspected they might show some higher results than other species. Sampling confirmed this to be true. The largemouth bass chlordane result was unexpected, but, as noted above, at this time there is serious doubt that the chlordane was over 0.3 ppm, especially when there were no PCB's detected. (Generally, fish have more PCB's than chlordane in them.) The PCB/chlordane ratio alone casts doubt on the chlordane issue.

One large (by Pennsylvania standards) lake trout (32 inches) revealed elevated levels of chlordane. This was confirmed by the Michigan Department of Public Health. However, even though they did find chlordane, Michigan found about four times less chlordane than did Pennsylvania. Michigan did find PCB's in this fish above FDA standards, while Pennsylvania found a lower amount. It should be noted this fish was unusual in that it had a very large head for its body weight. It may not have been representative of another fish that same length. In fact, a fish of similar length with a more normal (i.e., heavier) weight quite likely could have different levels of contaminants. One smaller lake trout did also have chlordane slightly above the FDA action level. This was determined by the Pennsylvania laboratory and was not confirmed.

As noted earlier, as the techniques are refined, better data will be generated.

It should be noted that this study is not unique in coming up with different results compared to other agencies. In the 1987 Report on Great Lakes Water Quality, it was pointed out, "Differences in absolute values between agency programs may result from differing analytical methods. . ."13 It is obvious that more uniform protocols be developed so that fish samples can be more easily compared and verified.

In looking at the reported PCB contaminant levels, it was found that our study (with the exception of freshwater drum "sheepshead") mirrors information put out by the Ontario Ministry of the Environment.<sup>14</sup> In their 1987 Guide to Eating Ontario Sport Fish they show an inverted pyramid. At the top of the pyramid are Lake Trout (most PCB's) and at the bottom are northern pike and freshwater drum (least PCB's). In ascending order are: level one - northern pike and freshwater drum; level two - coho salmon, rainbow trout and chinook salmon; level three - brown bullheads and white bass; level four - channel catfish; level five - brown trout; level six - carp; and the top, level seven - lake trout. In looking at PCB levels (except the drum) in our study using the larger lake trout and larger of the two rainbow (trout composites), we find that the progression is identical with Ontario's relative levels (see Appendix F, Table II). If this "condition" reflects actual lake-wide

conditions in Pennsylvania, because there are current advisories on channel catfish and carp, it seems more attention needs to be focused on brown trout and lake trout.

(Although outside the scope of this report, it has been agreed to focus attention in 1989 on collecting and analyzing additional Lake trout samples. Large brown trout are not particularly common; consequently, getting enough to sample is more difficult. However, if possible, it might be well to also look at them.)

### VIII. Recommendations

It is recommended that, in future studies, not only should internal quality control/quality assurance be practiced but, as was done in this study, an outside laboratory or laboratories should also analyze the fish as early in the study as is practical. This would better insure that any discrepancies in test results could be resolved prior to testing all the fish.

The issue of lead analysis should be followed up as well as looking at and verifying other metals besides lead.

The DER laboratory is committed to further investigating the chlordane issue. This should help resolve this matter. There remains some question as to the PCB results. While not as an apparent difference, as was noted in the chlordane issue, there is evidence to indicate that the Pennsylvania DER PCB results are not always consistent with outside references. It is also evident that there is a question as to whether the PCB's identified are 1260 or 1254. It is recommended that procedures be reviewed regarding PCB interpretation, as is currently being done with chlordane.

It is also recommended that all Great Lake states and appropriate federal agencies come to a consensus of opinion as to proper sampling techniques, field preparation and laboratory protocol, in order to better assure reliable, valid results.

It is recommended that lake trout, particularly larger size fish, be evaluated, and if a reasonable amount of larger brown trout can be collected that they also be checked for organics.

It is recommended that improved field protocol be developed in cleaning the scales and slime from fish prior to their being homogenized (ground up) in the laboratory. Possibly de-ionized water in a suitable, contaminant-free container could be employed to rinse the fish fillets. This would better duplicate what fishermen actually do. Presently, if scales are not rinsed from the sample before freezing, they are homogenized into the sample, as it is easier to grind up frozen fish than soft, thawed-out fish.

It is recommended, if at all possible, fish not be processed in the field in the winter. This is due to problems associated with frozen knife blades, cutting fingers, etc.

When future studies are conducted, it would be better to run fewer samples during phases of a study so that the results could be made public in a more timely fashion or even change direction in sampling priorities as data comes in. In other words, if the lab and field protocol was better established and in a pre-printed format, it would be relatively easy to prepare a report by just adding species, locations and test results. Perhaps the time from sampling to usable results could be reduced from months or years to perhaps weeks or months.

## IX. Summary

This study provided meaningful insight into the complexities of fish contaminant studies, and as a result there will likely be changes in procedures both in the field and in the laboratory.

Bay fish that were sampled, including yellow perch, sunfish, bluegills and black crappies, did not have detectable levels of the organic contaminants. This provides some degree of reference for potential consumers, if they choose to apply the FDA action level.

One fish, the large lake trout, had levels of chlordane above the FDA action level, and this was confirmed by more than one laboratory. Four other species of fish (carp, channel catfish, bass and gizzard shad) had chlordane levels exceeding the FDA action limit. However, when Michigan checked the same catfish and largemouth bass they found a chlordane level below the FDA action level on the catfish and did not detect any at all in the bass. Therefore, if one were to make allowances for methodology, that is, looking for a technical chlordane pattern versus adding up components of technical chlordane, the FDA limits may or may not have been exceeded (other than the lake trout). Some other fish, as determined by DER, had higher than expected chlordane results approaching the FDA action level. Again, the chlordane results are in question.

None of the DER PCB results exceeded the FDA action level. However, Michigan did find elevated levels of PCB in the large lake trout. Because of the differences in quality

assurance in test results among laboratories, the DER's PCB results should not necessarily be taken as absolute values. They may be higher or lower than reported values.

Lead results likely are much lower than this document's "first-run" reported values. Other metal results should be looked at in the future by the laboratory to see if only the lead results were high or if the techniques used also inflated (or underestimated) other reported metal results.

It should be noted that in all fish tested, all mercury results were not only below the FDA action level but were also all below 0.5 ppm, which is a standard used by some other agencies.

Because of the variables in testing procedures and consequently in the analysis results, it is recommended that Pennsylvania, along with all Great Lake states and other appropriate governmental agencies, adopt strict uniform testing procedures. It is difficult to evaluate risk assessments or determine the validity of issuing a consumption advisory (or not issuing a consumption advisory) based on only a few fish, when, in fact, laboratory results among agencies are not always consistent.

While the results of this study show the presence of some contamination in fish, it is somewhat comforting to note that at least some fish species showed all test results below the laboratories detection levels for the fourteen organics. This study also reaffirms the fact that our area does not appear to be as contaminated as some other waters

in the Great Lakes area. Hopefully, with increased emphasis on cleaning up the environment, someday all fish will be found to be below even lower detection levels, and for all practical purposes will then all be termed "safe." At least for now, however, prudence and caution should be exercised until more is known about the subject.



**A P P E N D I X    A**

**FDA Fish "Action Levels"**

**United States Department of Health and Human Services**

**Public Health Service**

**Food and Drug Administration**

**FDA "Action Levels" for Fish**

|  |         |
|--|---------|
| Aldrin and Dieldrin (edible portion)               | 0.3 ppm |
| DDT, TDE and DDE (edible portion)                  | 5.0 ppm |
| Endrin (edible portion)                            | 0.3 ppm |
| Heptachlor and Heptachlor Epoxide (edible portion) | 0.3 ppm |
| Mercury (methyl mercury in edible portion)         | 1.0 ppm |
| Mirex (edible portion)                             | 0.1 ppm |
| PCB  | 2.0 ppm |
| Toxaphene (edible portion)                         | 5.0 ppm |

**A P P E N D I X B**

**Sampling Locations**

## Locations of Study Areas

### Study Area A

Presque Isle Bay (south-central area of bay)

North Latitude: 42°07'36"                      North Latitude: 42°07'37"  
West Longitude: 80°07'09"                      and                      West Longitude: 80°06'48"

### Study Area B

Presque Isle State Park (lagoons - backwater off Presque Isle)

North Latitude: 42°09'33"  
West Longitude: 80°06'00"

### Study Area C

Presque Isle Bay (western end of bay)

North Latitude: 42°07'03"                      North Latitude: 42°06'51"  
West Longitude: 80°08'28"                      and                      West Longitude: 80°09'00"

### Study Area D

Trout Run is a small tributary to Lake Erie, about 10 miles west from Erie, near PA Rt. 98

### Study Area E

off Shades Beach (35 - 40 ft. water)

North Latitude: 42°11'58"                      North Latitude: 42°14'19"  
West Longitude: 79°57'53"                      and                      West Longitude: 79°57'39"

Note: Nets for yellow perch sample #CZM291 were set off Shades Beach in about 64 ft. water.

**A P P E N D I X C**

**Species Sampled**

**Common and Scientific Names for Fish**

## Species Sampled

| <u>Common Name</u>           | <u>Scientific Name</u>   |
|------------------------------|--|
| Bass (largemouth)            | <i>Micropterus salmoides</i>                                     |
| Bass (smallmouth)            | <i>Micropterus dolomieu</i>                                      |
| Bluegill                     | <i>Lepomis macrochirus</i>                                       |
| Bullhead (brown)             | <i>Ictalurus nebulosus</i>                                       |
| Carp                         | <i>Cyprinus carpio</i>   |
| Catfish (channel)            | <i>Ictalurus punctatus</i>                                       |
| Crappie (black)              | <i>Pomoxis nigromaculatus</i>                                    |
| Gizzard Shad                 | <i>Dorosoma cepedianum</i>                                       |
| Muskellunge                  | <i>Esox masquinongy</i>  |
| Perch (white)                | <i>Morone americana</i>  |
| Perch (yellow)               | <i>Perca flavescens</i>  |
| Pike (northern)              | <i>Esox lucius</i>   |
| Sheepshead (freshwater drum) | <i>Aplodinotus grunniens</i>                                     |
| Sucker (white)               | <i>Catostomus commersoni</i>                                     |
| Sunfish                      | <i>Lepomis gibbosus</i>  |
| Trout (lake)                 | <i>Salvelinus namaycush</i>                                      |
| Trout (rainbow)              | <i>Oncorhynchus mykiss</i><br>(formerly <i>Salmo gairdneri</i> ) |
| Walleye                      | <i>Stizostedion vitreum vitreum</i>                              |

**A P P E N D I X   D**

**Field Collection and Preparation Protocols**

Field Protocol - Fish Tissue Sampling

1. Collect fish (Electrofishing, Seine, Gill Net, Rotenone, Angling, other) taking care not to contaminate specimens with gasoline, motor oil, sediment, or soil. Record method on Field Data Sheet.
2. Measure total length in MM of each specimen in sample. Record on Field Data Sheet. Weigh each specimen in sample to nearest gram. Record on Field Data Sheet.
3. Note general condition, tumors, lesions. Record on Field Data Sheet as needed.
4. Prepare sample:
  - A. Whole Fish - Wrap composite sample (or individual fish if necessary, for specific study) in clean, commercial (restaurant) grade aluminum foil allowing only the dull foil surface to contact fish tissue. Indicate sample type on Field Data Sheet.
  - B. Fillets - Rinse clean fillet knife with purified hexane labeled as suitable for pesticide residue analysis.
    1. Inland Waters - Remove entire edible portion (fillet) from both sides of each specimen and remove skin. Wrap composite sample (or individual fish samples if necessary) in clean aluminum foil (dull side in contact with fish). Indicate sample type on Field Data Sheet.
    2. Lake Erie - Follow above procedure, but do not remove skin. Scale each specimen prior to filleting and leave skin on fillet. This complies with our agreement with the EPA Great Lakes National Program Office and the other Great Lakes states to provide uniform methodologies for Great Lakes tissue samples.
5. Clearly label each sample with the station number or water body name and location, date, time, and collector number (if necessary).
6. Place foil wrapped sample in a food grade protective plastic bag and freeze sample immediately on dry ice, if possible.
7. Be sure Field Data Sheet has been completed.



**FIELD DATA SHEET**  
**Tissue Sampling - Commonwealth of Pennsylvania**

Station # \_\_\_\_\_ Water Body: \_\_\_\_\_ Date: \_\_\_\_\_

Location: \_\_\_\_\_

County: \_\_\_\_\_ Municipality: \_\_\_\_\_

Collector: \_\_\_\_\_ Agency: \_\_\_\_\_ Coll.# \_\_\_\_\_

Method: Electrofishing ( ) Seine ( ) Gill Net ( ) Rotenone ( )  
 Angling ( ) Other ( ): \_\_\_\_\_

Reason: \_\_\_\_\_

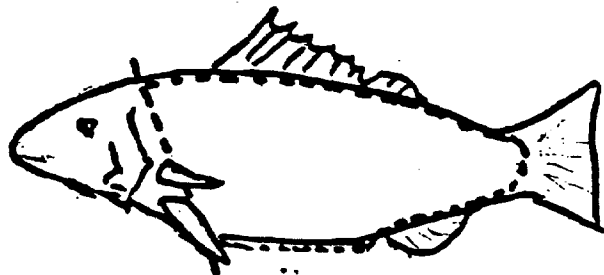
| <u>SPECIES</u> | <u>TL-MM</u> | <u>WT-G</u> | <u>*CONDITION</u> |
|----------------|--------------|-------------|-------------------|
| 1. _____       | _____        | _____       | _____             |
| 2. _____       | _____        | _____       | _____             |
| 3. _____       | _____        | _____       | _____             |
| 4. _____       | _____        | _____       | _____             |
| 5. _____       | _____        | _____       | _____             |
| 6. _____       | _____        | _____       | _____             |
| 7. _____       | _____        | _____       | _____             |
| 8. _____       | _____        | _____       | _____             |
| 9. _____       | _____        | _____       | _____             |
| 10. _____      | _____        | _____       | _____             |

\*Note tumors, lesions, & general condition (if needed).

Tissue Type: Whole Fish ( ) Skinless Fillet ( ) Skin-on Fillet ( )  
 Scaled (Y or N) ( )  
 Blood ( ) Organ ( ): \_\_\_\_\_  
 Other ( ): \_\_\_\_\_

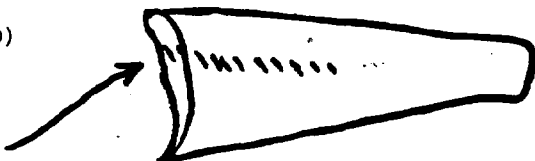
Comments (water/weather conditions, man-hours expended, problems etc.)

FILLETS USED BY ERIE COUNTY DEPARTMENT OF HEALTH



SKIN ON FILLET FOR FISH WITH ANTERIOR PELVIC FINS

(SCALES REMOVED)



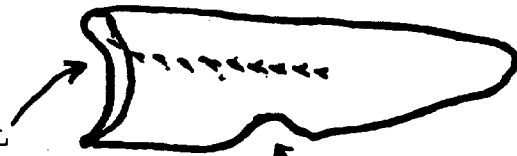
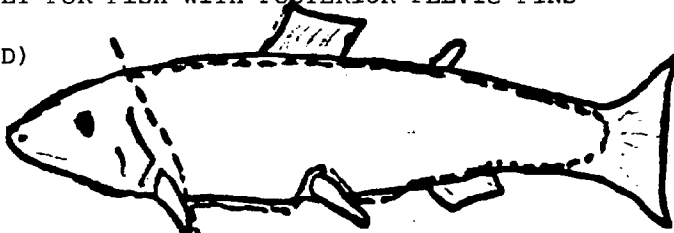
NOTE SOME SMALL IMBEDDED BONES REMAIN IN FILLET BUT TRUE RIBS ARE REMOVED

EG YELLOW PERCH  
BASS

---

\*SKIN ON FILLET FOR FISH WITH POSTERIOR PELVIC FINS

(SCALES REMOVED)

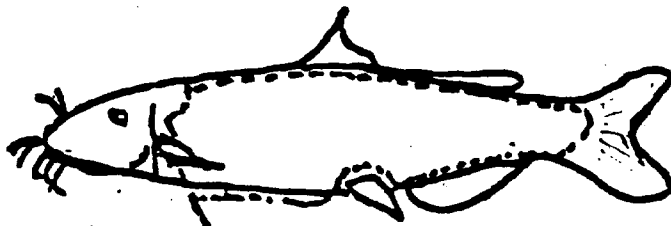


NOTE SOME SMALL BONES REMAIN IN FILLET TRUE RIBS ARE REMOVED NOTE PELVIC FINS REMOVED

EG NORTHERN PIKE  
RAINBOW TROUT  
LAKE TROUT

---

SKIN ON FILLET FOR MEMBERS OF THE CATFISH FAMILY



EG CATFISH  
BULLHEAD

NOTE PELVIC FINS REMOVED

**A P P E N D I X E**

**Study Areas, Lengths and Weights of Fish,  
Collection Methods and Dates**

Species, collection location, length and weight of Presque Isle Bay fish submitted for chemical analysis

S T U D Y   A R E A   A

| <u>Sample #</u> | <u>Date Collected</u> | <u>Species</u> | <u>Method of Collection</u> | <u>Length</u><br>in.      mm | <u>Weight</u><br>lb./oz.      grams |
|-----------------|-----------------------|----------------|-----------------------------|------------------------------|-------------------------------------|
| 0692266         | 10/20/87              | Walleye        | Gill Net                    | 21      533                  | 3 lb. 8 oz.      1,589              |
|                 |                       |                |                             | 27      686                  | 6 lb. 10 oz.      3,008             |
|                 |                       |                |                             | 22      559                  | 3 lb. 8 oz.      1,589              |
|                 |                       |                |                             | 20      508                  | 2 lb. 15 oz.      1,334             |
| 0692267         | 10/20/87              | Northern Pike  | Gill Net                    | 34      864                  | 7 lb. 3 oz.      3,263              |
|                 |                       |                |                             | 32      813                  | 7 lb. 3 oz.      3,263              |
|                 |                       |                |                             | 25.5      648                | 3 lb.      1,362                    |
|                 |                       |                |                             | 28      711                  | 4 lb. 1 oz.      1,844              |
|                 |                       |                |                             | 31      787                  | 6 lb.      2,724                    |
| 0692268         | 10/20/87              | Yellow Perch   | Gill Net                    | 8.5      216                 | 4 oz.      113                      |
|                 |                       |                |                             | 11.5      292                | 10 oz.      283                     |
|                 |                       |                |                             | 8.5      216                 | 4 oz.      113                      |
|                 |                       |                |                             | 7.5      191                 | 3 oz.      85                       |
|                 |                       |                |                             | 9      229                   | 4.5 oz.      127                    |
| 0692269         | 10/20/87              | Black Crappie  | Gill Net                    | 9      229                   | 6 oz.      170                      |
|                 |                       |                |                             | 8      203                   | 5 oz.      142                      |
|                 |                       |                |                             | 7.75      197                | 4 oz.      113                      |
|                 |                       |                |                             | 8      203                   | 5 oz.      142                      |
| 0692276         | 5/4/88                | White Sucker   | Hoop Net                    | 18.5      470                | 2 lb. 1 oz.      936                |
|                 |                       |                |                             | 16.45      418               | 1 lb. 8 oz.      681                |
|                 |                       |                |                             | 14.2      361                | 15 oz.      425                     |
|                 |                       |                |                             | 16.4      417                | 1 lb. 12 oz.      795               |
|                 |                       |                |                             | 15.27      388               | 1 lb. 3 oz.      539                |

Species, collection location, length and weight of Presque Isle Bay fish submitted for chemical analysis

S T U D Y   A R E A   A

| <u>Sample #</u> | <u>Date Collected</u> | <u>Species</u>  | <u>Method of Collection</u> | <u>Length in.</u> | <u>mm</u> | <u>lb./oz.</u> | <u>Weight grams</u> |
|-----------------|-----------------------|-----------------|-----------------------------|-------------------|-----------|----------------|---------------------|
| 0692277         | 5/4/88                | Brown Bullhead  | Hoop Net                    | 13.46             | 342       | 1 lb.          | 4 oz. 562           |
|                 |                       |                 |                             | 12.8              | 325       |                | 15 oz. 430          |
|                 |                       |                 |                             | 12.7              | 323       |                | 15.6 oz. 442        |
|                 |                       |                 |                             | 12.2              | 310       |                | 13.7 oz. 390        |
|                 |                       |                 |                             | 12.2              | 310       |                | 14 oz. 400          |
| 0692279         | 5/12/88               | Yellow Perch    | Gill Net                    | 8.66              | 220       |                | 2.8 oz. 80          |
|                 |                       |                 |                             | 9.25              | 235       |                | 3.6 oz. 103         |
|                 |                       |                 |                             | 9.84              | 250       |                | 4.9 oz. 140         |
|                 |                       |                 |                             | 9                 | 230       |                | 3.5 oz. 100         |
|                 |                       |                 |                             | 9                 | 230       |                | 2.8 oz. 80          |
| 0692280         | 5/12/88               | Smallmouth Bass | Gill Net                    | 17.1              | 435       | 2 lb.          | 10 oz. 1,192        |
|                 |                       |                 |                             | 17.5              | 445       | 2 lb.          | 7 oz. 1,107         |
|                 |                       |                 |                             | 18.1              | 460       | 3 lb.          | 2 oz. 1,419         |
|                 |                       |                 |                             | 16.5              | 420       | 2 lb.          | 5 oz. 1,050         |
| 0692281         | 5/12/88               | Muskellunge     | Gill Net                    | 35.2              | 895       | 10 lb.         | 12 oz. 4,881        |
| 0692282         | 5/12/88               | White Perch     | Gill Net                    | 9.8               | 250       |                | 9 oz. 255           |
|                 |                       |                 |                             | 11.4              | 290       |                | 15 oz. 425          |
|                 |                       |                 |                             | 9.4               | 240       |                | 8 oz. 227           |
|                 |                       |                 |                             | 8.66              | 220       |                | 7 oz. 198           |
|                 |                       |                 |                             | 8.8               | 225       |                | 6 oz. 170           |
| 0692283         | 5/12/88               | Gizzard Shad    | Gill Net                    | 15.74             | 400       | 1 lb.          | 4 oz. 568           |
|                 |                       |                 |                             | 15.74             | 400       | 1 lb.          | 4 oz. 568           |
|                 |                       |                 |                             | 17.7              | 450       | 1 lb.          | 13 oz. 823          |

Species, collection location, length and weight of Presque Isle Bay fish submitted for chemical analysis

S T U D Y   A R E A   A

| <u>Sample #</u> | <u>Date Collected</u> | <u>Species</u>  | <u>Method of Collection</u> | <u>Length</u> |           | <u>Weight</u>  |              |
|-----------------|-----------------------|-----------------|-----------------------------|---------------|-----------|----------------|--------------|
|                 |                       |                 |                             | <u>in.</u>    | <u>mm</u> | <u>lb./oz.</u> | <u>grams</u> |
| 0692284         | 6/22/88               | Yellow Perch    | Gill Net                    | 9.2           | 233       | 5.2 oz.        | 148          |
|                 | 6/30/89               |                 |                             | 9.5           | 242       | 6.2 oz.        | 175          |
| 0692285         | 6/30/88               | Channel Catfish | Gill Net                    | 18.8          | 478       | 2 lb. 8 oz.    | 1,135        |
|                 |                       |                 |                             | 22.2          | 564       | 2 lb. 7 oz.    | 1,107        |
|                 |                       |                 |                             | 21.45         | 545       | 3 lb. 8 oz.    | 1,589        |
| 0692286         | 6/22/88               | Carp            | Gill Net                    | 17.7          | 450       | 2 lb. 8 oz.    | 1,135        |
|                 |                       |                 |                             | 20.08         | 510       | 3 lb. 4.5 oz.  | 1,490        |
| 0692287         | 5/12/88               | Walleye         | Gill Net                    | 25.2          | 640       | 5 lb. 13 oz.   | 2,639        |
|                 |                       |                 |                             | 24.8          | 630       | 5 lb. 15 oz.   | 2,696        |
|                 |                       |                 |                             | 20.5          | 520       | 3 lb. 9 oz.    | 1,617        |
|                 |                       |                 |                             | 23.8          | 605       | 4 lb. 15 oz.   | 2,242        |
|                 |                       |                 |                             | 21.25         | 540       | 3 lb. 13 oz.   | 1,731        |
| 0692288         | 5/4/88                | Largemouth Bass | Hoop Net                    | 12.6          | 320       | 14 oz.         | 398          |
| 0692289         | 5/4/88                | Bluegill        | Hoop Net                    | 7.7           | 195       | 5 oz.          | 138          |
|                 |                       |                 |                             | 7.4           | 188       | 4 oz.          | 122          |
|                 |                       |                 |                             | 8.3           | 210       | 7 oz.          | 200          |
|                 |                       |                 |                             | 7.2           | 184       | 4 oz.          | 110          |

Species, collection location, length and weight of Presque Isle Bay fish submitted for chemical analysis

S T U D Y   A R E A   B

| <u>Sample #</u> | <u>Date Collected</u> | <u>Species</u> | <u>Method of Collection</u> | <u>Length in.</u> | <u>Length mm</u> | <u>lb./oz.</u> | <u>Weight grams</u> |
|-----------------|-----------------------|----------------|-----------------------------|-------------------|------------------|----------------|---------------------|
| 0692278         | 5/4/88                | Sunfish        | Hoop Net                    | 7.24              | 184              | 4.58 oz.       | 130                 |
|                 |                       |                |                             | 6.89              | 175              | 4.51 oz.       | 128                 |
|                 |                       |                |                             | 6.89              | 175              | 4.44 oz.       | 126                 |
|                 |                       |                |                             | 7.28              | 185              | 5.2 oz.        | 148                 |
|                 |                       |                |                             | 7.04              | 179              | 4.23 oz.       | 120                 |

Species, collection location, length and weight of Presque Isle Bay fish submitted for chemical analysis

S T U D Y   A R E A   C

| <u>Sample #</u> | <u>Date Collected</u> | <u>Species</u> | <u>Method of Collection</u> | <u>Length</u><br>in.      .mm | <u>Weight</u><br>lb./oz.      grams |
|-----------------|-----------------------|----------------|-----------------------------|-------------------------------|-------------------------------------|
| 0692272         | 3/2/88                | Bluegill       | Angling                     | 7.2      183                  | 4.2      oz.      118               |
|                 |                       |                |                             | 7.4      188                  | 5.2      oz.      148               |
|                 |                       |                |                             | 6.9      175                  | 3.8      oz.      108               |
|                 |                       |                |                             | 7.5      191                  | 4.6      oz.      130               |
|                 |                       |                |                             | 7.8      198                  | 5.6      oz.      160               |
| 0692273         | 2/17/88               | Yellow Perch   | Angling                     | 9.25      235                 | 5.4      oz.      154               |
|                 |                       |                |                             | 7.1      180                  | 2.5      oz.      72                |
|                 |                       |                |                             | 7.5      191                  | 2.7      oz.      78                |
|                 |                       |                |                             | 7.25      184                 | 2.25      oz.      64               |
|                 |                       |                |                             | 7.25      184                 | 2.3      oz.      65                |



Species, collection location, length and weight of Trout Run (Lake Erie) fish submitted for chemical analysis

S T U D Y   A R E A   D

| <u>Sample #</u> | <u>Date Collected</u> | <u>Species</u> | <u>Method of Collection</u> | <u>Length in.</u> | <u>Length mm</u> | <u>lb./oz.</u> | <u>Weight grams</u> |
|-----------------|-----------------------|----------------|-----------------------------|-------------------|------------------|----------------|---------------------|
| 0692270         | 11/19/87              | Rainbow Trout  | Netted                      | 24.3              | 617              | 6 lb. 3 oz.    | 2,810               |
|                 |                       |                |                             | 21.93             | 557              | 4 lb.          | 1,820               |
|                 |                       |                |                             | 23.03             | 585              | 4 lb. 3 oz.    | 1,900               |
|                 |                       |                |                             | 22.64             | 575              | 4 lb. 12 oz.   | 2,160               |
|                 |                       |                |                             | 23.07             | 586              | 5 lb.          | 2,280               |
| 0692271         | 11/19/87              | Rainbow Trout  | Netted                      | 17.5              | 445              | 1 lb. 15 oz.   | 900                 |
|                 |                       |                |                             | 15.4              | 392              | 1 lb. 7 oz.    | 660                 |
|                 |                       |                |                             | 15.7              | 400              | 1 lb. 11 oz.   | 780                 |
|                 |                       |                |                             | 16.3              | 415              | 1 lb. 9 oz.    | 720                 |
|                 |                       |                |                             | 18.2              | 463              | 2 lb. 3 oz.    | 1,000               |

Species, collection location, length and weight of Lake Erie fish submitted for chemical analysis

S T U D Y   A R E A   E  
(35' depth)

| Sample # | Date Collected | Species         | Method of Collection | Length |     | Weight       |       |
|----------|----------------|-----------------|----------------------|--------|-----|--------------|-------|
|          |                |                 |                      | in.    | mm  | lb./oz.      | grams |
| 0692260  | 10/20/87       | Yellow Perch    | Gill Net             | 11.5   | 292 | 10 oz.       | 283   |
|          |                |                 |                      | 11.25  | 286 | 9 oz.        | 255   |
|          |                |                 |                      | 10.0   | 254 | 6 oz.        | 170   |
|          |                |                 |                      | 11.0   | 279 | 9 oz.        | 255   |
|          |                |                 |                      | 11.0   | 279 | 9 oz.        | 255   |
| 0692261  | 10/20/87       | Walleye         | Gill Net             | 27.5   | 699 | 7 lb. 9 oz.  | 3,433 |
|          |                |                 |                      | 26.0   | 660 | 6 lb. 10 oz. | 3,008 |
| 0692262  | 10/20/87       | Smallmouth Bass | Gill Net             | 20.0   | 508 | 4 lb. 12 oz. | 2,157 |
|          |                |                 |                      | 21.75  | 552 | 5 lb. 2 oz.  | 2,327 |
|          |                |                 |                      | 18.0   | 457 | 3 lb. 3 oz.  | 1,447 |
|          |                |                 |                      | 19.0   | 483 | 3 lb. 3 oz.  | 1,447 |
|          |                |                 |                      | 19.0   | 483 | 3 lb.        | 1,362 |
| 0692263  | 10/20/87       | Sheepshead      | Gill Net             | 18.0   | 457 | 2 lb. 13 oz. | 1,277 |
| 0692264  | 10/20/87       | Lake Trout      | Gill Net             | 32.75  | 832 | 10 lb. 9 oz. | 4,795 |
| 0692265  | 10/20/87       | Lake Trout      | Gill Net             | 23.5   | 597 | 4 lb. 6 oz.  | 1,986 |

Species, collection location, length and weight of Lake Erie fish submitted for chemical analysis

S T U D Y   A R E A   E

(64' depth)

| <u>Sample #</u> | <u>Date Collected</u> | <u>Species</u> | <u>Method of Collection</u> | <u>Length in.</u> | <u>Length mm</u> | <u>1b./oz.</u> | <u>Weight grams</u> |
|-----------------|-----------------------|----------------|-----------------------------|-------------------|------------------|----------------|---------------------|
| 0692291         | 7/8/88                | Yellow Perch   | Gill Net                    | 10.1              | 257              | 6.7 oz.        | 190                 |
|                 |                       |                |                             | 9.8               | 249              | 7.04 oz.       | 200                 |
|                 |                       |                |                             | 9.7               | 246              | 5.46 oz.       | 155                 |
|                 |                       |                |                             | 9.2               | 234              | 5.46 oz.       | 155                 |
|                 |                       |                |                             | 9.2               | 234              | 5.64 oz.       | 160                 |

**A P P E N D I X F**

**Sample Results  
Organic and Inorganic,  
Percent Moisture and Percent Lipids.**

Concentrations of elemental and organic contaminants in edible fillet samples collected from Presque Isle Bay area (results in ppm wet weight\*)

**S T U D Y   A R E A   A**

Sample Number 0692\_\_ and Species\*\*

|                    | 266<br>Walleye | 267<br>Northern<br>Pike | 268<br>Yellow<br>Perch | 269<br>Black<br>Crappie | 276<br>White<br>Suckers |
|--------------------|----------------|-------------------------|------------------------|-------------------------|-------------------------|
| %Lipid             | 1.36           | .81                     | .21                    | .58                     | .32                     |
| %Moisture          | 78.5           | 78.4                    | 75.8                   | 77.6                    | 75.6                    |
| <u>Elements</u>    |                |                         |                        |                         |                         |
| Lead***            | 0.623          | 0.599                   | 0.530                  | 0.478                   | 0.162                   |
| Cadmium            | <0.013         | 0.015                   | 0.014                  | <0.013                  | 0.017                   |
| Chromium           | 0.187          | 0.187                   | 0.202                  | 0.214                   | 0.212                   |
| Arsenic            | <0.5           | <0.5                    | <0.5                   | <0.5                    | <0.5                    |
| Copper             | <0.5           | <0.5                    | <0.5                   | <0.5                    | <0.5                    |
| Selenium           | 0.277          | 0.294                   | 0.437                  | 0.327                   | 0.427                   |
| Antimony           | <1.25          | <1.25                   | <1.25                  | <1.25                   | <1.25                   |
| Silver             | <0.05          | <0.05                   | <0.05                  | <0.05                   | <0.05                   |
| Barium             | <0.25          | <0.25                   | <0.25                  | <0.25                   | 0.299                   |
| Beryllium          | <0.025         | <0.025                  | <0.025                 | <0.025                  | <0.025                  |
| Mercury            | 0.253          | <0.1                    | <0.1                   | <0.1                    | <0.1                    |
| <u>Organics</u>    |                |                         |                        |                         |                         |
| PCB's              | .13            | ND                      | ND                     | ND                      | ND                      |
| Chlordane          | .061           | ND                      | ND                     | ND                      | ND                      |
| Toxaphene          | ND             | ND                      | ND                     | ND                      | ND                      |
| pp'DDE             | .021           | .020                    | ND                     | ND                      | ND                      |
| pp'-DDD            | ND             | ND                      | ND                     | ND                      | ND                      |
| pp'-DDT            | ND             | ND                      | ND                     | ND                      | ND                      |
| Aldrin             | ND             | ND                      | ND                     | ND                      | ND                      |
| Dieldrin           | ND             | ND                      | ND                     | ND                      | ND                      |
| Endrin             | ND             | ND                      | ND                     | ND                      | ND                      |
| Lindane            | ND             | ND                      | ND                     | ND                      | ND                      |
| Heptachlor         | ND             | ND                      | ND                     | ND                      | ND                      |
| Heptachlor epoxide | ND             | ND                      | ND                     | ND                      | ND                      |
| Methoxychlor       | ND             | ND                      | ND                     | ND                      | ND                      |
| Mirex              | ND             | ND                      | ND                     | ND                      | ND                      |

ND = not detected (see Appendix J for information on detection limits)

ppm = parts per million - reported on lab sheets as micrograms/gram (= milligrams/kilogram)

\*wet weight = as received basis, i.e., what a consumer would buy in a market

\*\*All scaled species of fish consisted of skin left on but scales removed fillets (skin was also left on the catfish family samples)

\*\*\*See special section on lead analysis difficulties, page 39 and Appendix G

Concentrations of elemental and organic contaminants in edible fillet samples collected from Presque Isle Bay area (results in ppm wet weight)

S T U D Y   A R E A   A

Sample Number 0692\_\_ and Species

|                    | 277<br>Brown<br>Bullhead<br>(Note:<br>skin-on<br>fillets) | 280<br>Small<br>Mouth | 281<br>Muskel-<br>lunge | 282<br>White<br>Perch |
|--------------------|---|-----------------------|-------------------------|-----------------------|
| %Lipid             | 2.19  | 2.09                  | 1.09                    | 7.37                  |
| %Moisture          | 78.4  | 77.4                  | 75.5                    | 71.9                  |
| <u>Elements</u>    |   |                       |                         |                       |
| Lead               | 0.525   | 0.552                 | 0.176                   | 1.25                  |
| Cadmium            | 0.029   | 0.034                 | 0.014                   | 0.038                 |
| Chromium           | 0.175   | <0.125                | 0.264                   | 0.663                 |
| Arsenic            | <0.5  | <0.5                  | <0.5                    | <0.5                  |
| Copper             | <0.5  | <0.5                  | <0.5                    | 1.7                   |
| Selenium           | 0.492   | 0.610                 | 0.334                   | 0.630                 |
| Antimony           | <1.25   | <1.25                 | <1.25                   | <1.25                 |
| Silver             | <0.05   | <0.05                 | <0.05                   | <0.05                 |
| Barium             | 0.35  | 0.274                 | <0.25                   | 0.375                 |
| Beryllium          | <0.025  | <0.025                | <0.025                  | <0.025                |
| Mercury            | <0.1  | 0.125                 | 0.135                   | 0.1                   |
| <u>Organics</u>    |   |                       |                         |                       |
| PCB's              | .690  | .270                  | ND                      | *                     |
| Chlordane          | .230  | ND                    | ND                      | .170                  |
| Toxaphene          | ND  | ND                    | ND                      | ND                    |
| pp'-DDE            | .070  | .071                  | ND                      | .052                  |
| pp'-DDD            | .060  | ND                    | ND                      | ND                    |
| pp'-DDT            | ND  | ND                    | ND                      | ND                    |
| Aldrin             | ND  | ND                    | ND                      | ND                    |
| Dieldrin           | ND  | ND                    | ND                      | **                    |
| Endrin             | ND  | ND                    | ND                      | ND                    |
| Lindane            | ND  | ND                    | ND                      | ND                    |
| Heptachlor         | ND  | ND                    | ND                      | ND                    |
| Heptachlor epoxide | ND  | ND                    | ND                      | ND                    |
| Methoxychlor       | ND  | ND                    | ND                      | ND                    |
| Mirex              | ND  | ND                    | ND                      | ND                    |

ND = not detected

\*possible trace, estimated at .170

\*\*possible trace, estimated at .013

Concentrations of elemental and organic contaminants in edible fillet samples collected from Presque Isle Bay area (results in ppm wet weight)

**S T U D Y   A R E A   A**

Sample Number 0692\_\_\_ and Species

|                    | 283<br>Gizzard<br>Shad | 284<br>Yellow<br>Perch | 285*<br>Channel<br>Catfish<br>(Note:<br>skin on<br>fillets) | 286<br>Carp | 287<br>Walleye |
|--------------------|------------------------|------------------------|---|-------------|----------------|
| %Lipid             | 12.67                  | 0.24                   | 9.73  | 8.33        | 5.86           |
| %Moisture          | 71.8                   | 76.7                   | 69.4  | 75.9        | 75.2           |
| <u>Elements</u>    |                        |                        |   |             |                |
| Lead               | 0.716                  | 0.797                  | 1.69  | 0.853       | 1.2            |
| Cadmium            | <0.013                 | 0.034                  | 0.034   | 0.035       | 0.019          |
| Chromium           | 0.272                  | 0.511                  | 0.717   | 0.702       | 0.451          |
| Arsenic            | <0.5                   | <0.5                   | <0.5  | <0.5        | <0.5           |
| Copper             | 1.11                   | 0.847                  | <0.5  | <0.5        | 0.801          |
| Selenium           | 0.568                  | 0.272                  | 0.269   | 0.439       | 0.636          |
| Antimony           | <1.25                  | <1.25                  | <1.25   | <1.25       | <1.25          |
| Silver             | <0.05                  | <0.05                  | <0.05   | <0.05       | <0.05          |
| Barium             | <0.25                  | 0.349                  | 0.553   | 0.251       | <0.25          |
| Beryllium          | <0.025                 | <0.025                 | <0.025  | <0.025      | <0.025         |
| Mercury            | <0.1                   | 0.108                  | 0.18  | 0.205       | <0.1           |
| <u>Organics</u>    |                        |                        |   |             |                |
| PCB's              | 1.2                    | ND                     | .920  | 1.000       | .190           |
| Chlordane          | 0.53                   | ND                     | .720  | .560        | .220           |
| Toxaphene          | ND                     | ND                     | ND  | ND          | ND             |
| pp'DDE             | 0.21                   | ND                     | .240  | .200        | .070           |
| pp'-DDD            | 0.069                  | ND                     | .130  | .040        | ND             |
| pp'-DDT            | ND                     | ND                     | ND  | ND          | ND             |
| Aldrin             | ND                     | ND                     | ND  | ND          | ND             |
| Dieldrin           | ND                     | ND                     | ND  | ND          | ND             |
| Endrin             | ND                     | ND                     | ND  | ND          | ND             |
| Lindane            | ND                     | ND                     | ND  | ND          | ND             |
| Heptachlor         | ND                     | ND                     | ND  | ND          | ND             |
| Heptachlor epoxide | ND                     | ND                     | ND  | ND          | ND             |
| Methoxychlor       | ND                     | ND                     | ND  | ND          | ND             |
| Mirex              | ND                     | ND                     | ND  | ND          | ND             |

ND = not detected

\*See results - Michigan CZM 32, duplicate analysis, right fillets of same fish

Concentrations of elemental and organic contaminants in edible fillet samples collected from Presque Isle Bay area (results in ppm wet weight)

**S T U D Y   A R E A   A**

Sample Number 0692\_\_ and Species

|                    | 288*<br>Large<br>Mouth<br>Bass | 289<br>Bluegill |
|--------------------|--------------------------------|-----------------|
| %Lipid             | 1.01                           |                 |
| %Moisture          | 78.8                           | 79.5            |
| <u>Elements</u>    |                                |                 |
| Lead               | 1.04                           | 2.35            |
| Cadmium            | 0.024                          | <0.013          |
| Chromium           | 0.401                          | 0.574           |
| Arsenic            | <0.5                           | <0.5            |
| Copper             | 0.652                          | 1.2             |
| Selenium           | 0.391                          | 0.432           |
| Antimony           | <1.25                          | <1.25           |
| Silver             | <0.05                          | <0.05           |
| Barium             | 0.652                          | 1.20            |
| Beryllium          | <0.025                         | <0.025          |
| Mercury            | <0.1                           | 0.141           |
| <u>Organics</u>    |                                |                 |
| PCB's              | ND                             |                 |
| Chlordane          | .320                           |                 |
| Toxaphene          | ND                             |                 |
| pp'DDE             | .030                           |                 |
| pp'-DDD            | ND                             |                 |
| pp'-DDT            | ND                             |                 |
| Aldrin             | ND                             |                 |
| Dieldrin           | ND                             |                 |
| Endrin             | ND                             |                 |
| Lindane            | ND                             |                 |
| Heptachlor         | ND                             |                 |
| Heptachlor epoxide | ND                             |                 |
| Methoxychlor       | ND                             |                 |
| Mirex              | ND                             |                 |

ND = not detected

\*See results - Michigan CZM 18, duplicate analysis, right fillet of same fish



Concentrations of elemental and organic contaminants in edible fillet samples collected from Presque Isle Bay area (results in ppm wet weight)

S T U D Y   A R E A   B

Sample Number 0692\_\_ and Species

---

278  
Sunfish

---

%Lipid .32  
%Moisture 76.0

Elements

Lead 1.18  
Cadmium 0.031  
Chromium 0.125  
Arsenic <0.5  
Copper <0.5  
Selenium 0.387  
Antimony <1.25  
Silver <0.05  
Barium <0.25  
Beryllium <0.025  
Mercury 0.170

Organics

PCB's ND  
Chlordane ND  
Toxaphene ND  
pp'DDE ND  
pp'-DDD ND  
pp'-DDT ND  
Aldrin ND  
Dieldrin ND  
Endrin ND  
Lindane ND  
Heptachlor ND  
Heptachlor epoxide ND  
Methoxychlor ND  
Mirex ND

ND = not detected

Concentrations of elemental and organic contaminants in edible fillet samples collected from Presque Isle Bay area (results in ppm wet weight)

S T U D Y   A R E A   C

Sample Number 0692\_\_ and Species

|                    | 272<br>Bluegill | 273<br>Yellow<br>Perch |
|--------------------|-----------------|------------------------|
| %Lipid             | .2              | .16                    |
| %Moisture          | 79.8            | 75.9                   |
| <u>Elements</u>    |                 |                        |
| Lead               | 0.545           | 0.627                  |
| Cadmium            | 0.026           | 0.028                  |
| Chromium           | 0.396           | 0.589                  |
| Arsenic            | <0.5            | <0.5                   |
| Copper             | <0.5            | 3.66                   |
| Selenium           | 0.277           | 0.366                  |
| Antimony           | <1.25           | <1.25                  |
| Silver             | <0.05           | <0.05                  |
| Barium             | <0.25           | <0.25                  |
| Beryllium          | <0.025          | <0.025                 |
| Mercury            | 0.108           | <0.1                   |
| <u>Organics</u>    |                 |                        |
| PCB's              | ND              | ND                     |
| Chlordane          | ND              | ND                     |
| Toxaphene          | ND              | ND                     |
| pp'DDE             | ND              | ND                     |
| pp'-DDD            | ND              | ND                     |
| pp'-DDT            | ND              | ND                     |
| Aldrin             | ND              | ND                     |
| Dieldrin           | ND              | ND                     |
| Endrin             | ND              | ND                     |
| Lindane            | ND              | ND                     |
| Heptachlor         | ND              | ND                     |
| Heptachlor epoxide | ND              | ND                     |
| Methoxychlor       | ND              | ND                     |
| Mirex              | ND              | ND                     |

ND = not detected

Concentrations of elemental and organic contaminants in edible fillet samples collected from Trout Run, a tributary of Lake Erie (results in ppm wet weight)

**S T U D Y   A R E A   D**

Sample Number 0692 and Species

|                    | 270<br>Rainbow<br>Trout | 271<br>Rainbow<br>Trout |
|--------------------|-------------------------|-------------------------|
| %Lipid             | 7.56                    | 6.7                     |
| %Moisture          | 68.6                    | 80.6                    |
| <u>Elements</u>    |                         |                         |
| Lead               | 0.663                   | 0.843                   |
| Cadmium            | 0.013                   | 0.020                   |
| Chromium           | 0.253                   | 0.397                   |
| Arsenic            | <0.5                    | <0.5                    |
| Copper             | <0.5                    | <0.5                    |
| Selenium           | 0.366                   | 0.489                   |
| Antimony           | <1.25                   | <1.25                   |
| Silver             | <0.05                   | <0.05                   |
| Barium             | <0.25                   | <0.25                   |
| Beryllium          | not run                 | <0.025                  |
| Mercury            | <0.1                    | 0.178                   |
| <u>Organics</u>    |                         |                         |
| PCB's              | 0.44                    | 0.24                    |
| Chlordane          | 0.28                    | 0.18                    |
| Toxaphene          | ND                      | ND                      |
| pp'DDE             | 0.095                   | 0.084                   |
| pp'-DDD            | ND                      | 0.018                   |
| pp'-DDT            | ND                      | ND                      |
| Aldrin             | ND                      | ND                      |
| Dieldrin           | ND                      | *                       |
| Endrin             | ND                      | ND                      |
| Lindane            | ND                      | ND                      |
| Heptachlor         | ND                      | ND                      |
| Heptachlor epoxide | ND                      | ND                      |
| Methoxychlor       | ND                      | ND                      |
| Mirex              | ND                      | ND                      |

ND = not detected

\*possible trace, estimated at .012

Concentrations of elemental and organic contaminants in edible fillet samples collected from Lake Erie off Shades Beach (results in ppm wet weight)

**S T U D Y   A R E A   E**

Sample Number 0692\_\_ and Species

|                    | 260<br>Yellow<br>Perch | 261<br>Walleye | 262<br>Small<br>Mouth<br>Bass | 263<br>Sheeps-<br>head |
|--------------------|------------------------|----------------|-------------------------------|------------------------|
| %Lipid             | 0.15                   | 5.39           | 4.16                          | 11.88                  |
| %Moisture          | 78.7                   | 74.8           | 75.5                          | 68.5                   |
| <u>Elements</u>    |                        |                |                               |                        |
| Lead               | 0.758                  | 0.410          | <0.125                        | 0.785                  |
| Cadmium            | 0.015                  | 0.019          | <0.013                        | 0.0137                 |
| Chromium           | 0.386                  | 0.348          | 0.226                         | 0.224                  |
| Arsenic            | <0.5                   | <0.5           | <0.5                          | <0.5                   |
| Copper             | <0.5                   | <0.5           | <0.5                          | <0.5                   |
| Selenium           | <0.25                  | 0.463          | 0.819                         | 0.377                  |
| Antimony           | <1.25                  | <1.25          | <1.25                         | <1.25                  |
| Silver             | <0.05                  | <0.05          | <0.05                         | <0.05                  |
| Barium             | <0.25                  | <0.25          | <0.25                         | <0.25                  |
| Beryllium          | <0.025                 | <0.025         | <0.025                        | <0.025                 |
| Mercury            | 0.197                  | 0.171          | 0.306                         | 0.121                  |
| <u>Organics</u>    |                        |                |                               |                        |
| PCB's              | ND                     | 0.21           | 0.35                          | 0.46                   |
| Chlordane          | ND                     | 0.22           | 0.16                          | 0.15                   |
| Toxaphene          | ND                     | ND             | ND                            | ND                     |
| pp'DDE             | ND                     | 0.072          | 0.067                         | 0.058                  |
| pp'-DDD            | ND                     | 0.019          | 0.016                         | **                     |
| pp'-DDT            | ND                     | ND             | ND                            | ND                     |
| Aldrin             | ND                     | ND             | ND                            | ND                     |
| Dieldrin           | ND                     | *              | trace                         | ND                     |
| Endrin             | ND                     | ND             | ND                            | ND                     |
| Lindane            | ND                     | ND             | ND                            | ND                     |
| Heptachlor         | ND                     | ND             | ND                            | ND                     |
| Heptachlor epoxide | ND                     | ND             | ND                            | ND                     |
| Methoxychlor       | ND                     | ND             | ND                            | ND                     |
| Mirex              | ND                     | ND             | ND                            | ND                     |

ND = not detected

\*possible trace, estimated at .015  
 \*\*possible trace, estimated at .007

Concentrations of elemental and organic contaminants in edible fillet samples collected from Lake Erie off Shades Beach (results in ppm wet weight)

S T U D Y   A R E A   E

Sample Number 0692 \_\_\_ and Species

|                    | 264*<br>Lake<br>Trout<br>(First<br>Analysis) | 264<br>Lake<br>Trout<br>(Second<br>Analysis) | 265<br>Lake<br>Trout |
|--------------------|--|--|----------------------|
| %Lipid             | 10.91  | 9.63   | 11.55                |
| %Moisture          |  |  | 68.0                 |
| <u>Elements</u>    |  |  |                      |
| Lead               |  |  | 0.690                |
| Cadmium            |  |  | <0.0125              |
| Chromium           |  |  | 0.188                |
| Arsenic            |  |  | <0.5                 |
| Copper             |  |  | <0.5                 |
| Selenium           |  |  | 0.296                |
| Antimony           |  |  | <1.25                |
| Silver             |  |  | <0.05                |
| Barium             |  |  | <0.25                |
| Beryllium          |  |  | <0.025               |
| Mercury            |  |  | <0.1                 |
| <u>Organics</u>    |  |  |                      |
| PCB's              | 1.2  | 1.3  | 0.24                 |
| Chlordane          | 1.1  | 0.9  | 0.32                 |
| Toxaphene          | ND   | ND   | ND                   |
| pp'DDE             | 0.39   | 0.4  | 0.09                 |
| pp'-DDD            | 0.11   | 0.12   | ND                   |
| pp'-DDT            | ND   | ND   | ND                   |
| Aldrin             | ND   | ND   | ND                   |
| Dieldrin           | **   | ND   | ND                   |
| Endrin             | ND   | ND   | ND                   |
| Lindane            | ND   | ND   | ND                   |
| Heptachlor         | ND   | ND   | ND                   |
| Heptachlor epoxide | ND   | ND   | ND                   |
| Methoxychlor       | ND   | ND   | ND                   |
| Mirex              | ND   | ND   | ND                   |
| BHC                | ***  |  |                      |

ND = not detected

\*See results - Michigan CZM 6A, duplicate analysis, right fillet of same fish

\*\*estimated at 0.010

\*\*\*estimated at 0.012

Concentrations of elemental and organic contaminants in edible fillet samples collected from Lake Erie off Shades Beach (results in ppm wet weight)

S T U D Y   A R E A   E

Sample Number 0692\_\_\_ and Species

---

|                    |                        |
|--------------------|------------------------|
|                    | 291<br>Yellow<br>Perch |
| <hr/>              |                        |
| %Lipid             | 0.14                   |
| %Moisture          | 77.5                   |
| <u>Elements</u>    |                        |
| Lead               | 0.423                  |
| Cadmium            | 0.024                  |
| Chromium           | 0.361                  |
| Arsenic            | <0.5                   |
| Copper             | 0.373                  |
| Selenium           | 0.632                  |
| Antimony           | <1.25                  |
| Silver             | <0.05                  |
| Barium             | 0.373                  |
| Beryllium          | <0.025                 |
| Mercury            | 0.112                  |
| <u>Organics</u>    |                        |
| PCB's              | ND                     |
| Chlordane          | ND                     |
| Toxaphene          | ND                     |
| pp'DDE             | ND                     |
| pp'-DDD            | ND                     |
| pp'-DDT            | ND                     |
| Aldrin             | ND                     |
| Dieldrin           | ND                     |
| Endrin             | ND                     |
| Lindane            | ND                     |
| Heptachlor         | ND                     |
| Heptachlor epoxide | ND                     |
| Methoxychlor       | ND                     |
| Mirex              | ND                     |

ND = not detected

Table I

Fish fillets in descending order of % lipids ("fat") actual percentage of individual fish or study averages, where more than one fish of the same species was encountered from combined lake and/or bay samples

|                              |        |
|------------------------------|--------|
| Gizzard Shad                 | 12.67* |
| Sheepshead (Freshwater Drum) | 11.88  |
| Lake Trout                   | 11.23* |
| Channel Catfish              | 9.73*  |
| Carp                         | 8.33*  |
| White Perch                  | 7.37   |
| Rainbow Trout                | 7.13   |
| Walleye                      | 4.20   |
| Smallmouth Bass              | 4.16   |
| Muskellunge                  | 1.09   |
| Largemouth Bass              | 1.01*  |
| Northern Pike                | 0.81   |
| Black Crappie (Calico Bass)  | 0.58   |
| Sunfish (Pumpkinseed)        | 0.32   |
| White Sucker                 | 0.32   |
| Bluegill                     | 0.2    |
| Yellow Perch                 | 0.18   |

\*Fish fillets exceeding FDA "Technical Chlordane" action level as determined by the Pennsylvania DER (please read special discussion on laboratory procedures on quality assurance and interlaboratory differences in results)

**Table II**

Concentrations of PCB's found by the Pennsylvania Department of Environmental Resources. This study mirrors the Ontario Ministry of the Environment's "Relative Levels of PCB's in Lake Ontario Fish Species."<sup>14</sup>

|                                    | <u>PCB's</u>                  |
|------------------------------------|-------------------------------|
| Lake Trout (large)                 | 1.3 ppm                       |
| Carp                               | 1.0 ppm                       |
| Brown Trout                        | no information<br>on Pa. fish |
| Channel Catfish                    | 0.92 ppm                      |
| Brown Bullhead                     | 0.69 ppm                      |
| Rainbow Trout<br>(large composite) | 0.44 ppm                      |
| Northern Pike                      | none detected                 |
| Sheepshead<br>(Freshwater Drum)    | 0.46*                         |

\*Freshwater drum is the exception of the relative Ontario PCB concentrations. It may be due to the fact that only one fish was sampled in Pennsylvania and that fish may or may not reflect the average PCB concentrations.



**A P P E N D I X G**

**Lead Results - Walleye Sections**

LEAD RESULTS WALLEYE CZM329

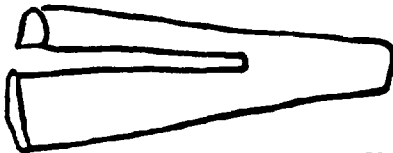
FIVE FISH COMPOSITE

A



ORIGINAL LEFT SIDE FILLETS  
SCALED - SKIN ON - NOT RINSED

B



RIGHT SIDE FILLETS  
SKIN REMOVED - WEDGE CUT OUT  
REMOVING SOME FLESH & ALL IMBEDDED  
BONES - RINSED IN TAP WATER

C



WEDGE REMOVED FROM ABOVE  
FLESH & BONES - NO SKIN-RINSED  
IN TAP WATER

D

RESIDUAL SKIN FROM THE 5 RIGHT FILLETS  
SOME "TIPS" FROM BONES - LOOSE SCALES  
& "SLIME" - NOT RINSED

ORIGINAL  
1988 RESULTS

RETEST IN 1989

| A         | B (FLESH) | C (WEDGE) | D (SKIN)  |
|-----------|-----------|-----------|-----------|
| 1.20 PPM  | 0.106 PPM | 0.091 PPM | 0.037 PPM |
| DUPLICATE |           |           |           |
| 1.75 PPM  |           |           |           |

**A P P E N D I X H**

**Preparing PCB Contaminated Fish  
for Human Consumption**

COMMONWEALTH OF PENNSYLVANIA  
DEPARTMENT OF ENVIRONMENTAL RESOURCES

PREPARING PCB CONTAMINATED FISH FOR HUMAN CONSUMPTION

Polychlorinated biphenyls (PCB's) are a group of very stable industrial chemicals used since 1929. PCB's were discovered in fish in the United States in 1967, and domestic production was banned in 1977. PCB's are, however, still used in closed electrical equipment. Because of their stability, PCB's do not break down in the environment and, in fact, tend to accumulate through the food chain.

The U.S. Food and Drug Administration establishes limits for deleterious substances in human food. The current FDA "Action Level" for PCB's in fish flesh is 2 parts per million (ppm). This standard became effective August 20, 1984. The level had previously been 5 ppm.

While the hazards of PCB consumption are largely undetermined, they are a suspected carcinogen. Therefore, anglers and others may wish to limit their consumption of fish containing PCB's. Pregnant women and those breast feeding their children may wish to avoid all food contaminated with PCB's. This information sheet does not recommend consumption of PCB contaminated fish. The consumption of contaminated fish is a matter of personal choice. The following guidelines are intended to help the angler and his family reduce their dietary intake of PCB's.

Ways to Limit Intake:

1. Learn to differentiate between stocked and stream-bred fish - Recently-stocked fish will contain less PCB than carryover or native fish. Stocked trout can be differentiated from native trout by observing the fish's color and fin size. Stocked trout will be a dull color and have much smaller, often misshapen or tattered fins.
2. Eat smaller, younger fish - PCB's are stored in fat tissue; therefore, the older, larger, and fattier a fish becomes, the more likely it will contain PCB's. A reduction in dietary intake of PCB's can be achieved by keeping smaller fish for the table.

3. Prepare and cook the fish properly - While the method of cooking does not appear to greatly affect PCB content, proper preparation and care of the fish before and during cooking will reduce the amount of PCB's consumed.
  - a. Remove the skin before cooking.
  - b. Remove fat - The dorsal layer of fat (under the dorsal fin along the fish's back), the belly fat, and a layer of fat along the lateral line (along the mid-line of the side of the fish) should be trimmed off. The Wisconsin Department of Natural Resources found that such trimming reduced PCB content of the fish by 30%.
  - c. The juices and fats that cook out of the fish should not be eaten or reused for cooking other foods. The cooked fish should not be left in the pan with its juices and fats.

Information concerning PCB contamination of particular fish species in specific streams in Pennsylvania is disseminated through press releases. It is also available in "PCB's in Pennsylvania Waters," Publication #51 of the Bureau of Water Quality Management, Department of Environmental Resources. The address is P.O. Box 2063, Harrisburg, PA, 17120. Free copies are available.

This information sheet was prepared by Donald F. Knorr, Water Pollution Biologist, in cooperation with the Division of Water Quality. Publication #12-3600(78) of the Wisconsin Department of Natural Resources, was the chief data source.

**A P P E N D I X I**

**Food and Drug Administration  
Guides - PCB's and Chlordane**

CHAPTER 8 - FISH AND SEAFOOD INDUSTRY

SUBJECT: Polychlorinated Biphenyls (PCB's) in Certain Freshwater Fish

BACKGROUND:

The Commissioner of Foods and Drugs promulgated a regulation (21 CFR 109.30a in the Federal Register of July 6, 1973 (38 F.R. 18096) that established limits (temporary tolerances) on the amounts of PCB's that may be lawfully present in food as a result of unavoidable contamination.

PCB's are a class of toxic industrial chemicals that are highly stable, heat resistant and nonflammable. One consequence of the contamination of the environment with PCB's is the indirect contamination of certain foods, principally those of animal origin. Surveillance data gathered by FDA and USDA subsequent to the effective date of the temporary tolerances have shown that, with the exception of certain freshwater fish, the presence of PCB's in those individual foods subject to the tolerance continues to be sporadic, and that there has been an overall and substantial decline in frequency and levels.

Fish collected from commercial establishments contained PCB's substantially below levels which would pose a risk to health. However, considerably higher levels were detected in various species of freshwater fish--particularly sports fish, e.g., coho and chinook salmon, lake trout--from several areas in the United States. The commissioner is concerned about the public health implications for sportfishermen and others who may regularly consume fish that are caught in PCB contaminated waters.

On October 20, 1975 the New York Department of Environmental Conservation was informed that FDA will initiate seizures of interstate shipments of fish found to exceed the temporary tolerance for PCB's and would support the state in similar action against intrastate shipments. Should additional regulatory actions become necessary in the future, FDA will promptly take all appropriate steps consistent with its statutory authority.

POLICY:

Even though the temporary tolerances for PCB's in selected commodities (21 CFR 109.30(a) are currently under review, they remain in effect for unavoidable contamination of food products.

Date: 10/01/80

PAGE 1

ISSUING OFFICE: EDRO, Division of Field Regulatory Guidance

AUTHORITY: Associate Commissioner for Regulatory Affairs

NOTE: The detailed "Statement of Policy", concerning PCB's in certain freshwater fish, appears in the Federal Register of February 26, 1976 (39 F.R. 8409/10).

CHLORDANE

The following action levels are for residues of chlordane, including heptachlor and its epoxide, cis and trans chlordane, cis and trans nonachlor, oxychlordane (octachlor epoxide), alpha, beta and gamma chlordene and chlordene. Levels of individual components must be quantitated at 0.02 ppm or above and confirmed in order to be added into the "chlordane" total value. Also see "analytical notes" at the bottom.

| <u>Commodity</u> <sup>a</sup>   | <u>Action Level (ppm)</u> |
|---|---------------------------|
| Animal fat, rendered  | 0.3                       |
| Animal feed, processed  | 0.1                       |
| Asparagus   | 0.1                       |
| Bananas   | 0.1                       |
| Beans   | 0.1                       |
| Beets (with or without tops)  | 0.1                       |
| Beet greens   | 0.1                       |
| Brassica (cole) leafy vegetables (except broccoli raab,<br>Chinese mustard cabbage, and rape greens)        | 0.1                       |
| Carrots   | 0.1                       |
| Celery  | 0.1                       |
| Citrus fruits   | 0.1                       |
| Corn  | 0.1                       |
| Cucumbers   | 0.1                       |
| Eggplant  | 0.1                       |
| Fish<br>portion)  | 0.3 (edible               |
| Lettuce   | 0.1                       |
| Melons  | 0.1                       |
| Okra  | 0.1                       |
| Onions  | 0.1                       |
| Papayas   | 0.1                       |
| Parsnips  | 0.1                       |
| Peanuts   | 0.1                       |
| Peas  | 0.1                       |
| Peppers   | 0.1                       |
| Pineapple   | 0.1                       |
| Pome fruits (except crabapples and loquats)   | 0.1                       |
| Potatoes  | 0.1                       |
| Radishes (with or without tops)   | 0.1                       |
| Radish tops   | 0.1                       |
| Rutabagas (with or without tops)  | 0.1                       |
| Rutabaga tops   | 0.1                       |
| Small fruits and berries (except cranberries, currants,<br>elderberries, gooseberries, and olallie berries) | 0.1                       |

DATE April 1, 1987



|   |     |
|---|-----|
| Spinach   | 0.1 |
| Squash  | 0.1 |
| Stone fruits (except Chickasaw, Damson, and Japanese plums) | 0.1 |
| Sweet potatoes  | 0.1 |
| Swiss chard   | 0.1 |
| Tomatoes  | 0.1 |
| Turnips (with or without tops)                              | 0.1 |
| Turnip greens   | 0.1 |

<sup>a</sup>Action levels for crop groups cover all commodities specified in 40 CFR 180.34(f), except where an exception is noted.

Analytical Notes:

- (1) When the GLC pattern of the residue matches that of technical chlordane, quantitate against a reference standard of technical chlordane. When the residue consists of identifiable individual components, quantitate against their respective reference standards and sum the components.
- (2) If heptachlor and/or its epoxide are proportionally higher than the amount of chlordane present, follow the guidance in PAM Volume I, section 300.64c and apply the action levels established specifically for heptachlor and heptachlor epoxide.

Date: April 1, 1987

**A P P E N D I X J**

**Quality Assurance Data**

**Comparisons to Other Agencies - Fish and Wildlife  
Service, U.S. Food and Drug Administration,  
U.S. Environmental Protection Agency and  
Michigan Department of Public Health**

## Quality Assurance/Quality Control Project Plan

### Organics Analysis

Pesticide/PCB analysis in fish flesh - work-up of samples:

1. Fish flesh samples will be prepared following procedure as described in the Method Section (see Methods Manual "Draft," Appendix D).
2. Samples will be analyzed by GC/ECD according to the following protocol:

When available, sets of 6 samples (including QA samples) will be run at a time. One set will consist of 4 samples, a spike and a duplicate; another set will consist of 4 (different) samples, a spike and a blank, as follows:

4 fish flesh samples  
1 fish flesh sample to be run as a duplicate  
1 fish flesh sample to be run as a spike

alternating with

4 fish flesh samples  
1 fish flesh sample to be run as a spike  
1 fish flesh sample to be run as a blank

Spiking will be performed as follows:

| <u>Parameter</u> | <u>Spiking Level (mg/kg)</u> |
|------------------|------------------------------|
| Aroclor 1260     | 0.5                          |
| Chlordane        | 0.1                          |
| Dieldrin         | 0.05                         |
| Toxaphene        | 1.0                          |

These parameters will be separately spiked into selected samples.

3. The Department of Environmental Resources will analyze for the following:

| <u>Parameter</u>   | <u>Approximate Reporting Limit (mg/kg wet wt.)</u> |
|--------------------|--|
| PCB's              | 0.2  |
| Chlordane          | 0.05   |
| Toxaphene          | 0.2  |
| pp'DDE             | 0.02   |
| pp'-DDD            | 0.02   |
| pp'-DDT            | 0.05   |
| Aldrin             | 0.1  |
| Dieldrin           | 0.02   |
| Endrin             | 0.02   |
| Lindane            | 0.01   |
| Heptachlor         | 0.02   |
| Heptachlor epoxide | 0.02   |
| Methoxychlor       | 0.1  |
| Mirex              | 0.02   |

Additional pesticides will be identified and/or quantified as matrix conditions permit.

Percent lipids will also be determined and reported.

Precision for the above parameters in fish flesh can be determined from the duplicate samples; accuracy can be estimated from analysis of samples with known concentrations of parameters.

4. Data from fish flesh duplicates and blanks are to be written on one of the sheets of the sample sets.

The lab will supply its own blanks that contain no demonstrable pesticides/PCB's. The lab will also do duplicate analysis of selected fish.

## Metals Analysis

1. Sample preparation for metal analysis of fish tissue
  - A. Samples will be analytically weighed prior to digestion procedure.
  - B. Samples will be either lyophilized or dried by microwave technique.
  - C. Dried samples will be digested by microwave procedures.
  - D. Exception - Mercury separate sample is used for this digestion. A modified Method 245.1 procedure is used.
2. Analytical procedures for metal analysis of fish tissue
  - A. The following metals will be analyzed using the designated EPA methods:

|           |                    |              |
|-----------|--------------------|--------------|
| Mercury   | Cold vapor, manual | Method 245.1 |
| Antimony  | AA, furnace        | Method 204.2 |
| Arsenic   | AA, furnace        | Method 206.2 |
| Barium    | ICP                | Method 200.7 |
| Beryllium | ICP                | Method 200.7 |
| Cadmium   | AA, furnace        | Method 213.2 |
| Chromium  | AA, furnace        | Method 218.2 |
| Lead      | AA, furnace        | Method 239.2 |
| Selenium  | AA, furnace        | Method 270.2 |
| Silver    | AA, furnace        | Method 272.2 |
3. Quality assurance procedure for analysis of metals in fish tissue
  - A. Samples will be analyzed in groups of six and will be as follows:
    - 4 fish tissue samples
    - 1 duplicate of one of the 4 above
    - 1 spike of the duplicate sample
  - B. There will be a reagent blank run after every 3 groups.
  - C. Standard operating procedure for AA furnace methods is to run a spike for each metal determination and show a recovery factor of  $\pm 20\%$  before the data is considered valid.

4. Reporting Limits

- A. The Division has not established MDL's on fish tissue.
- B. Reporting limits will be as low as the QA program allows.

5. Moisture determination procedure

- A. We propose to perform the moisture determination of the fish tissue using the CEM microwave moisture balance.
- B. The quality assurance for the moisture determination will be as follows:
  - 5 fish tissue samples
  - 1 duplicate of one of the 5 above
- C. Report the determination as calculated from the wet and dry weights.

**QUALITY CONTROL**

Units are ug/gram wet weight

| <u>Sample #</u>    | <u>Element</u> | <u>Duplicates</u> |                  | <u>Amount Spiked and % Recovered</u> |                   |
|--------------------|----------------|-------------------|------------------|--------------------------------------|-------------------|
|                    |                | <u>Sample</u>     | <u>Duplicate</u> | <u>Spike</u>                         | <u>% Recovery</u> |
| 0692266<br>Walleye | Lead           | 0.623             | 0.550            | 3.18                                 | 86.5              |
|                    | Cadmium        | <0.013            | <0.013           | 1.17                                 | 108               |
|                    | Chromium       | 0.187             | 0.200            | 2.88                                 | 99.5              |
|                    | Arsenic        | <0.5              | <0.5             | 2.45                                 | 98                |
|                    | Copper         | <0.5              | <0.5             | 11.7                                 | 94                |
|                    | Selenium       | 0.277             | 0.290            | 1.66                                 | 110               |
|                    | Antimony       | <1.25             | <1.25            | 6.27                                 | 100               |
|                    | Silver         | <0.05             | <0.05            | 1.04                                 | 104               |
|                    | Barium         | <0.25             | <0.25            | 24.5                                 | 97.4              |
|                    | Beryllium      | <0.025            | <0.025           | 2.47                                 | 98                |
|                    | Mercury        | 0.253             | 0.219            | 0.605                                | 77.3              |

| <u>Sample #</u>            | <u>Element</u> | <u>Duplicates</u> |                  | <u>Amount Spiked and % Recovered</u> |                   |
|----------------------------|----------------|-------------------|------------------|--------------------------------------|-------------------|
|                            |                | <u>Sample</u>     | <u>Duplicate</u> | <u>Spike</u>                         | <u>% Recovery</u> |
| 0692276<br>White<br>Sucker | Lead           | 0.162             | 0.225            | 2.19                                 | 80                |
|                            | Cadmium        | 0.017             | 0.013            | 1.1                                  | 110               |
|                            | Chromium       | 0.212             | 0.175            | 2.2                                  | 81.5              |
|                            | Arsenic        | <0.5              | <0.5             | 2.47                                 | 101               |
|                            | Copper         | <0.5              | <0.5             | 11.3                                 | 91                |
|                            | Selenium       | 0.427             | 0.544            | 1.56                                 | 67                |
|                            | Antimony       | <1.25             | <1.25            | 6.14                                 | 100               |
|                            | Silver         | <0.05             | <0.05            | 1.01                                 | 82.6              |
|                            | Barium         | 0.299             | 0.399            | 26.2                                 | 104               |
|                            | Beryllium      | <0.025            | <0.025           | 2.27                                 | 91                |
|                            | Mercury        | <0.1              | <0.1             | 0.489                                | 90                |

| <u>Sample #</u>           | <u>Element</u> | <u>Duplicates</u> |                  | <u>Amount Spiked and % Recovered</u> |                   |
|---------------------------|----------------|-------------------|------------------|--------------------------------------|-------------------|
|                           |                | <u>Sample</u>     | <u>Duplicate</u> | <u>Spike</u>                         | <u>% Recovery</u> |
| 0692282<br>White<br>Perch | Lead           | 1.25              | 1.36             | 3.27                                 | 80                |
|                           | Cadmium        | 0.038             | 0.031            | 1.17                                 | 112               |
|                           | Chromium       | 0.663             | 0.623            | 2.92                                 | 89.5              |
|                           | Arsenic        | <0.5              | <0.5             | 2.39                                 | 95                |
|                           | Copper         | 1.7               | 1.7              | 13.2                                 | 100               |
|                           | Selenium       | 0.630             | 0.698            | 1.90                                 | 101               |
|                           | Antimony       | <1.25             | <1.25            | 6.51                                 | 104               |
|                           | Silver         | <0.05             | <0.05            | .931                                 | 92.5              |
|                           | Barium         | 0.375             | <0.25            | 26.1                                 | 100               |
|                           | Beryllium      | <0.025            | <0.025           | 2.59                                 | 100               |
|                           | Mercury        | 0.1               | <0.1             | .439                                 | 72                |

### QUALITY CONTROL

Units are ug/gram wet weight

| <u>Sample #</u> | <u>Element</u> | <u>Duplicates</u> |                  | <u>Amount Spiked and % Recovered</u> |                   |
|-----------------|----------------|-------------------|------------------|--------------------------------------|-------------------|
|                 |                | <u>Sample</u>     | <u>Duplicate</u> | <u>Spike</u>                         | <u>% Recovery</u> |
| 0692283         | Lead           | 0.716             | 0.583            | 2.33                                 | 65                |
| Gizzard         | Cadmium        | <0.013            | 0.016            | 1.09                                 | 110               |
| Shad            | Chromium       | 0.272             | 0.261            | 2.99                                 | 110               |
|                 | Arsenic        | <0.5              | <0.5             | 2.2                                  | 89                |
|                 | Copper         | 1.11              | 0.744            | 12.69                                | 101               |
|                 | Selenium       | 0.568             | 0.551            | 1.84                                 | 106               |
|                 | Antimony       | <1.25             | <1.25            | 6.10                                 | 98.8              |
|                 | Silver         | <0.05             | <0.05            | 0.969                                | 98                |
|                 | Barium         | <0.25             | <0.25            | 23.9                                 | 95.2              |
|                 | Beryllium      | <0.025            | <0.025           | 2.36                                 | 95                |
|                 | Mercury        | <0.1              | <0.1             | 0.439                                | 76                |

| <u>Sample #</u> | <u>Element</u> | <u>Duplicates</u> |                  | <u>Amount Spiked and % Recovered</u> |                   |
|-----------------|----------------|-------------------|------------------|--------------------------------------|-------------------|
|                 |                | <u>Sample</u>     | <u>Duplicate</u> | <u>Spike</u>                         | <u>% Recovery</u> |
| 0692287         | Lead           | 1.2               | 1.75             | 3.18                                 | 78                |
| Walleye         | Cadmium        | 0.19              | 0.014            | 0.855                                | 83                |
|                 | Chromium       | 0.451             | 0.52             | 2.36                                 | 76                |
|                 | Arsenic        | <0.5              | <0.5             | 2.11                                 | 84                |
|                 | Copper         | 0.801             | 1.44             | 14.1                                 | 107               |
|                 | Selenium       | 0.636             | 0.582            | 1.72                                 | 86.4              |
|                 | Antimony       | <1.25             | <1.25            | 5.68                                 | 90.4              |
|                 | Silver         | <0.05             | <0.05            | 0.935                                | 93                |
|                 | Barium         | <0.25             | <0.25            | 27.1                                 | 109               |
|                 | Beryllium      | <0.025            | <0.025           | 2.61                                 | 105               |
|                 | Mercury        | <0.1              | <0.1             | 0.416                                | 78                |

| <u>Sample #</u> | <u>Element</u> | <u>Duplicates</u> |                  | <u>Amount Spiked and % Recovered</u> |                   |
|-----------------|----------------|-------------------|------------------|--------------------------------------|-------------------|
|                 |                | <u>Sample</u>     | <u>Duplicate</u> | <u>Spike</u>                         | <u>% Recovery</u> |
| 0692270         | Lead           | 0.663             | 1.19             | 2.82                                 | 89.5              |
| Rainbow         | Cadmium        | 0.013             | <0.013           | 0.963                                | 98.6              |
| Trout           | Chromium       | 0.253             | 0.531            | 2.60                                 | 97.5              |
|                 | Arsenic        | <0.5              | <0.5             | 2.27                                 | 94.0              |
|                 | Copper         | <0.5              | <0.5             | 12.1                                 | 100.2             |
|                 | Selenium       | 0.366             | 0.425            | 1.58                                 | 93.4              |
|                 | Antimony       | <1.25             | <1.25            | 6.27                                 | 104               |
|                 | Silver         | <0.05             | <0.05            | 0.934                                | 96.7              |
|                 | Barium         | <0.25             | <0.25            | 23.5                                 | 97.9              |
|                 | Beryllium      | not run           | not run          | not run                              | not run           |
|                 | Mercury        | <0.1              | <0.1             | 0.562                                | 80.0              |



QUALITY CONTROL

Units are ug/gram wet weight

| <u>Sample #</u> | <u>Element</u> | <u>Duplicates</u> |                  | <u>Amount Spiked and % Recovered</u> |                   |
|-----------------|----------------|-------------------|------------------|--------------------------------------|-------------------|
|                 |                | <u>Sample</u>     | <u>Duplicate</u> | <u>Spike</u>                         | <u>% Recovery</u> |
| 0692271         | Lead           | 0.843             | not run          | not run                              | not run           |
| Rainbow         | Cadmium        | 0.020             | not run          | not run                              | not run           |
| Trout           | Chromium       | .397              | not run          | not run                              | not run           |
|                 | Arsenic        | <0.5              | <0.5             | 2.29                                 | 92                |
|                 | Copper         | <0.5              | <0.5             | 11.71                                | 94.4              |
|                 | Selenium       | 0.489             | 0.449            | 1.75                                 | 101               |
|                 | Antimony       | <1.25             | <1.25            | 5.55                                 | 89.2              |
|                 | Silver         | <0.05             | <0.05            | 1.00                                 | 100               |
|                 | Barium         | <0.25             | <0.25            | 24.8                                 | 99.8              |
|                 | Beryllium      | <0.025            | <0.025           | 2.53                                 | 102               |
|                 | Mercury        | 0.178             | 0.179            | 0.679                                | 90                |



DER spike results

| sample    | spiked with           | % recovery |
|-----------|-----------------------|------------|
| ORG-6064  | 0.50 ppm Aroclor 1260 | 104        |
| ORG-5610  | 0.50 ppm Aroclor 1260 | 98         |
| ORG-5606  | 0.50 ppm Aroclor 1260 | 118        |
| ORG-5602  | 0.50 ppm Aroclor 1260 | 86         |
| ORG-5598  | 0.50 ppm Aroclor 1260 | 96         |
| ORG-3693  | 0.50 ppm Aroclor 1260 | 96         |
| ORG-4607  | 0.50 ppm Aroclor 1260 | 118        |
| ORG-3685  | 0.50 ppm Aroclor 1260 | 74         |
| ORG-5310  | 0.10 ppm chlordane    | 73         |
| ORG-3303  | 1.00 ppm Aroclor 1260 | 107        |
| ORG-3773  | 1.00 ppm Aroclor 1260 | 99         |
| ORG-3681  | 0.20 ppm chlordane    | 74         |
| ORG-3677  | 0.05 ppm dieldrin     | 74         |
| ORG-3773  | 0.05 ppm dieldrin     | 78         |
| ORG-3303  | 0.05 ppm dieldrin     | 70         |
| ORG-4603  | 0.1 ppm chlordane     | 71         |
| ORG-4607  | 0.5 ppm Aroclor 1260  | 118        |
| DER blank | 0.5 ppm Aroclor 1242  | 132        |
| DER blank | 0.5 ppm Aroclor 1248  | 134        |
| DER blank | 2.0 ppm toxaphene     | 100        |
| DER blank | 0.2 ppm chlordane     | 110        |
| DER blank | 0.05 ppm dieldrin     | 106        |

DER blank results

| sample                | comment |
|-----------------------|---------|
| DER blank             | N.D.    |
| solvent/reagent blank | N.D.    |

DER duplicate results

| sample   | parameters found | ppm               |
|----------|------------------|-------------------|
| ORG-3673 | DDE              | 0.034 0.033       |
|          | chlordane        | 0.14 0.15         |
|          | Aroclor 1260     | 0.19 0.18         |
|          | dieldrin         | trace trace       |
|          | % lipids         | 0.57 0.53         |
| ORG-3680 | DDE              | 0.10 0.094        |
|          | DDD              | 0.009 0.008       |
|          | chlordane        | 0.17 0.16         |
|          | Aroclor 1260     | 0.48 0.40         |
|          | dieldrin         | trace trace       |
| % lipids | 6.69 7.03        |                   |
| ORG-3683 | DDE              | 0.017 0.014       |
|          | chlordane        | 0.058 0.053       |
|          | Aroclor 1260     | 0.065 indications |
|          | a-BHC            | 0.019 0.017       |
|          | dieldrin         | trace trace       |
| % lipids | 0.74 0.66        |                   |
| ORG-3689 | DDE              | 0.011 0.012       |
|          | chlordane        | 0.024 0.025       |

|          |                   |             |             |
|----------|-------------------|-------------|-------------|
|          | Aroclor 1260      | ~0.12       | ~0.12       |
|          | % lipids          | 0.51        | 0.46        |
| ORG-4603 | DOE               | 0.013       | 0.019       |
|          | Aroclor 1260      | 0.560       | 0.620       |
|          | chlordane         | 0.045       | 0.069       |
| ORG-3693 | DOE               | ~0.011      | ~0.019      |
|          | Aroclor 1254      | ~0.09       | ~0.17       |
|          | chlordane         | ~0.021      | ~0.045      |
|          | % lipids          | 0.88        | 1.59        |
| ORG-3694 | Aroclor 1260      | indications | indications |
|          | chlordane         | indications | ~0.041      |
|          | % lipids          | 3.19        | 3.12        |
| ORG-5600 |                   | N.D.        | N.D.        |
|          | % lipids          | 0.16        | 0.18        |
| ORG-5604 |                   | N.D.        | N.D.        |
|          | % lipids          | 0.14        | 0.09        |
| ORG-5607 | Aroclor 1260      | 1.4         | 1.4         |
|          | chlordane         | 0.085       | 0.11        |
|          | dieldrin          | ~0.011      | ~0.010      |
|          | % lipids          | 1.15        | 1.06        |
| ORG-5612 | DOE               | 0.040       | 0.043       |
|          | Aroclor 1260      | 0.19        | 0.22        |
|          | chlordane         | 0.050       | 0.050       |
|          | dieldrin          | trace       | trace       |
|          | Aroclor 1242/1248 | indications | indications |
|          | % lipids          | 2.35        | 2.45        |
| ORG-6063 | DOE               | 0.072       | 0.072       |
|          | DDD               | 0.019       | 0.019       |
|          | chlordane         | 0.22        | 0.21        |
|          | Aroclor 1260      | 0.21        | 0.23        |
|          | dieldrin          | ~0.015      | ~0.014      |
|          | % lipids          | 5.39        | 5.20        |
| ORG-5308 | DOE               | 0.39        | 0.37        |
|          | DDD               | 0.11        | 0.10        |
|          | Aroclor 1260      | 1.2         | 1.2         |
|          | a-BHC             | ~0.012      | ~0.010      |
|          | dieldrin          | ~0.010      | 0.021       |
|          | % lipids          | 10.91       | 10.74       |

Sample No. 88-202

Site: Pennsylvania

Set: \_\_\_\_\_

Field ID: CZ#6A

Species: \_\_\_\_\_

%Fat: 13.5 (DUP.13)

Length (cm): \_\_\_\_\_

Weight (gm): \_\_\_\_\_

Sample Type: \_\_\_\_\_

| COMPOUND                    | RESULT<br>mg/Kg | REMARK<br>Duplicate | RDL<br>mg/Kg |
|-----------------------------|-----------------|---------------------|--------------|
| Hexachlorobenzene           | 0.012           | 0.012               | 0.001        |
| Mirex                       | <0.005          | <0.005              | 0.005        |
| <u>gamma</u> -BHC (Lindane) | <0.005          | <0.005              | 0.005        |
| Heptachlor                  | <0.005          | <0.005              | 0.005        |
| Aldrin                      | <0.005          | <0.005              | 0.005        |
| Octachlorostyrene           | 0.011           | 0.011               | 0.001        |
| Pentachlorostyrene          | 0.010 est.      | 0.010 est.          | 0.001        |
| Hexachlorostyrene           | 0.003 est.      | 0.003 est.          | 0.001        |
| Heptachlorostyrene          | 0.003 est.      | 0.003 est.          | 0.001        |
| Oxychlorane                 | 0.014           | 0.011               | 0.003        |
| Heptachlor Epoxide          | 0.016           | 0.014               | 0.003        |
| <u>gamma</u> -Chlordane     | 0.030           | 0.029               | 0.003        |
| <u>trans</u> -Nonachlor     | 0.153           | 0.149               | 0.003        |
| <u>alpha</u> -Chlordane     | 0.086           | 0.084               | 0.003        |
| 4,4'-DDE                    | 0.560           | 0.522               | 0.003        |
| Dieldrin                    | 0.074           | 0.073               | 0.005        |
| <u>cis</u> -Nonachlor       | 0.071           | 0.074               | 0.003        |
| 4,4'-DDD                    | 0.243           | 0.236               | 0.005        |
| 4,4'-DDT                    | 0.063           | 0.059               | 0.005        |
| FF-1 (FBB)                  | <0.005          | <0.005              | 0.005        |
| Aroclor 1242 (PCB)          |                 |                     | 0.025        |
| Aroclor 1254 (PCB)          | 2.30            | 2.24                | 0.025        |
| Aroclor 1260 (PCB)          |                 |                     | 0.025        |
| Total PCB                   | 2.30            | 2.24                | 0.025        |
| Toxaphene                   | <0.050          | <0.050              | 0.050        |
| Total Chlordane             | 0.340           | 0.336               | 0.020        |

MICHIGAN DEPARTMENT OF PUBLIC HEALTH large mouth bass 0692288 CEHS 179 (2)  
 CEHS-DIVISION OF LABORATORY SERVICES FISH MONITORING PROGRAM

Sample No. 88-201

Site: Pennsylvania

Set: \_\_\_\_\_

Field ID: CZM#18

Species: \_\_\_\_\_

%Fat: 0.65 (DUP.0.5)

Length (cm): \_\_\_\_\_

Weight (gm): \_\_\_\_\_

Sample Type: \_\_\_\_\_

| COMPOUND                    | RESULT<br>mg/Kg | REMARK<br>Duplicate | RDL<br>mg/Kg |
|-----------------------------|-----------------|---------------------|--------------|
| Hexachlorobenzene           | <0.001          | <0.001              | 0.001        |
| Mirex                       | <0.005          | <0.005              | 0.005        |
| <u>gamma</u> -BHC (Lindane) | <0.005          | <0.005              | 0.005        |
| Heptachlor                  | <0.005          | <0.005              | 0.005        |
| Aldrin                      | <0.005          | <0.005              | 0.005        |
| Octachlorostyrene           | <0.001          | <0.001              | 0.001        |
| Pentachlorostyrene          | <0.001          | <0.001              | 0.001        |
| Hexachlorostyrene           | <0.001          | <0.001              | 0.001        |
| Heptachlorostyrene          | <0.001          | <0.001              | 0.001        |
| Oxychlordanes               | <0.003          | <0.003              | 0.003        |
| Heptachlor Epoxide          | <0.003          | <0.003              | 0.003        |
| <u>gamma</u> -Chlordane     | <0.003          | <0.003              | 0.003        |
| <u>trans</u> -Nonachlor     | <0.003          | <0.003              | 0.003        |
| <u>alpha</u> -Chlordane     | <0.003          | <0.003              | 0.003        |
| 4,4'-DDE                    | 0.010           | 0.009               | 0.003        |
| Dieldrin                    | <0.005          | <0.005              | 0.005        |
| <u>cis</u> -Nonachlor       | <0.003          | <0.003              | 0.003        |
| 4,4'-DDD                    | <0.005          | <0.005              | 0.005        |
| 4,4'-DDT                    | <0.005          | <0.005              | 0.005        |
| FF-1 (PBB)                  | <0.005          | <0.005              | 0.005        |
| Aroclor 1242 (PCB)          |                 |                     | 0.025        |
| Aroclor 1254 (PCB)          | 0.075           | 0.079               | 0.025        |
| Aroclor 1260 (PCB)          |                 |                     | 0.025        |
| Total PCB                   | 0.075           | 0.079               | 0.025        |
| Toxaphene                   | <0.050          | <0.050              | 0.050        |
| Total Chlordane             | Not detected    | Not detected        | 0.020        |

Sample No. 88-200 Site: Pennsylvania Set: \_\_\_\_\_  
 Field ID: CZY#32 Species: \_\_\_\_\_ %Fat: 15.3 (DUP 15.  
 Length (cm): \_\_\_\_\_ Weight (gm): \_\_\_\_\_ Sample Type: \_\_\_\_\_

| COMPOUND                    | RESULT<br>mg/Kg | REMARK<br>Duplicate | RDL<br>mg/Kg |
|-----------------------------|-----------------|---------------------|--------------|
| Hexachlorobenzene           | 0.008           | 0.007               | 0.001        |
| Mirex                       | <0.005          | <0.005              | 0.005        |
| <u>gamma</u> -BHC (Lindane) | <0.005          | <0.005              | 0.005        |
| Heptachlor                  | <0.005          | <0.005              | 0.005        |
| Aldrin                      | <0.005          | <0.005              | 0.005        |
| Octachlorostyrene           | 0.007           | 0.007               | 0.001        |
| Pentachlorostyrene          | 0.005 est.      | 0.005 est.          | 0.001        |
| Hexachlorostyrene           | 0.002 est.      | 0.002 est.          | 0.001        |
| Heptachlorostyrene          | 0.002 est.      | 0.002 est.          | 0.001        |
| Oxychlordane                | 0.008           | 0.006               | 0.003        |
| Heptachlor Epoxide          | 0.014           | 0.012               | 0.003        |
| <u>gamma</u> -Chlordane     | 0.028           | 0.028               | 0.003        |
| <u>trans</u> -Nonachlor     | 0.070           | 0.069               | 0.003        |
| <u>alpha</u> -Chlordane     | 0.066           | 0.065               | 0.003        |
| 4,4'-DDE                    | 0.236           | 0.230               | 0.003        |
| Dieldrin                    | 0.058           | 0.059               | 0.005        |
| <u>cis</u> -Nonachlor       | 0.032           | 0.035               | 0.003        |
| 4,4'-DDD                    | 0.120           | 0.118               | 0.005        |
| 4,4'-DDT                    | 0.079           | 0.072               | 0.005        |
| FF-1 (PBB)                  | <0.005          | <0.005              | 0.005        |
| Aroclor 1242 (PCB)          |                 |                     | 0.025        |
| Aroclor 1254 (PCB)          | 1.40            | 1.43                | 0.025        |
| Aroclor 1260 (PCB)          |                 |                     | 0.025        |
| Total PCB                   | 1.40            | 1.43                | 0.025        |
| Toxaphene                   | <0.050          |                     | 0.050        |
| Total Chlordane             | 0.196           | 0.197               | 0.020        |

MICHIGAN DEPARTMENT OF PUBLIC HEALTH  
 CEHS-DIVISION OF LABORATORY SERVICES - FISH MONITORING PROGRAM  
 EPA FAT CONTROL #137

88 ADV. SET \_\_\_\_\_ CONTROL NO. 1

| COMPOUND                | %RECOVERY | MDPH VALUE mg/Kg | TRUE VALUE mg/Kg |
|-------------------------|-----------|------------------|------------------|
| Hexachlorobenzene       | 102       | 0.050            | 0.049            |
| <u>beta</u> -BHC        | 89        | 0.268            | 0.300            |
| Oxychlorane             | 101       | 0.113            | 0.112            |
| Heptachlor Epoxide      | 103       | 0.077            | 0.075            |
| <u>trans</u> -Nonachlor | 100       | 0.119            | 0.119            |
| 4,4'-DDE                | 96        | 2.120            | 2.200            |
| Dieldrin                | 103       | 0.041            | 0.040            |
| 4,4'-DDT                | 103       | 0.180            | 0.175            |
| Aroclor 1254            | 100       | 1.00             | 1.00             |
| Mirex                   | 99        | 0.128            | 0.129            |

MICHIGAN DEPARTMENT OF PUBLIC HEALTH  
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 FISH LIPID CONTROL #4

CONTROL NO. 2

| COMPOUND                | %RECOVERY | MDPH VALUE mg/Kg | TRUE VALUE mg/Kg |
|-------------------------|-----------|------------------|------------------|
| Dieldrin                | 100       | 0.656            | 0.658            |
| Hexachlorobenzene       | 105       | 0.043            | 0.041            |
| <u>cis</u> -Nonachlor   | 94        | 0.955            | 1.02             |
| Oxychlorane             | 99        | 0.820            | 0.830            |
| Heptachlor Epoxide      | 98        | 0.691            | 0.705            |
| <u>trans</u> -Nonachlor | 97        | 1.01             | 1.04             |
| p,p'DDE                 | 104       | 7.34             | 7.09             |
| p,p'DDD                 | 89        | 1.18             | 1.32             |
| p,p'DDT                 | 96        | 1.47             | 1.53             |
| Aroclor 1254            | 99        | 14.74            | 14.89            |
| <u>alpha</u> -Chlordane | 97        | 0.714            | 0.736            |
| <u>gamma</u> -Chlordane | 97        | 0.732            | 0.755            |



**A P P E N D I X   K**

**Memo - Discussion on Testing Procedures**

The following are excerpts from a memo to Richard Shertzer, Chief Quality Assessment Unit, Bureau of Water Quality Management, Pennsylvania DER, from Alan Bruzel, Organic Chemistry Section, Bureau of Laboratories, Pennsylvania DER, dated July 7, 1989

The discussion below refers to conversations/correspondence with the following concerning pesticide residue analysis in fish tissue:

John Austin, US EPA, Annapolis, MD  
Dan Donnelly, US EPA, Annapolis, MD  
Charles Finsterwalder, US FDA, Cincinnati, OH  
Jim Longbottom, US EPA, Cincinnati, OH  
Bob Welch, Michigan Dept. of Public Health, Lansing, MI

Mr. Longbottom is coordinating a study for an upcoming Chlordane Conference in Missouri that may ultimately yield a uniform fish extraction/analysis procedure. The results of this study should lead to a situation where there is one fish analysis procedure whose results are comparable and laboratory independent. (Currently, there are a variety of techniques which can differ in extraction, chromatography, and interpretation for fish flesh analysis). Participating labs in this study will use capillary column chromatography (probably DB-5) with a temperature gradient; Bureau of Labs currently uses packed column isothermal chromatography for fish tissue analysis. The extraction procedure will probably involve Soxhlet extraction followed by a cleanup step. Mr. Longbottom expects the results to be published in a few months time.

In terms of enforcement action, Mr. Finsterwalder notes that the results of both original and check analyses must exceed the tolerance limit for the analyte in question before it is necessary to take action. He indicated to me that the results of original and check analyses may either be from one laboratory or from different laboratories. The action guidelines require that both the original and check analyses are to be in "reasonably close agreement" which has been interpreted by Mr. Finsterwalder to be a judgement decision made on a case-by-case basis.

Precision between analyses has also been addressed by Mr. Austin who stated that the maximum difference between duplicate chlordane in water analyses is to be no more than eighteen percent per US EPA Method 608. We have calculated that historically the results from the Bureau of Labs for chlordane analysis (in EPA Water Pollution and Water Supply studies) have only varied between 1% more than actual EPA values and 12% less than actual EPA values - a difference within the 18% limit.

Mr. Welch sent the Bureau of Labs his original preparation of fish tissue extracts. After check pesticide/PCB analysis by the Bureau of Labs, aliquots of Mr. Welch's extracts were sent for further check analysis to Mr. Donnelly's EPA Annapolis, MD laboratory. The following is a brief description of the results obtained:

Fish extracts supplied by the State of Michigan were analyzed by Michigan, PA DER Bureau of Labs (BurLabs) and EPA, Annapolis laboratories for alpha- and gamma-chlordane (a- and g-chlordane). Each lab used the same extracts for analysis thereby eliminating differences in quantitation of analytes due to extraction technique. There were differences, however, in the gas chromatographic conditions that were used by the different labs. For ease of presentation, the data from packed columns is presented below.

Analytical results of two fish extracts prepared by the State of Michigan and analyzed by Michigan, BurLabs, and USEPA:

below are in ppm

| analyte     | Sample 88-0202 |          |        | Sample 88-0202 Dup |          |        |
|-------------|----------------|----------|--------|--------------------|----------|--------|
|             | Michigan       | Bur Labs | US EPA | Michigan           | Bur Labs | US EPA |
| a-chlordane | .086           | .27      | .089   | .084               | .20      | .085   |
| g-chlordane | .03            | .07      | .061   | .029               | .07      | .064   |

The following table details the gas chromatographic analytical columns used in analysis of the fish extracts. These are all packed columns. It should be stressed that each participating lab used different columns in the analysis of chlordane. We have at hand a memorandum from Mr. Austin through Mr. Donnelly that shows that responses to both a- and g-chlordane differ depending upon what chromatographic columns are used to analyze the compounds. DB-5 and DB-608 capillary columns used by EPA showed a higher response to a-chlordane; g-chlordane's responses did not vary as much.

Packed gas chromatographic columns used by labs in chlordane analysis of fish tissue

| BurLabs                    | Michigan                | US EPA |
|----------------------------|-------------------------|--------|
| 1.5% SP-2250/1.95% SP-2401 | 3% SE-30                | OV-1   |
| 4% SE-30/6% SP-2401        | 1.5% DV-17/1.95% DV-210 |        |
|                            | 4% SE-30/6% DV-210      |        |

From the above it can be concluded that in comparison to US EPA's results:

- 1.) BurLabs gives higher quantitation for both a- and g-chlordane:
  - 2.7 times higher for a-chlordane
  - 1.1 times higher for g-chlordane
- 2.) Michigan gives lower quantitation for g-chlordane:
  - 1.0 times lower for a-chlordane, i.e. results not different from US EPA
  - 2.1 times lower for g-chlordane

It would thus appear from the data that, compared to US EPA's results, BurLabs overestimates a-chlordane (and, to a lesser extent, g-chlordane) and Michigan underestimates g-chlordane.

Because of the apparent discrepancies of a- and g-chlordane results between all labs, the Bureau of Labs compared its a- and g-chlordane standards used in the above quantitations with other available a- and g-chlordane standards. This was done to eliminate the possibility that the Bureau of Labs incorrectly prepared its a- or g-chlordane standards. It was found that the gas chromatographic responses of a- and g-chlordane standards used in the analysis of the Michigan extracts were comparable to responses from other available a- and g-chlordane standards (US EPA and Accu-Standard, New Haven, CT). Incorrect preparation of chlordane standards by the Bureau of Labs can be ruled out as the origin of the discrepancies.

The differences in the results of this shared fish extract may be resolvable once the interlaboratory comparisons performed as part of the upcoming Missouri Chlordane Conference report are published. That report may answer the question regarding acceptable precision between fish tissue analyses. Different chromatography conditions may be responsible for the higher a-chlordane levels reported by the Bureau of Laboratories and the lower g-chlordane levels reported by the State of Michigan (in comparison with the results from US EPA). It is conceivable that a co-eluting metabolite or chlordane constituent is responsible for the enhanced a-chlordane value reported by the Bureau of Labs. The Bureau of Labs will refrain from quantitating a- and g-chlordane using packed columns and will quantitate technical chlordane only using packed columns.

We will keep current with the progress of Mr. Longbottom's study so that we can be prepared for whatever methodology he proposes for pesticide residue analysis in fish tissue. He anticipates the analytical method will involve Soxhlet extraction, a cleanup step and capillary chromatography with quantitation of each chlordane constituent reported. We will try to get details from this as yet unwritten Missouri Chlordane Conference method as his study progresses.

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