

# Residues of Mirex in Channel Catfish and Other Aquatic Organisms

by  
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Mirex bait has been used by USDA and State agencies in 9 southern states to control the imported fire ant, Solenopsis invicta Buren (formerly referred to as Solenopsis saevissima richteri Forel) since 1963 (LOFGREN et al, 1964; STRINGER et al, 1964). This bait presently is applied at 1.25 lbs/acre to an estimated 15 million acres of land yearly (USDA, 1970). Within the last few years residues of mirex have been found in various nontarget organisms (BUTLER, 1964; MCKENZIE, 1970; MARKIN et al, 1969). As a result of these findings, the safety of the use of this insecticidal bait has been questioned in court by several conservation groups (ROGERS and BROWN, 1970). Various articles condemning the use of this pesticide also have been published recently (ANONYMOUS, 1970; COON and FLEET, 1970). One of the major criticisms of the use of this pesticide has been that too little is known of the ecological impact of widespread bait applications, specifically the accumulation of insecticide in the food chain.

Commercial production of channel catfish (Ictalurus punctatus) for human consumption presently is a multimillion dollar a year industry in many of the states presently infested with the imported fire ant.

Treatment of infested land near or adjacent to commercial catfish ponds means that the possibility of contamination of a human food source exists. Local newspaper articles have been published charging that mirex residues in catfish might render the fish unsuitable for human consumption or interstate shipment. As yet no tolerance level for mirex in catfish has been determined by FDA, but the level in fat of farm animals is .1 ppm and .01 ppm for raw agricultural products (USDA, 1969).

## METHODS AND MATERIALS

A 3-acre fish pond with an average depth of 3.5', located near Gulfport, Mississippi, was chosen as a study site. The pond had been used previously for commercial production of minnows for fish bait. Thus, prior to the initiation of the present study,

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minnows, Opsopoeodus emiliae (Hay), and the mosquito fish (Gambusia affinis), were the only fish in the pond.

The pond and surrounding drainage area (120 acres) were treated with mirex bait by a Piper Pawnee aircraft on October 23, 1970. The plane was equipped with a Swathmaster<sup>®</sup> granular spreader calibrated to deliver the presently recommended dosage of 1.25 lbs. standard 4X mirex bait/acre (1.7 grams toxicant/acre). Guidance for the aircraft and swath spacing were provided by the helium-filled Kytoons tended by ground personnel.

On October 17, 1970 (5 days prior to treatment), 1,400 channel catfish (8-12" in length) and approximately 400 fingerlings (1-3" in length) were released into the pond. A wire cage 3' x 3' x 6' constructed with 1/2" mesh galvanized wire tacked to a wooden frame was used to confine 50 of the larger fish. The cages were constructed on wooden legs 18" long which were driven about 12" into the bottom of the cage from the pond bottom. Caged fish periodically were fed commercial catfish food. Uncaged fish were not fed, thereby forcing them to feed on an abundance of natural food such as minnows, crayfish, insect larvae, etc., which inhabited the pond.

Mortality of fish in the cages was assessed, and the accumulation of mirex residues in whole body samples of both caged and uncaged catfish was determined by GLC analysis. Each sample analyzed represented a composite of 3 individual fish.

The accumulation of mirex residues in 6 other aquatic organisms also was determined. The 6 indicator species chosen for this phase of our study included minnows (Opsopoeodus emiliae), mosquito fish (Gambusia affinis), cricket frogs (Acris gryllus), tadpoles (undetermined species), crayfish (Camarellus schufeldtii), and dragonfly nymphs (5 or more undetermined species). Each of these samples analyzed consisted of a composite of 6 or more individuals.

#### ANALYTICAL PROCEDURE

Composited samples of blended whole body tissues were processed by grinding a 5 gm portion with  $\text{Na}_2\text{SO}_4$  in a mortar and pestle and extracting by 1 hr rotation in 200 ml of hexane on a concentric rotor. Cleanup was accomplished by elution through columns packed with 10 gm of silica gel.

All samples were concentrated under a fume hood by heating on electric hot plates and distillation through Snyder columns to approximately 15 ml and transferred to conical test tubes. Further concentration, if needed, was accomplished by heating in a water bath with an air stream flowing through a Drierite filter into the conical test tubes.

After concentration to desired volume, the samples were analyzed on a Unilab 400 model Glowall gas chromatograph equipped

with an electron capture detector. The 4' x 3.4 mm spiral glass column was packed with 4% DC-200 on the supelcoport 100-120 mesh, conditioned isothermally at 190 C with the detector at 190 C and the injection port at 200 C. Purified grade nitrogen served as the carrier gas; the retention time for mirex was 10 minutes. Residues as low as 0.01 ppm were detectable by this procedure.

## RESULTS AND DISCUSSION

Both caged fingerlings and adults were maintained in the previously described cages for 6 months after treatment of the pond, at which time the wire bottom of the cage deteriorated and allowed the fish to escape. No mortality, deformation, or other ill effects of caged fish were observed during this time. Two dead catfish were observed floating on the pond surface 5 days after treatment. Analysis of these fish indicated that no detectable levels of mirex were present. We therefore assumed that the death of these fish was not related to the mirex treatment.

As shown in Table 1, buildup of mirex residues in uncaged fish did occur whereas the larger caged fish acquired no detectable mirex, and only minute levels of mirex were detected in caged fingerlings. The most probable explanation of this is that the uncaged fish did not have a ready access to the natural food chain, as did the caged fish, thus detectable residues of mirex did not accumulate in the larger caged fish. Had the uncaged fish obtained their residues by feeding directly on the mirex bait particles, rather than through the food chain, residues would have been quite high soon after treatment and decreased with time. However, as shown in Table 1, residues in these fish at 5 days after treatment were not detected, but began to increase after that, reaching a maximum of 0.65 ppm 6 months after treatment. 16 months after treatment (the last sampling date) uncaged catfish contained 0.44 ppm mirex.

A second reason for assuming that catfish do not feed directly on the bait itself is the fact that the caged fish did not acquire residues soon after treatment (Table 1). Bait particles were readily available to these fish due to the open top construction of the cage and size of the wire mesh used. Had the fish fed on the bait as it fell into the cage during treatment or floated in later, detectable levels of mirex should have been evident after GLC analysis of those fish.

The hypothesis that the uncaged catfish acquired their mirex residues through the food chain is supported by the data in Table 2. All aquatic indicators analyzed contained detectable levels of mirex. These residues decreased substantially in almost every indicator one year after treatment, but were still detectable at 16 months.

We feel that the conditions imposed upon the caged catfish probably more closely resemble conditions encountered by commercially grown fish than do conditions encountered by the uncaged fish.

TABLE 1

Mirex residues (ppm) in channel catfish following an aerial application of mirex bait (1.25 lbs/acre) to a fish pond and the surrounding drainage area.

Type Fish	Pre-Treatment 1/	Interval after Treatment					
		1 day	5 days	11 days	30 days	6 mos	12 mos
Caged Fingerlings	ND	ND	ND	-	0.01	0.03	-
Caged Adults (8-12")	ND	ND	ND	-	ND	ND	-
Uncaged Fish	ND	-	ND	0.20	0.32	0.65	0.31
							0.44

1/ ND means not detected at level of sensitivity .01 ppm.

TABLE 2

Mirex residues (ppm) in various aquatic organisms following an aerial application of mirex bait (1.25 lbs/acre) to a fish pond and the surrounding drainage area.

Organism	Pre-Treatment 1/	Interval after Treatment					
		5 days	38 days	6 months	12 months	16 months	
Crayfish	ND	0.40	0.14	0.08	0.01	0.02	
Mosquito Fish	ND	-	0.08	0.07	0.02	0.07	
Cricket Frogs	ND	0.03	2.26	2.88	0.02	0.05	
Minnows	ND	0.02	0.11	0.09	0.07	0.07	
Dragonfly nymphs	ND	0.70	ND	ND	ND	0.03	
Tadpoles	-	0.09	0.03	0.01	-	-	

1/ ND means not detected at lowest level of sensitivity, .01 ppm.

This belief is based on several facts:

In order to facilitate seining, commercial fish are grown in ponds free of all obstructions such as stumps, trees, and aquatic vegetation. Commercial ponds also are drained down each season to facilitate seining. These operations must reduce tremendously the supply of natural food in a commercial pond, whereas our study pond was very "old" and had a very abundant supply of natural food. Thus commercial fish are almost totally dependent upon artificial food placed in the pond by the grower, whereas the uncaged fish in our study pond were totally dependent upon natural food which was enhanced by abundant aquatic vegetation, dead trees, and thousands of minnows left in the pond from a former fish bait producing operation.

Thus, in short, we feel that if a true commercial catfish pond had been available for use in this experiment, the residue levels acquired by uncaged catfish would not have gone as high as they did under the conditions described herein. Routine monitoring by our laboratory has shown mirex residues ranging from 0.008 to 2.59 ppm in wild catfish from areas which received a blanket application of mirex bait, whereas commercially grown fish from ponds contained no detectable mirex residues.

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