

# Equilibrium Between Uptake and Elimination of Dieldrin by Channel Catfish, *Ictalurus punctatus*<sup>1</sup>

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## Abstract

Channel catfish, *Ictalurus punctatus*, measuring 150 (+5) mm were exposed continuously to dieldrin for 70 days to determine if equilibrium between dieldrin uptake and elimination was reached by muscle tissue. Fish exposed to 13 parts per trillion (pptr) dieldrin reached equilibrium in 56 days, fish exposed to 27 pptr dieldrin were near equilibrium after 56 days, and fish exposed to 49 pptr dieldrin did not reach a balance between uptake and elimination after 70 days of exposure.

Evidence of this study showed that balance between uptake and elimination was reached in less time at lower exposure levels than at higher levels.

## INTRODUCTION

In Iowa, farmers have used large amounts of aldrin to control rootworm and other corn insects for a number of years. Between 1961 and 1965, aldrin was applied to Iowa soils at annual rates of 5.0 to 6.5 million pounds; during 1968 through 1973, an estimated 2.0 million pounds of aldrin per year were used on Iowa soils (personal communication, Harold J. Stockdale, Extension Entomologist, Department of Entomology, Iowa State University, Ames, Iowa, 1973).

Evidently, this pesticide in the form of dieldrin, is finding its way into surface waters of the state. Dieldrin concentrations up to 1600 ppb (mg/kg) have been found in the edible portion of channel catfish (*Ictalurus punctatus*) (MORRIS and JOHNSON, 1971), exceeding by more than 5 times the permissible concentration of dieldrin in food allowed by the Food and Drug Administration. Catfish tested from many rivers consistently contained greater concentrations of dieldrin than did small catfish from Iowa streams.

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A field study by BULKLEY et al. (1974) indicated that dieldrin levels in channel catfish muscle were seasonal. According to their study, dieldrin levels in catfish increased within a few weeks after the peak of the corn planting season. Evidently, agricultural activity played a role in determining the level of dieldrin in catfish muscle.

MACEK et al. (1970) reported that DDT and dieldrin levels were dose-dependent and that fish exposed to 1 ppm per week of dieldrin reached equilibrium after 140 days. Their study further established that some organs reached dieldrin equilibrium before other organs, and in each instance equilibrium was dose-dependent. Skeletal muscles reached equilibrium in 28 days of exposure to 1 ppm in food in their study with rainbow trout.

The channel catfish was used in this study because of its importance as one of Iowa's most sought-after sports fish. This study attempted to determine, under laboratory conditions, if equilibrium between uptake and elimination of dieldrin was dependent on the level of exposure.

#### MATERIALS AND METHODS

Channel catfish measuring 150 ( $\pm$ 5) were obtained from a commercial fish culturist. Fish were maintained in aquaria containing 45 liters of water and fed a diet of commercially pelleted food obtained from Astra Pharmaceutical Products, Inc., Worcester, Mass. The contents and analyses of the diet are reported in SHANNON (1974).

Water from the Iowa State University well was maintained at 21 $\pm$ 1 C and was dechlorinated before entering the tanks (SHANNON 1974).

Chemicals used for extractions contained less than 10 parts per trillion (ppt) total pesticides and were considered suitable for pesticide determinations. All apparatus was washed and pre-rinsed with petroleum ether or hexane before use. The technical dieldrin used was obtained from Shell Chemical Company and contained not less than 87% of 1,2,3,4,10,10 hexachloro-6, 7-epoxy-1, and not more than 13% of insecticidally active related compounds (SHELL CHEMICAL CORPORATION, 1959).

Dieldrin was dissolved in acetone before mixing with water. Triton X-100, an organic solvent, was used to reduce loss of dieldrin from solution (SEBA, 1970). The acetone and Triton X-100 levels (25 ppm and 1 ppm, respectively) were kept well below the lethal concentrations for catfish. A 50% proportional diluter, which delivered 1 liter at 5 concentrations, was used to meter the various dieldrin concentrations into individual tanks (MOUNT and BRUNGS, 1967). A complete change of water occurred every 6 hr. Uncontaminated water, which triggered the diluter system, flowed through tank 1 containing control fish. Fish in tank 2 were exposed to 49 ppt of dieldrin; tank 3 contained fish exposed to

27 ppb of dieldrin, and in tank 4 the fish were exposed to 13 ppb of dieldrin. The fish in tanks 5 and 6, containing 8.5 and 4.0 ppb of dieldrin, respectively, died of unknown causes before the experiment was completed. Three samples of two fish each were taken from each tank every 2 weeks for 70 days. Dilution ratios for the water dieldrin solutions were based on gas chromatographic analyses of weekly samples.

Samples of water from the test chambers were analyzed according to methods described by the U.S. ENVIRONMENTAL PROTECTION AGENCY (1971). Water samples of 1 liter were extracted by mixing with 120 ml of 15% diethyl ether in hexane in a 3-liter separatory funnel. The mixture was shaken vigorously for 1 min and allowed to separate into layers. The aqueous layer was extracted a second time with 100 ml of hexane. The two hexane layers were combined and filtered through a 50-mm column of anhydrous sodium sulfate. The filtrate was evaporated to 1 ml before a sample was injected into the gas chromatograph.

Catfish dorsal muscle samples were analyzed according to the guidelines of the U.S. DEPARTMENT OF HEALTH, EDUCATION and WELFARE (1970). For analysis, 10 g of dorsal muscle tissue were ground with 350 ml of 35% distilled water-acetonitrile solution in a Waring blender for 10 min. The samples were filtered through fluted No. 40 Whatman filter paper. The filtrate (260 ml) was transferred to a 1-liter separatory funnel to which was added 100 ml of petroleum ether and shaken vigorously for 2 min. Distilled water (600 ml) and a saturated saline solution (10 ml) were added to the samples and mixed thoroughly by vigorous tumbling action for 15 sec. The layers were allowed to separate, and the aqueous layer was discarded. The solvent layers were gently washed with two 100-ml portions of water. The washings were discarded, and the solvent layers each were filtered through a 50-mm column of anhydrous sodium sulfate into a 100-ml graduated cylinder. The volume of each was recorded.

The samples were filtered through a 115-mm column of Florisil, which was topped by a 25-mm column of anhydrous sodium sulfate. Dieldrin was eluted from the column with 200 ml of 15% diethyl ether in petroleum ether following 200 ml of a 6% mixture of diethyl ether and petroleum ether. The two mixtures were collected in separate flasks and evaporated to 10 ml. The samples were then ready for injection into the chromatograph.

A Beckman GC-5 gas chromatograph was used to identify and quantify the dieldrin levels. An electron capture detector was used with a helium flow rate of 80 ml/min, a temperature of 180°C, and an attenuation of  $2 \times 10^3$  on the 5% OV-210 column. A 4% SE-30/QF-1 column was used as a qualitative check with a gas flow rate of 120 ml/min, a temperature of 200°C and an attenuation of  $2 \times 10^3$ .

Periodically throughout the study, known levels of aldrin were added to samples of fish muscle and water before chemical extraction was started. The portion of aldrin recovered in the extraction process ranged from 83 to 90%, with an average of 85%. Dieldrin extraction efficiency was assumed similar, even though the aldrin added was not incorporated within the muscle cells as was dieldrin.

## RESULTS AND DISCUSSION

Fish exposed to 49 pptr of dieldrin for the 70day test period continued to accumulate dieldrin linearly throughout the exposure period (Table 1), Figure 1). After 70 days, dieldrin concentration in this test group was significantly greater ( $p=0.01$ ) than the dieldrin level observed after 56 days of exposure. Catfish exposed to 27 pptr of dieldrin accumulated a mean dieldrin total of 39 ppb after 56 days and 48 ppb after 70 days of exposure. The trend in accumulation was still upward, although the difference between levels at 56 and 70 days was not statistically significant. Fish exposed to 13 pptr of dieldrin contained a mean concentration of 29 ppb after 56 days and 31 ppb at the end of the 70-day test period. These levels also were not significantly different. Because this study dealt only with equilibrium between uptake and elimination of dieldrin, other factors, such as growth, were not noted.

TABLE 1.

Mean levels of dieldrin in dorsal muscle of 150 mm channel catfish exposed to dieldrin for 70 days. Concentration in ppb (standard deviation in parentheses)<sup>a</sup>.

Exposure level	Time (days) from beginning of exposure					
	0	14	28	42	56	70
0	7(1)	4(1)	4(1)	4(2)	4(2)	4(1)
49 pptr	7(1)	45(11)	57(5)	84(10)	130(14)	164(11)
27 pptr	7(1)	20(4)	29(1)	38(4)	39(10)	48(6)
13 pptr	7(1)	9(2)	13(3)	19(7)	29(3)	31(3)

<sup>a</sup> All values based on 3 samples of 2 fish each.

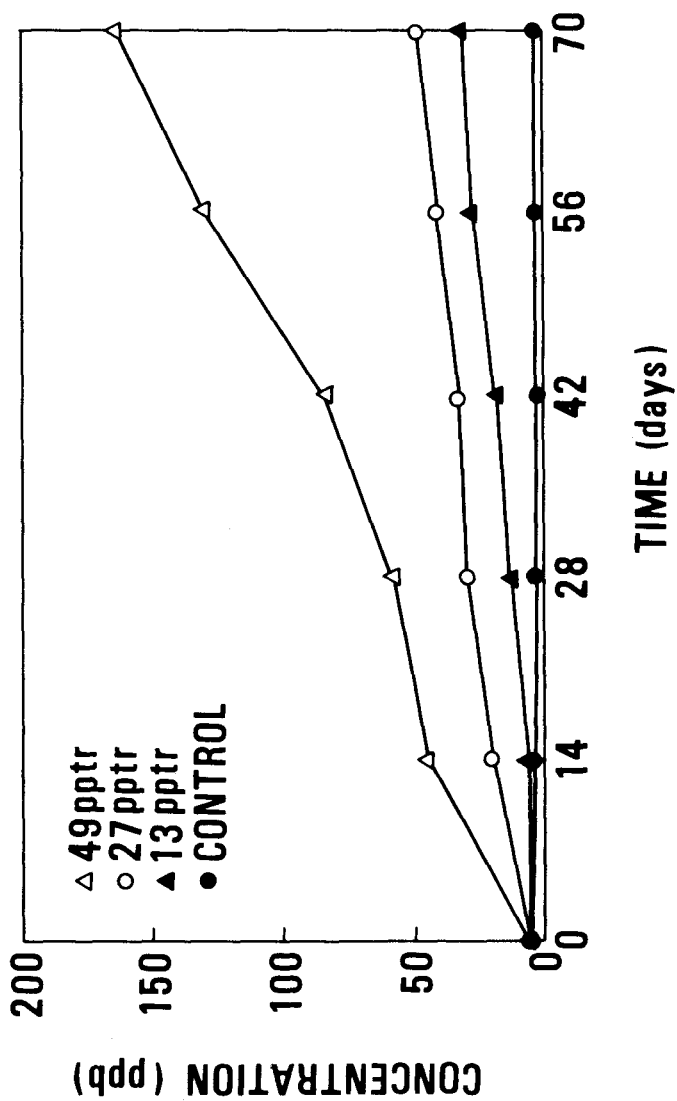


Figure 1. Uptake of diel-drin by dorsal muscle of 150-mm channel catfish during 70-day exposure in water.

Experimental evidence indicates that exposure to a cyclo-diene insecticide (such as dieldrin) results in an upper limit of accumulation in biological systems, and this level is dependent on the level of exposure (BROOKS, 1971; MACEK et al., 1970). My study upheld this idea. Fish exposed to 13 pptr of dieldrin reached a balance between uptake and elimination in dorsal muscle in 56 days of exposure. Catfish exposed to 27 pptr were very near the state of balance between dieldrin uptake and elimination in muscle tissue by 56 days. Catfish exposed to 49 pptr of dieldrin still were accumulating dieldrin linearly at the end of the 70-day test period; hence, equilibrium was not reached during the test period at this exposure level.

Under natural conditions, it also seems that the level of dieldrin found in catfish muscle is dependent upon the level of dieldrin in their environment. Dieldrin levels at a given time in the environment could determine whether fish will continue dieldrin uptake linearly from the water, eliminate dieldrin at a more rapid rate than dieldrin is taken up, or if equilibrium between uptake and equilibrium is established. Seasonal fluctuations in dieldrin concentrations in water and catfish muscle were noted by BULKLEY et al. (1974). Dieldrin concentrations in Des Moines (Iowa) River water increased a few weeks after aldrin was applied to the soil during planting season. The dieldrin level decreased in the water a few weeks later. The same trend was evident in dieldrin concentrations found in catfish muscle. Hence, fluctuations in exposure levels is one factor determining equilibrium between dieldrin uptake and elimination. Evidence of this study showed that balance was reached in less time at lower exposure levels than at higher levels.

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## REFERENCES

- BROOKS, G. T.: p. 111-136. In: A. S. Tahoni (ed), Pesticide Terminal Residues. Tel-Aviv, Israel. 1971.
- BULKLEY, R. V., L. R. SHANNON, and R. L. KELLOGG: Contamination of channel catfish with dieldrin from agriculture runoff. Iowa State Water Resources Research Institute Completion Report 62. Iowa State Univ. Ames. 144pp. 1974.
- MACEK, K. J. et al.: Trans. Am. Fish. Soc. 99:689-695. (1970).
- MORRIS, R. L. and L. G. JOHNSON: Pesticide levels in fish from Iowa streams. Iowa State Hygienic Lab. Rept. No. 71-23. 6p. 1971.
- MOUNT, D. I., and W. A. BRUNGS: Water Res. 1, 21 (1967).
- SEBA, D. B.: Ph.D. thesis. Univ. of Miami (Libr. Congr. Card No. Mic.-70-18167). 104pp. Univ. Microfilms, Ann Arbor, Mich. 1970.
- SHANNON, L. R.: Ph.d. thesis. Iowa State Univ. (Library Congress Card No. Mic.-74-23766). 82pp. Univ. Microfilms, Ann Arbor, Mich. (1974).
- SHELL CHEMICAL CORPORATION: Handbook of aldrin, dieldrin, and endrin formulations. The Woodrow Press, Inc. New York. 195pp. 1959.
- U.S. DEPARTMENT OF HEALTH, EDUCATION, AND WELFARE: Pesticide and analytical manual. 1(212.1). U.S. Government Printing Office, Washington, D.C. 1970.
- U.S. Environmental Protection Agency: Methods for organic pesticides for water and wastewater. Nat. Environ. Res. Center, Cincinnati, Ohio. 38pp. 1970.