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Pesticide Residues in Channel Catfish From Nebraska[1]

N. P. Stucky

ABSTRACT

Channel catfish (Ictalurus punctatus) *were collected in all of the major watersheds in Nebraska during the summer of 1964. Individual fat samples and composite blood samples obtained from these fish were analyzed to determine the concentrations of residues of DDT and its metabolites (o,p'-DDT, p,p'-DDT, p,p'-DDD, and DDE) and dieldrin. A total of 178 fish, collected from 18 sites, were analyzed. As expected, the fat samples contained higher concentrations of the pesticides than did the blood samples. DDT residues were found in all fat samples, and average levels from 10 fish sampled at each site ranged from a low of 2.2 ppm in the sandhills region to a high of 92.2 ppm in Salt Creek below Lincoln, Nebr. Average dieldrin residues in fat samples ranged from 0.1 to 6.7 ppm. All values are expressed as ppm (μg/g) of residue detected in each sample of fat. The composite blood samples were found to contain DDT residue concentrations ranging from a low of less than 0.01 ppm to a high of 0.16 ppm. Dieldrin residue concentrations ranged from a low of less than 0.01 ppm to a high of 0.07 ppm.*

Introduction

In 1964, the Research Division of the Nebraska Game and Parks Commission initiated a statewide exploratory study to determine the extent of environmental contamination by pesticides. The primary objective of this investigation was to determine the concentrations of DDT (including its isomers and metabolites) and dieldrin residues in Nebraska watersheds using the channel catfish, *Ictalurus punctatus*, as an indicator species. In 1968, Lyman *et al* (*7*) used fish to demonstrate the presence of DDT in an aquatic environment. Anderson and Everhard (*1*) conducted similar studies relating to DDT in fish, and Weiss (*11*) discussed the use of fish as indicator organisms to determine the extent of environmental contamination by pesticides.

The channel catfish was chosen as the species to be analyzed primarily because of its ubiquitous occurrence, omnivorous food habits, and value as a sport and food fish.

During the summer of 1964, fat samples and composite blood samples were obtained from channel catfish in all major watersheds throughout the State. Analyses for DDT and dieldrin were performed in our laboratory. Data were evaluated to determine the relative concentrations of pesticide residues in channel catfish within each watershed.

Due to the mobility of channel catfish as reported by Welker (*12*) and Muncy (*9*), the levels expressed in this study should be interpreted as quantitative values representing the amount of DDT and dieldrin contamination of fish at the collection site but not throughout entire watersheds. However, results of these analyses can indicate areas in the State where pesticide concentrations exceed the maximum allowable level established by the Food and Drug Administration and therefore warrant additional study.

Sampling Procedures

Eighteen collection sites were selected for study (Fig. 1), representing all of the major drainage systems in Nebraska. Topography and land use in the watersheds were the primary considerations in selection of the collection sites. With respect to these physical factors, homogeneity of the watersheds above the collection sites was sought as much as possible.

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Samples were collected during a 2-week period in the summer of 1964. Ten channel catfish were collected at each site by means of either a back-pack shocker or rotenone. An exception to this was the site on the Middle Loup River from which only eight fish were collected after several days of sampling. To eliminate the possibility of introducing additional variables, an effort was made to obtain fish ranging from 25 cm to 35 cm in total length. At several sites however, it was necessary to deviate from this range in order to obtain a sample of 10 fish.

In the field a sample of visceral fat was obtained from each fish, and one or more composite blood samples were obtained for each collection site. Blood samples were obtained by removing the caudal fin with scissors and allowing blood to drain into a vial. Blood and fat samples were then placed in a cooler where they were held until freezing.

Analytical Procedures

Both the quantitative and qualitative analyses were carried out using an Aerograph Model 204, dual column, electron capture gas chromatograph. The following instrument parameters were used:

Columns:

For dieldrin and DDE—Metal, ⅛″ x 5′ containing 4½′ of 4% SE-30/6% QF-1 and 6″ of 3% OV-17 on 60/80 Chromosorb W, regular solid support

For o,p'-DDT, p,p'-DDT, and p,p'-DDD—Glass, ⅛″ x 5′ containing 11% OV-17/QF-1 on 80/100 Gas Chrom Q, DMCS treated solid support

Temperatures:

Column	185 C
Detector	195 C
Injector	230 C

Carrier Gas Flow Rate (Nitrogen):

For dieldrin and DDE column—75 and 35 ml/minute for fat and blood, respectively

For o,p'-DDT, p,p'-DDT and p,p'-DDD Column—42 and 35 ml/minute for fat and blood, respectively

The method employed in the extraction of pesticides was rapid and convenient for an exploratory study of this nature; however, it is not recommended for a study where extreme accuracy is of paramount importance. The single distribution method was first applied to the extraction of pesticides in 1965 by Beroza and Bowman (*2*). The solvent system used was as follows:

Lower phase: 70% DMF (dimethylformamide)
Upper phase: isooctane

Each fat sample was carefully weighed to within 0.0001 g, attempting to maintain a range within 0.1-0.3 g. The sample was then placed in 5 ml of 70% DMF and subjected to 10 minutes of ultrasonic vibration. Five ml of isooctane was added, and this mixture was shaken vigorously for 2 minutes. Equilibration between the two phases was accomplished by either centrifugation or allowing the mixture to stand for a period of 2 to 24 hours. Aliquots (1-6 μl) of the upper phase were then qualitatively and quantitatively analyzed for pesticides by injection into the columns described above. Each sample was analyzed by this method, and a separate injection for each of the five standards (pesticide samples of a known concentration) followed every sample.

The minimum level to be recorded quantitatively was set at 0.01 ppm for dieldrin, DDE, *o,p'*-DDT, *p,p'*-DDT, and *p,p'*-DDD. Recovery effectiveness ranged from 76% to 99%. The results expressed in this study were corrected accordingly.

Results and Discussion

The exploratory nature of this study warranted expression of the results only as average concentrations present at each collection site. Data were not subjected to statistical treatment such as the calculation of standard error because the mobility of channel catfish makes it unreasonable to assume that all fish at a given location should have the same residue concentration. As pointed out by Buhler *et al.* (*4*), residue concentrations may also be a function of size of fish. To demonstrate extremes in concentrations found at each collection site, ranges are included in the data presented for fat samples. Because blood samples were comprised of up to five fish, ranges are not given.

FAT

DDT

The residue concentrations of DDT and its isomers and metabolites found in the fat of channel catfish in Nebraska watersheds are presented in Table 1 and Fig. 1. The values represent averages for the 10 fish collected from each sampling site. While samples were analyzed for the residual concentration of each specific isomer and metabolite of DDT and are expressed as such, the most significant value is the "total DDT" (Table 1) because, as pointed out by Spencer (*10*), the ratio of DDT to its metabolites changes in accordance with the length of storage time prior to analysis. The samples collected in this study were analyzed over a 1-year period.

Residues ranged from a low of 2.16 ppm (Niobrara River) in the sandhills region, to a high of 92.16 ppm in Salt Creek below Lincoln, Nebr.

A followup study to locate the main source of DDT pollution is presently being done on Salt Creek where the concentration was found to be 92.2 ppm. Laboratory analyses showed these fish to be comprised of approximately 9% fat. Therefore, by extrapolation, these fish contained approximately 10.3 ppm DDT, on a whole-fish basis, well above the maximum allowable level of 5.0 ppm established by the Food and Drug Administration.

The results of this investigation are supported by a study by Henderson *et al.* (*5*). Three samples comprised of a total of 15 channel catfish, collected from the Missouri River at Nebraska City, were analyzed for residues of DDT and its metabolites. Concentrations ranged from 0.21 to 2.03 ppm on a whole-fish basis. This compares to the range of 0.15 to 2.20 (1.67 - 24.46 ppm on a fat basis) found in fish from the Missouri River in this study.

Dieldrin

Dieldrin residues were found in fat samples from channel catfish collected from all 18 Nebraska watersheds. Residual concentrations found in the various watersheds are presented in Table 2 and Fig. 2. Values shown are averages of the 10 fish collected at each site. Residues ranged from a low of 0.08 ppm in the Middle Loup River to a high of 6.71 ppm in the Missouri River.

FIGURE 1.—*Residues of DDT (including its isomers and metabolites) in fat of channel catfish from Nebraska watersheds (values expressed to nearest 0.1 ppm)*

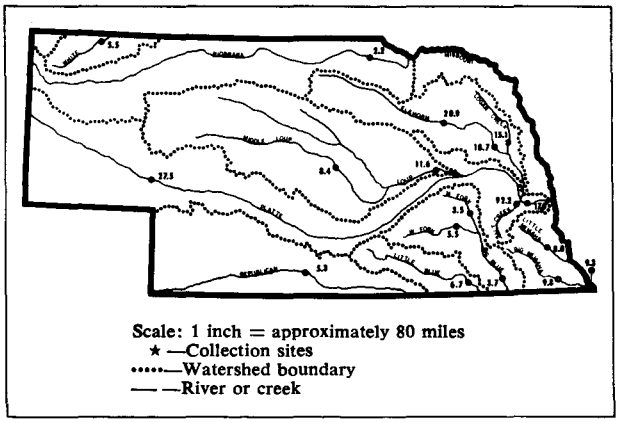

FIGURE 2.—*Residues of dieldrin in fat of channel catfish from Nebraska watersheds (values expressed to nearest 0.1 ppm)*

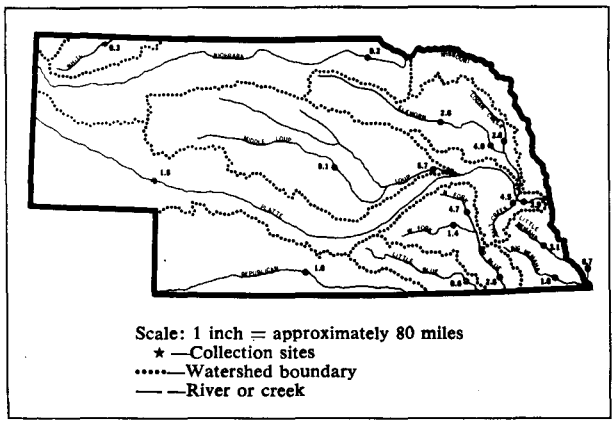

TABLE 1.—*Concentration of DDT in fat samples from 10 channel catfish collected at each site*

Watershed	Average Residue Levels in PPM					Range of total DDT and metabolites (10 fish) (ppm)
	DDE	*o,p'*-DDT	*p,p'*-DDT	*p,p'*-DDD	Total DDT	
Missouri River	3.54	0.13	3.17	2.41	9.25	1.67- 24.46
Niobrara River	1.01	0.03	0.80	0.32	2.16	0.72- 2.99
White River	0.92	0.03	3.81	0.75	5.51	2.19- 8.62
Platte River						
Lower	2.84	0.86	8.57	5.28	17.55	8.58- 35.86
Upper	17.89	0.24	5.06	4.35	27.54	5.86- 87.17
Salt Creek	10.57	4.50	39.85	37.24	92.16	13.61-258.68
Loup River	3.81	0.62	3.54	3.59	11.56	0.28- 26.59
Middle Loup River	4.52	0.01	2.66	1.25	8.44	2.80- 28.66
Elkhorn River						
Lower	5.13	3.11	5.23	5.24	18 71	2.71- 38.79
Upper	10.29	2.24	4.29	4.03	20.85	4.75- 56.86
Logan Creek	4.40	0.05	2.65	8.04	15.14	9.60- 23.22
Little Nemaha River	2.44	0.41	5.25	1.74	9.84	3.74- 31.24
Big Nemaha River	4.38	0.06	2.42	1.14	8.00	3.64- 35.50
Big Blue River	1.56	0.12	1.43	0.63	3.74	1.73- 10.44
West Fork Big Blue River	2.33	0.03	1.54	1.64	5.54	2.87- 10.83
North Fork Big Blue River	1.68	0.02	1.28	0.51	3.49	1.28- 6.91
Little Blue River	1.60	0.14	4.04	0.87	6.65	1.93- 21.02
Republican River	1.39	0.14	2.63	1.17	5.33	1.17- 16.91

The dieldrin residue concentration found by Henderson *et al.* (*5*) in channel catfish from the Missouri River ranged from 0.04 to 0.18 ppm on a whole-fish basis. The concentrations found in this study ranged from 0.32 to 1.43 ppm (2.88-12.86 on a fat basis).

BLOOD

As would be expected, the chlorinated hydrocarbon pesticide residue concentrations were considerably lower in blood samples than in fat. Results of the analyses of composite blood samples from each watershed (with the exception of the Middle Loup River and the Elkhorn River, upper site, from which no samples were obtained) are presented in Table 3. The composite blood samples were found to contain average DDT residue concentrations ranging from a trace (<0.01 ppm) to a high of 0.16 ppm. Dieldrin residue concentrations ranged from a trace to a high of 0.07 ppm.

TABLE 2.—*Concentration of dieldrin in fat samples from 10 channel catfish collected at each site*

Watershed	Dieldrin Residues in PPM	
	Average	Range
Missouri River	6.71	2.88-12.86
Niobrara River	0.16	<0.01- 0.72
White River	0.26	0.08- 0.53
Platte River		
Lower	2.88	1.36- 4.79
Upper	1.78	0.69- 5.58
Salt Creek	4.52	2.58- 7.35
Loup River	5.68	1.87- 9.55
Middle Loup River	0.08	<0.01- 0.27
Elkhorn River		
Lower	3.98	0.74-11.25
Upper	2.79	0.52- 5.76
Logan Creek	2.62	1.95- 3.10
Little Nemaha River	3.08	2.08- 4.13
Big Nemaha River	0.99	0.31- 1.82
Big Blue River	2.55	1.32- 3.97
West Fork Big Blue River	1.42	0.29- 2.19
North Fork Big Blue River	4.72	0.69- 7.64
Little Blue River	0.63	0.34- 1.17
Republican River	0.98	0.33- 2.03

TABLE 3.—*Concentration of pesticides in blood samples from channel catfish*

[T = Trace, <0.01 ppm]

Watershed	Residues in PPM	
	Total DDT	Dieldrin
Missouri River	0.15	0.07
Niobrara River	T	T
White River	0.01	T
Platte River		
Lower	0.05	0.04
Upper	0.13	0.01
Salt Creek	0.12	0.02
Loup River	0.05	0.03
Middle Loup River	—	—
Elkhorn River		
Lower	0.06	0.03
Upper	—	—
Logan Creek	0.16	0.07
Little Nemaha River	0.07	0.03
Big Nemaha River	0.06	0.02
Big Blue River	0.01	0.02
West Fork Big Blue River	0.12	0.01
North Fork Big Blue River	0.03	0.02
Little Blue River	0.12	0.01
Republican River	0.02	0.01

NOTE: Samples were comprised of blood from 1-5 fish.

A review of literature indicated that blood samples are not generally used in pesticides monitoring work. Bridges *et al.* (*3*) found that blood samples from black bullheads, *Ictalurus melas*, contained a high of 2.7 ppm DDT and metabolites. Samples were obtained 13 months after the farm pond in which the fish were held had been treated with 0.02 ppm DDT. Witt *et al.* (*13*) found that a good correlation existed between DDT in blood and the amount in adipose tissue. Studies of the Mississippi River fish kills by Mount *et al.* (*8*) indicate that acute toxicity, resulting from endrin, can be diagnosed from blood concentrations, which are independent of time of exposure and water concentration. Johnson (*6*) suggests that for monitoring work, analyses of blood samples may be more practical than other techniques more commonly employed.

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See Appendix for chemical names of compounds mentioned in this paper.

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