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The toxicity to warm-water fishes of certain cyanide plating baths before and after treatment by the alkali-chlorination method[1]

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by

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The safe disposal of wastes containing cyanide salts has always offered many difficulties to the operators of electroplating plants because of the toxic nature of these substances to aquatic life and the necessity of using large amounts of water for washing purposes. When cyanide wastes are discharged directly into a stream large volumes of dilution water are required to render the effluent innocuous, and in many instances the water supplies essential for such dilutions are limited. The plant therefore either must resort to some other method of disposal or must run the risk of contaminating public waters. Although several methods have been developed in the past to eliminate or reduce the hazard of cyanide disposal, they have not been universally accepted by the plating industry because of the high cost of the equipment involved or the amount of technical help required to follow such procedures.

Since many of the plating plants of today are small establishments, it is considered highly desirable that a method be developed which can be used satisfactorily in both small and large plants. The alkali-chlorination method, sponsored by E. I. Du Pont de Nemours & Company, appears to be applicable to such operations. It consists of treatment of plating wastes with chlorine in the presence of an alkaline medium, breaking down the CN radical through a series of chemical reactions (Dobson, 1947), and destroying the characteristic toxic ion. Naturally, following the development of this method it was necessary to determine the extent in reduction of toxicity. Since many plating wastes eventually reach streams containing fish populations, it was believed that fish should be used as experimental animals in a series of tests aimed at establishing an index of toxicity which would facilitate direct comparisons of the toxicity of untreated and treated wastes. A study of this nature was undertaken during the winter and spring of 1946 and 1947 by the Institute for E. I. Du Pont de Nemours & Company which has borne the cost of the experiments.

Acknowledgements

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Manager, Sodium and Cyanide Products Development, of the Du Pont Company, for his cooperation and suggestions during the course of the study.

Materials and methods

The chemicals used in these tests, supplied by Du Pont, consisted of untreated and treated copper plating baths, zinc plating baths, and carburizing salts solution. These baths were prepared specifically for the present study, at higher than normal concentrations. The analyses covering these plating solutions, as supplied by Du Pont, are quoted as follows:

"A. Du Pont High Speed Copper Plating Bath

Du Pont High Speed Copper Plating Salts were mixed with 9 gallons of water in an amount sufficient to obtain a solution with a total cyanide content of 5 percent expressed as NaCN.

1. Untreated - Two 1-gallon portions of this solution were set aside (NaCN content 5.0 percent; pH over 13).
2. Chlorinated - The remainder of the solution (approximately 7 gallons) was treated with 20 pounds of NaOH and 16 pounds of Cl_2 (4.9 pounds Cl_2 per pound NaCN). Properties of the final liquor were: specific gravity, 1.32 $\left(\frac{25}{4}\right)$; pH, 11.8; NaCNO, none detected; available Cl_2, none detected; NaCN, less than 1 ppm; Cu in filtrate, 1 ppm; and Cu in sludge 12.1 percent (dry basis).

B. Du Pont "Durobrite" Zinc Plating Bath

A sufficient amount of Du Pont "Durobrite" Zinc Plating Salts was mixed with 9 gallons of water to obtain a solution with a total cyanide content of 5 percent expressed as NaCN.

1. Untreated - Two 1-gallon portions of this solution were set aside (NaCN content 5.0 percent; pH over 13).
2. Chlorinated - The remainder of the solutions (approximately 7 gallons) was treated with 9 pounds of NaOH and 10 pounds of Cl_2 (3.1 pounds Cl_2 per pound NaCN). Properties of the final liquor were: specific gravity, 1.25 $\left(\frac{25}{4}\right)$; pH, 11.0; available Cl_2, 0.02 percent; NaCNO, 0.13 percent; NaCN, less than 1 ppm; zinc in filtrate, 5 ppm; and zinc in sludge, 60.2 percent (dry basis).

C. <u>Du Pont Carburizing Salt Bath</u>

A sufficient amount of a mixture of one part Du Pont Carburizing Salt to two parts Du Pont Heat Treating Salt #6 (the recommended starting mixture for the Du Pont Carburizing Salt Bath) was dissolved in 9 gallons of water to obtain a solution with a total cyanide content of 2.5 percent expressed as NaCN.

1. Untreated - Two 1-gallon portions of this solution were set aside (NaCN content, 2.5 percent).
2. Chlorinated - The remainder of the solution (approximately 7 gallons) was treated with 9 pounds of NaOH and 6.5 pounds of Cl_2 (3.6 pounds Cl_2 per pound NaCN). Properties of the final liquor were: specific gravity, 1.29 $\left(\frac{25}{4}\right)$; pH, 11.6; available Cl_2, 0.037 percent; NaCNO, 0.5 percent; and NaCN, less than 1 ppm."

Test fish consisted of the bluegill (<u>Lepomis macrochirus</u>) and the brown bullhead (<u>Ameiurus nebulosus</u>). The bluegills, 1-1/2 to 2-1/2 inches in length, were secured from Deep Lake, Oakland County, Michigan, and from the Wolf Lake State Fish Hatchery in Van Buren County, Michigan. The bullheads, which ranged in length from 2 to 3-1/2 inches, were obtained from Portage Creek, Jackson County, Michigan, and from the Comstock Park State Fish Hatchery in Kent County, Michigan. Care was taken in the collection of these fish to select only healthy individuals.

The water used for retaining the stock supply of fish, for the dilution of the plating materials, and for other purposes directly concerned with the study, was secured from Fleming Creek, Washtenaw County, Michigan. This water was considered to be representative of normal stream waters in which a moderate population of fishes could be sustained successfully. The total hardness (M. O. alkalinity) was 188 ppm. The pH averaged about 8.4. No free carbon dioxide or other noxious gases were present.

The test equipment consisted of 1-gallon glass jars, each equipped with a cover and an air release. All contacts with the plating elements were made with either glass or non-corrosive fixtures. The solution temperature was approximately that of the laboratory, --- 72° F.

The test procedure employed to determine the relative toxicity of the plating solutions to fishes consisted of preparing a series of stock solutions representing a wide range of concentration for each of the products, and exposing from 3 to 5 fishes to each concentration. The test jars were supplied with 3,000 cubic centimeters of stock solution and the fish were retained in a given solution for a period not exceeding 96 consecutive hours. At frequent intervals within this period the condition of the fish was noted and if any dead were present they were immediately removed to prevent possible contamination. During the entire study, control jars were in constant operation and a periodic check was made of the reserve stock of fish being retained in a holding tank within the laboratory. The toxicity of the plating baths was measured in terms of the reaction of fish exposed to varying concentrations. The degree of toxicity was evaluated in terms of the "consistent tolerance limit," an expression denoting the highest concentration at which all of the test animals survived a 96-hour exposure.

Procedure

Four groups of experiments involving 214 individual tests were undertaken during the study. The group tests consisted of determining the relative toxicity of (1) each of the three untreated and treated wastes, (2) the supernatant of the treated waste, (3) the sludge of the treated waste, and (4) the sludge of the treated waste after undergoing slight acidification.

The three raw plating solutions were found to possess high toxic values; the consistent tolerance limit (in terms of the actual residual cyanide present) was 0.4 ppm for the bluegill and 0.5 for the bullhead for both the copper and zinc plating solutions. The toxicity of the carburizing salts solution was found to be somewhat greater, the non-lethal limit being established at 0.25 and 0.375 ppm, respectively, for the bluegill and the bullhead.

Results of the toxicity tests are best expressed in degrees of dilution of the basic plating baths (Table 1). On this basis the values are expressed in higher units than when expressed in terms of cyanide content. The tolerance levels for the bluegill were 8, 8, and 10 ppm in the copper, zinc, and carburizing baths, respectively. The consistent tolerance levels for the bullhead were 10, 10, and 15 ppm, respectively.

Table 1.--The toxicity of untreated copper plating bath, zinc plating bath and carburizing salts bath to bluegills and bullheads

Concentration of basic plating baths in ppm	Materials used in test								
	Copper plating bath			Zinc plating bath			Carburizing salts bath		
	Residual NaCN content in ppm	Species and survival time[1]		Residual NaCN content in ppm	Species and survival time[1]		Residual NaCN content in ppm	Species and survival time[1]	
		Bluegill	Bullhead		Bluegill	Bullhead		Bluegill	Bullhead
		Survival time	Survival time		Survival time	Survival time		Survival time	Survival time
2,000	100.00	No test	No test	100.00	5 to 7 min.	5 to 8 min.	50.000	No test	No test
1,000	50.00	9 to 12 min.	7 to 12 min.	50.00	5 to 7 min	No test	25.000	3 to 4 min.	2 to 6 min.
500	25.00	6 to 14 min.	No test	25.00	No test	7 to 12 min.	12.500	6 to 12 min.	No test
200	10.00	No test	16 to 38 min.	10.00	8 to 12 min.	No test	5.000	No test	45 to 87 min.
100	5.00	11 to 24 min.	3 to 5 hours	5.00	No test	93 to 187 min.	2.500	6 to 15 min.	88 to 161 min.
50	2.50	No test	6 to 13 hours	2.50	No test	96 to 139 min.	1.250	No test	No test
40	2.00	No test	No test	2.00	22 to 40 min.	No test	1.000	No test	No test
20	1.00	30 to 83 min.	31 to 96 hours	1.00	51 to 120 min.	3 to 5 hours	0.500	28 to 50 min.	31 to 96 hours
15	0.75	No test	No test	0.75	No test	No test	0.375	No test	96+ hours
12	0.60	60 to 142 min.	No test	0.60	3 to 4 hours	38 to 96 hours	0.300	No test	No test
10	0.50	3 to 3.5 hours	96+ hours	0.50	No test	96+ hours	0.250	96+ hours	96+ hours
8	0.40	96+ hours	96+ hours	0.40	96+ hours	No test	0.200	No test	No test
6	0.30	96+ hours	No test	0.30	96+ hours	No test	0.150	96+ hours	No test
5	0.25	No test	96+ hours	0.25	No test	96+ hours	0.125	No test	96+ hours
4	0.20	No test	No test	0.20	96+ hours	No test	0.100	96+ hours	No test
2	0.10	96+ hours	No test	0.10	No test	No test	0.050	96+ hours	No test
1	0.05	96+ hours	No test	0.05	No test	No test	0.025	96+ hours	No test
0	0.00	96+ hours	96+ hours	0.00	96+ hours	96+ hours	0.000	96+ hours	96+ hours

[1] 5 fish used in each test

Throughout the tests, the fish (both bluegills and bullheads) either succumbed within a few hours or less, or (in most instances) survived the 96-hour exposure. This result suggested that the test solutions lost some of their toxicity during the time interval of the testing. Since a constant stream of air was being forced through the test solutions, it is possible that through hydrolysis the active toxic substances were quickly broken down.

A very significant change in the degree of tolerance of the fishes was noted when they were subjected to the plating solutions which had undergone the alkali-chlorination treatment. For these tests the consistent tolerance limits are expressed in terms of the original cyanide value before treatment. For the bluegill the limits were fixed at 250, 350, and 175 ppm for the copper, zinc, and carburizing solutions, respectively (Table 2). For the bullhead the non-lethal limits established for copper, zinc, and carburizing solutions were 300, 400, and 225 ppm. These toxicity values become much more significant when expressed in terms of dilution of basic plating baths (stock material). The consistent tolerance limits of the bluegill for the three products, copper, zinc, and carburizing solutions, were 5,000, 7,000, and 7,000 ppm, respectively; for the bullhead the values, in the above order, were 6,000, 8,000 and 9,000 ppm.

Comparison of the data presented in Tables 1 and 2, shows that the toxic values for each of the products were changed materially by treatment. From a low of 8 ppm of basic stock of copper plating solution representing the highest non-lethal concentration to bluegills, the value was increased to 5,000 ppm; by treatment for the zinc solution from 8 to 7,000 ppm; and for the carburizing solution from 10 to 7,000 ppm.

Table 2.--The toxicity of treated copper plating, zinc plating and carburizing salts baths to bluegills and bullheads

Concentration of basic plating baths in ppm	Materials used in test								
	Copper plating bath			Zinc plating bath			Carburizing salts bath		
	Original NaCN content in ppm	Species and survival time[1]		Original NaCN content in ppm	Species and survival time[1]		Original NaCN content in ppm	Species and survival time[1]	
		Bluegill	Bullhead		Bluegill	Bullhead		Bluegill	Bullhead
		Survival time	Survival time		Survival time	Survival time		Survival time	Survival time
80,000	4,000	7 to 8 min.	5 to 7 min.	4,000	5 to 6 min.	5 to 20 min.	2,000	6 to 8 min.	7 to 10 min.
60,000	3,000	4 to 8 min.	6 to 7 min.	3,000	8 to 9 min.	7 to 17 min.	1,500	5 to 7 min.	9 to 10 min.
40,000	2,000	5 to 8 min.	8 to 12 min.	2,000	9 to 11 min.	20 to 28 min.	1,000	7 to 9 min.	18 to 20 min.
20,000	1,000	9 to 10 min.	11 to 26 min.	1,000	10 to 15 min.	39 to 47 min.	500	32 to 51 min.	23 to 29 min.
15,000	750	16 to 18 min.	15 to 48 min.	750	15 to 23 min.	45 to 89 min.	350	24 to 63 min.	49 to 57 min.
10,000	500	160 to 208 min.	19 to 56 min.	500	15 to 96 hours	68 to 138 min.	250	13 to 17 hours	16 to 96 hours
9,000	450	No test	No test	450	16 to 96 hours	4 to 67 hours	225	30 to 96 hours	96+ hours
8,000	400	3 to 96 hours	11 to 18 hours	400	28 to 96 hours	96+ hours	200	44 to 96 hours	96+ hours
7,000	350	5 to 96 hours	29 to 96 hours	350	96+ hours	96+ hours	175	96+ hours	96+ hours
6,000	300	22 to 96 hours	96+ hours	300	96+ hours	96+ hours	150	96+ hours	96+ hours
5,800	290	31 to 96 hours	96+ hours	290	No test	No test	145	96+ hours	No test
5,400	270	44 to 96 hours	96+ hours	270	No test	No test	135	96+ hours	No test
5,000	250	96+ hours	96+ hours	250	96+ hours	96+ hours	125	96+ hours	96+ hours
4,000	200	96+ hours	96+ hours	200	No test	No test	100	96+ hours	96+ hours
000	000	96+ hours	96+ hours	000	No test	No test	000	96+ hours	96+ hours

[1] Five fish used in each concentration tested

Similarly, for the bullhead, the index of toxicity of the copper plating solution changed from a low of 10 to 6,000 ppm following treatment; for the zinc, from 10 to 8,000; and for the carburizing solution, from 15 to 9,000 ppm.

It can also be noted that although a significant reduction in toxicity has been expressed in values for the treated products the reduction does not come up to expectations on the basis of the original analyses which indicated that all or at least most of the detectable cyanide had been destroyed.

In order to achieve a more thorough understanding of the toxic nature of the treated materials, toxicity tests were run on the basic component parts: supernatant and sludge. It was found that the supernatant of each of the three products was more toxic than the basic stock. The consistent tolerance limit established for the bluegill in terms of ppm was 3,500 for the copper plating solution, 4,000 for the zinc plate, and 4,000 for the carburizing solution. The values of tolerance established for the bullhead were in general somewhat lower (Table 3): 3,000 ppm in the copper plating solution, 4,000 in the zinc, and 3,500 in the carburizing solution.

From the finding that the supernatants possessed higher toxic values than the composite solutions, it was deduced that the sludge would be less toxic. The sludge constituted from 15 to 20 percent of the total volume, and should have had some important role in the toxicity, one way or the other. Samples of sludge were removed from each of the three solutions by filtration, thoroughly washed to assure the entire removal of the supernatant, and employed in a series of toleration tests. Results presented in Table 4, in comparison with the

Table 5.--Relative tolerance of bluegills and bullheads to the supernatant of treated copper, zinc and carburizing salts baths

Concentration of supernatant liquor in ppm	Materials used in tests: Copper plating bath — Species and survival time[1]		Zinc plating bath — Species and survival time[1]		Carburizing salts bath — Species and survival time[1]	
	Bluegill	Bullhead	Bluegill	Bullhead	Bluegill	Bullhead
	Survival time	Survival time	Survival time	Survival time	Survival time	Survival time
40,000	8 to 12 min.	No test	No test	No test	5 to 12 min.	No test
20,000	16 to 22 min.	6 to 14 min.	28 to 47 min.	28 to 47 min.	24 to 26 min.	10 to 20 min.
15,000	No test	No test	No test	No test	38 to 62 min.	No test
12,000	No test	No test	No test	No test	1.5 to 16 hours	No test
10,000	2 to 16 hours	42 to 95 min.	2 to 18 hours	2 to 18 hours	No test	2 to 4.5 hours
9,000	No test	No test	No test	No test	1 to 22.5 hours	No test
8,000	No test	No test	No test	No test	No test	No test
7,000	12 to 16 hours	No test	No test	No test	12 to 16 hours	No test
6,000	No test	No test	No test	No test	27 to 96 hours	No test
5,000	29 to 57 hours	No test	34 to 96 hours	34 to 96 hours	36 to 96 hours	No test
4,000	36 to 96 hours	28 to 51 hours	96+ hours	96+ hours	96+ hours	4 to 18 hours
3,500	96+ hours	42 to 96 hours	No test	No test	No test	96+ hours
3,000	96+ hours	96+ hours	96+ hours	96+ hours	96+ hours	96+ hours
000	96+ hours	96+ hours	96+ hours	96+ hours	96+ hours	96+ hours

[1] Five fish used in each concentration listed

Table 4.--The toxicity to bluegills and bullheads of sludges derived from treated copper plating, zinc plating and carburizing salts baths, together with comparison of the toxicity of alkaline and acidified sludges.

Concentration of sludge in ppm	Materials used in tests								
	Alkaline sludge solution, pH 11.0 to 11.6						Acid sludge solution, pH 6.2 to 6.6		
	Copper plating bath		Zinc plating bath		Carburizing salts bath		Copper plating bath	Zinc plating bath	Carburizing salts bath
	Species and survival time[1]		Species and survival time[1]		Species and survival time[1]		Species and survival time[1]		
	Bluegill	Bullhead	Bluegill	Bullhead	Bluegill	Bullhead	Bluegill	Bluegill	Bluegill
	Survival time	Survival time	Survival time	Survival time	Survival time	Survival time	Survival time	Survival time	Survival time
80,000	6 to 13 min.	96+ hours	21 to 48 min.	96+ hours	7 to 18 min.	96+ hours	No test	No test	No test
60,000	No test	No test	16 to 96 hours	No test	No test	No test	No test	No test	No test
40,000	11 to 24 min.	96+ hours	96+ hours	96+ hours	3 to 96 hours	96+ hours	No test	1.5 to 2 hours	No test
20,000	96+ hours	96+ hours	96+ hours	96+ hours	96+ hours	96+ hours	16 to 96 hours	3 to 65 hours	40 to 90 min.
10,000	96+ hours	96+ hours	96+ hours	96+ hours	96+ hours	96+ hours	96+ hours	No test	96+ hours
8,000	No test	No test	No test	No test	No test	No test	No test	96+ hours	No test
7,000	96+ hours	No test	No test	No test	96+ hours	No test	No test	No test	No test
6,000	No test	No test	No test	No test	96+ hours	No test	No test	96+ hours	96+ hours
4,000	96+ hours	No test	No test	No test	No test	No test	No test	No test	No test
000	96+ hours	96+ hours	96+ hours	96+ hours	96+ hours	96+ hours	96+ hours	96+ hours	96+ hours

[1] Three fish used in each concentration tested

figures in Table 3, show that the sludge was appreciably less toxic than the supernatant. Even though only wide range toleration tests were performed, the consistent tolerance limit was established at much higher concentrations for sludge than for supernatant. Bluegills were found to tolerate concentrations ranging somewhere between 20,000 and 40,000 ppm for the three products, and the bullheads in all cases in excess of 80,000 ppm.

Further examination was made of the toxic nature of the sludge after treatment with an acid buffer. Buffering made it possible to determine whether or not an increase in toxicity would result from ionization of the sludge. Techniques similar to those practiced in the above experiment were employed in sludge separation: the sludge was removed by filtration, carefully washed several times and then mixed into a hydrochloric acid solution and allowed to stand for 20 hours before use. The pH was adjusted at 6.2 for the copper plating solution, 6.4 for the zinc, and 6.6 for the carburizing salts solution. Only one species of fish (bluegill) was employed in these tests. The results (Table 4) indicate that the sludge for all three products became more toxic when buffered with an acid. The consistent tolerance limits were somewhere between 8,000 and 20,000 ppm as compared with 20,000 to 40,000 values, the range of toleration for the species prior to acidification.

Discussion and summary

Treatment of three electroplating wastes by the alkali-chlorination technique resulted in an appreciable reduction in toxicity. The treated copper plating solution was found to be 625 times less toxic to bluegills and 600 times less toxic to bullheads than was a homogeneous part of the untreated product; treatment of the zinc plating solution resulted in a reduction in toxicity of 875 and 800 times for the bluegill and bullhead, respectively; and for the carburizing salts solution, the reduction was calculated at 700 and 600 times for the two species (Figure 1). By presenting the collective data from the study in another form, i.e., in relation to the ratio of the dilution water used, the economic value can be easily illustrated. For the untreated wastes, namely copper plating and zinc plating, the dilution water necessary to attain the highest non-lethal concentration approximated a ratio of 125,000 parts of water to 1 part of the test product. Following treatment, however, the ratio between dilution water and the waste was considerably reduced, amounting to only 200 to 1 for the copper plating bath and 143 to 1 for the zinc plating bath. For the carburizing salts bath, which formerly required a 100,000 to 1 dilution, the figure was altered to a 143 to 1 ratio.

Referring to the analyses of the untreated and treated materials, presented on pages 000, it is difficult to explain why in this study even lower values of toxicity were not obtained for the treated products, since the analyses indicated that the remaining residual cyanide at no time exceeded 1 ppm. It is believed that other chemicals, either singly, collectively, or by group action, contribute a major portion of the remaining toxicity. For example, sodium cyanate (NaCNO) is present in lethal concentrations in two of the three plating baths, namely: zinc and carburizing salts. Through experimentation the writer roughly established the maximum tolerance limit for the creek chub (Semotilus a. atromaculatus), a hardy fish, to sodium cyanate at 75 ppm. Assuming that the bluegill and bullhead are comparable to the creek chub in tolerance, it can be demonstrated that a moderate degree of the toxicity

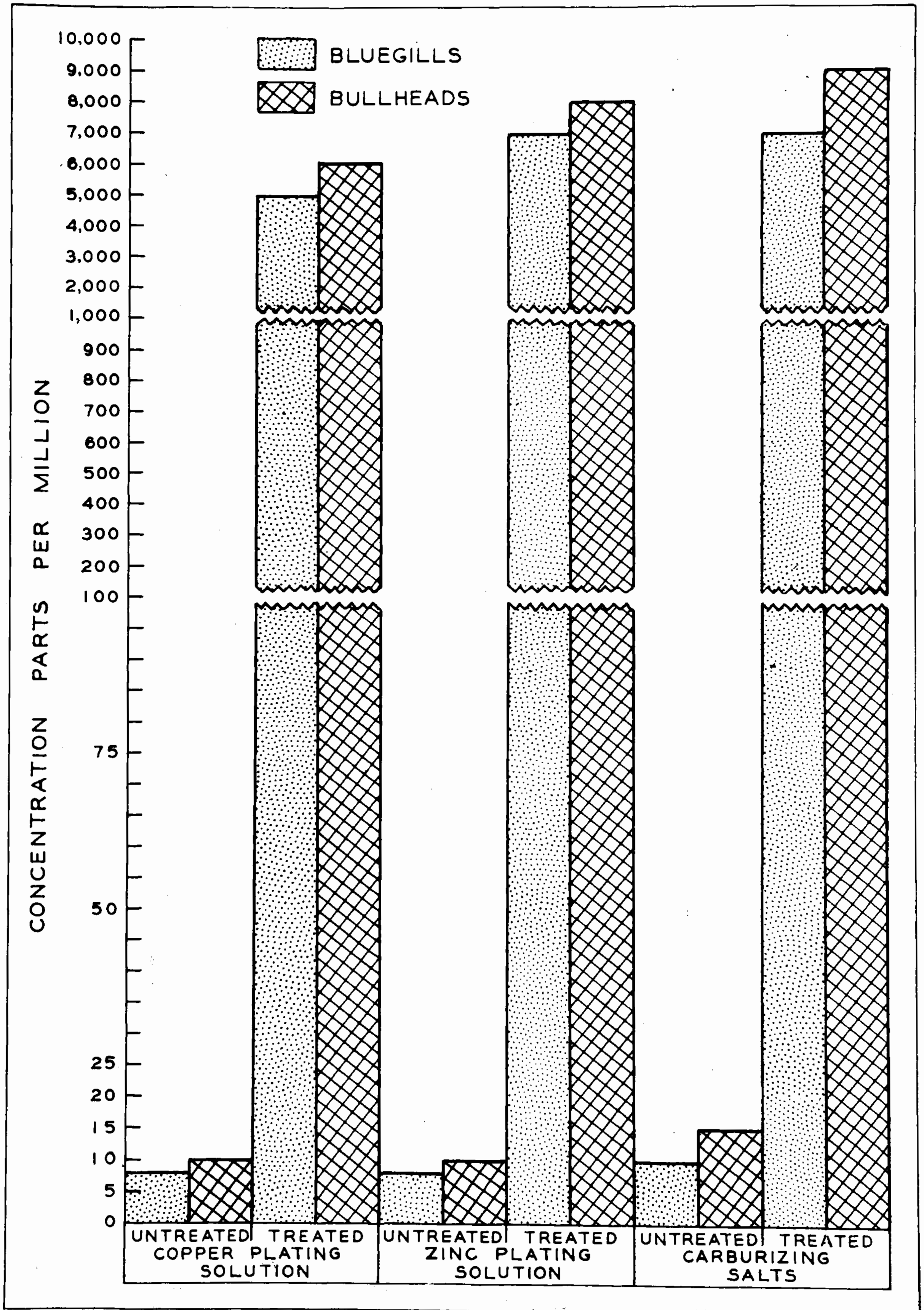

Figure 1.--Chart showing the tolerance of bluegills and bullheads to various concentrations of treated and untreated electroplating wastes.

in the treated products can be accounted for in this manner. It can be noted in Table 2 under the column headed "zinc plating bath" that the minimum concentrations which produced lethal effects to the bluegill and bullhead were established at 7,000 and 8,000 ppm, respectively, of basic stock. These concentrations, according to the analyses supplied, would contain a residual NaCNO content of approximately 9.1 and 10.4 ppm, thus providing a substantial proportion of the toxic elements present. The NaCNO values in the carburizing bath were even higher; at a minimum lethal concentration of 7,000 ppm for the bluegill and 9,000 ppm for the bullhead, the NaCNO content would be 35 and 45 ppm.

The presence of chloride (not included in the analyses supplied by the Du Pont Company) may be a contributing factor. Sodium chloride (NaCl) in a concentration of 10,000 ppm has been shown by Garrey (1916) to cause death to the pumpkinseed (Lepomis gibbosus) and the gold fish (Carassius auratus), fishes comparable in hardiness to the bluegill and bullhead; and Wiebe, et al. (1934) found that 10,000 ppm of calcium chloride ($CaCl_2$) was toxic to sunfishes. Since it is known that a high chloride content (in excess of 100,000 ppm) was present in these materials (Ridenour, 1948) it is only reasonable to assume that these salts are in part responsible for some of the toxicity. Many of the salts, being insoluble or only slightly soluble, undoubtedly were precipitated out in the sludge. However, since it was demonstrated in this study that the inert, slightly toxic sludge does possess high toxic values when acidified, it does constitute a menace when present in the composite treated plating baths.

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