THE EFFECTS OF TRACE AMOUNTS OF COPPER UPON SOME PHYSICAL PROPERTIES OF ELECTRODEPOSITED NICKEL

Thesis for the Degree of M. S.

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Robert James Rominski

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'The Effects of Trace Amounts of Copper Upon Some Physical Properties of Electrodeposited Nickel.'

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Robert James Rominski

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Physical Chemistry

J. T. Eyring

Major professor

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THE EFFECTS OF TRACE AMOUNTS OF COPPER UPON SOME PHYSICAL PROPERTIES OF ELECTRODEPOSITED NICKEL

By

Robert James Rominski

A THESIS

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I wish to express my sincere appreciation to Dr. D.T.Ewing for his quidance and assistance during the course of this research. I would like to extend my thanks to the American Electroplater's Society for the fellowship grant that made this research possible, and to the project committee, Mr. B.C.Case, Chairman, Hanson-VanWinkle-Munning Company; Mr. L.B.Sperry, Doehler-Jarvis Corporation: and Dr. R.C.Olsen, later succeeded by Mr. G.M.Cole, both of Fisher Body-Ternstedt Division, General Motors Corporation, for their contributions.

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INTRODUCTION

This investigation is the first of a series of the effects and removal of known amounts of impurities in four typical nickel plating solutions. This research was undertaken at the request of, and under a grant from the American Electroplater's Society. The material in this thesis consists of the effects of copper on electrodeposited nickel and its removal from nickel plating solutions.

The first study of the effects of copper in nickel plating solutions was carried out by the laboratories of Brass World in 1911 (1). In general they observed a darkening of the deposit upon the addition of small amounts of copper to the nickel solution. Later investigation carried out at the Bureau of Standards by Thompson and Thomas (2) and Haring (3) reported the permissable limits of copper in the nickel salts for nickel plating solutions, and its effects on the deposit, resulting in dark spongy deposits.

The limits of copper as an impurity in nickel solutions has been reported by Diggen (4) to be up to 0.5 oz./gal. (0.38 g./l.) in the gray nickel solutions and 0.001 oz./gal. (0.0075 g./l.) for the bright nickel solutions. From this observation, the bright nickel solutions have considerably lowered the upper limits of copper as an impurity. Eckelmann (5), Johnson (6), and Francis-Carter (7)

stated that the effects of copper in the bright nickel solutions will cause dull dark deposits in the low current density regions and a fogging or milky deposit in the other areas.

Several methods have been proposed for the removal of copper from nickel plating solutions. Among these is that set forth by Thompson and Thomas (2) in 1922. By making the bath slightly acid, scrap nickel is suspended in the nickel solution; the nickel precipitates the copper from the solution. The efficiency of this cementation process depends upon the surface area of the nickel exposed.

Fink and Rohrman (8), Raub and Bihlmaier (9), Diggin (4), and Waite (10) and others proposed the electrolytic removal of copper from nickel solutions by the use of low current densities.

The four typical nickel plating solutions used in this study consisted of a 2.2 pH and a 5.2 pH Watts type gray nickel solutions, a nickel cobalt alloy type bright nickel solution as per Weisberg Stoddard papent, and an organic type bright nickel solution as per Schlötter patent.

To study the general effect of copper as an impurity in nickel plating solutions and to set up limits of contamination, a series of panels were produced from the nickel solutions containing up to 500 milligrams of copper

per liter of solution. The upper limit of 100 milligrams was established, as the deposits obtained from the nickel solutions containing an excess of this amount were considered unusuable. Panels were produced from the four nickel solutions.

In this investigation, special attention has been given to the following properties:- appearance, adherence, salt-fog corrosion resistance, ductility, and hardness. In addition, the throwing power and current efficiency was studied. The removal of copper from nickel solutions was studied using the methods of electrolytic purification and high pH treatment.

No attempt has been made to evaluate these properties in exacting physical units. Rather, the trend of the change in the property under consideration was noted as the concentration of the impurity was changed. The results were reported in relative rather than absolute values using a deposit from a pure plating solution as a standard. The methods of testing were not the most precise, nor did they require expensive apparatus, but were designed for the layman operator to determine quickly whether or not impurities may be present.

To better study these effects of impurities, it was necessary to prepare nickel solutions free from impurities. Two methods have been used, chemical precipitation and electrolysis.

EXPERIMENTAL

A. Technique:

1. Preparation and purification of solutions.

The plating solutions used in this investigation are listed in the following tables:

Table No. 1.

Watts type nickel plating solution.

Nickel sulfate	240 grams/liter	32 oz./gallon
Nickel chloride	45 " "	6 " "
Boric acid	30 " "	4 " "
Temperature	50°C.	122°F.
pH (electrometric)	2.2 and 5.2	
Current density	40 amperes per square foot.	

Table No. 2.

Nickel cobalt (18%) alloy type solution.

Nickel sulfate	240 grams/liter	32 oz./gallon
Nickel chloride	45 " "	6 " "
Boric acid	30 " "	4 " "
Nickel formate	45 " "	6 " "
Cobalt sulfate	15 " "	2 " "
Ammonium sulfate	0.75 " "	0.10 " "
Formaldehyde	2.50 " "	0.33 " "
Temperature	55°C.	132°F.
pH (electrometric)	3.75	
Current density	40 amperes per square foot.	

Table No. 3.

Organic type nickel solution

Nickel sulfate	262.5 grams/liter	35 oz./gal.
Nickel chloride	60 " "	8 " "
Boric acid	34 " "	4.5 " "
Nickel benzene disulfonate	7.5 " "	1 " "
Triaminotolyldiphenylmethane	0.14 ml./liter	
Temperature	55°C.	132°F.
pH (electrometric)	3.2	
Current density	40 amperes per square foot.	

The plating solutions were made up in twenty liter stock solutions. The purification of these was accomplished by the high pH precipitation of 5.5 to 6.0 (electrometric) with nickel carbonate and electrolysis at a current density of 2-7 amperes per square foot on a corrugated cathode. It has been found that about 100 ampere hours per gallor of solution will remove the heavy metal ion concentration to spectroscopic traces. Any organic impurities present were removed after electrolysis had been completed by the addition of about 7.5 grams per liter of activated carbon of a good commercial grade. After 24 hours of agitation and a solution temperature of 70-75°C. the solution was filtered and ready for use. King (11) has described the proceedure in greater detail.

2. Preparation of nickel plated panels.

The steel used for the bent cathodes was low-carbon, cold rolled tin can stock of 0.01" thickness, with an R.M.S. value of 10 and supplied through the courtesy of Dr. Richard Wick of the Bethlehem Steel Corporation.

The strip steel was cut to a standard size of 2"x 8". A lip of 1.25" was formed by bending the lower edge of a 2"x 8" panel 90°. A line was scribed 3.5" from the bend across the panel. This gave an area of 0.08 square feet of surface to be plated.

The panel was degreased with carbon tetrachloride, numbered, and given an electrocleaning prior to plating. The composition of the electrocleaner is given in Table 4.

Table No. 4.

Electrocleaner composition		
Sodium hydroxide	21 grams/liter	2.81 oz./gallon
Sodium metasilicate	15 " "	2.01 " "
Tri-sodium phosphate	18 " "	2.41 " "
Temperature	80°C.	167°F.
Current density	75 amperes per square foot.	

The steel panel to be cleaned was made the anode in the electrocleaner and current was passed for two minutes. It was rinsed in running water, checked for water breaks, and then dipped in a 20% hydrochloric acid solution for two minutes. This was followed by two running water rinses, the latter being distilled water. The panel was then ready for plating.

The panels were plated in 1 liter glass battery jars with a current density of 40 amperes per square foot, operating temperature of the bath in use, and at an agitation rate of 4 feet of solution past the cathode per minute. The concentration of copper was maintained by

reference to depletion curves of the impurity from the nickel bath in use. The depletion rate data was obtained for each of the four nickel solutions by electrolysis at a current density of 40 amperes per square foot from the bath containing the upper limit os contamination. The electrolysis was maintained until the lower limits of contamination had been passed. Samples were taken at intervals and analysed for copper concentration using the colorimetric method of analysis as set forward by Serfass and Levine (12).

Figure 1. illustrates the rate of depletion of copper from the four nickel solutions at 40 amperes per square foot. Figure 2. is the calibration curve for the determination of copper in nickel solutions as copper diethyl dithiocarbamate in a mixture of amyl acetate and ethyl alcohol.

The impurity concentrations were maintained within 10% throughout the plating runs by timed additions. The solutions were analyzed for copper each hour of plating time as a further check. The pH of the solution was adjusted to the operating range before plating, and this pH was maintained by frequent checks. A Beckman electrometric pH meter was used for this purpose. The temperature of the solutions was maintained by means of a water bath. All the plating solutions were filtered before use daily, and in the case of the organic bright nickel bath, filtration

was necessary after each plated panel. A wetting agent, sodium lauryl sulfate, in sufficient amount to lower the surface tension to 35 dynes per square centimeter, was required to relieve the pitting encountered in this bath.

After plating was completed, the panel was removed from the solution, rinsed in running tap water and distilled water, and wiped dry with a clean cheese cloth. The dry panels were stored in a large dessicator containing calcium chloride.

The chemicals and anodes necessary for this project were furnished through the courtesy of the Hanson-Van Winkle-Munning Co. and the Udylite Corp.

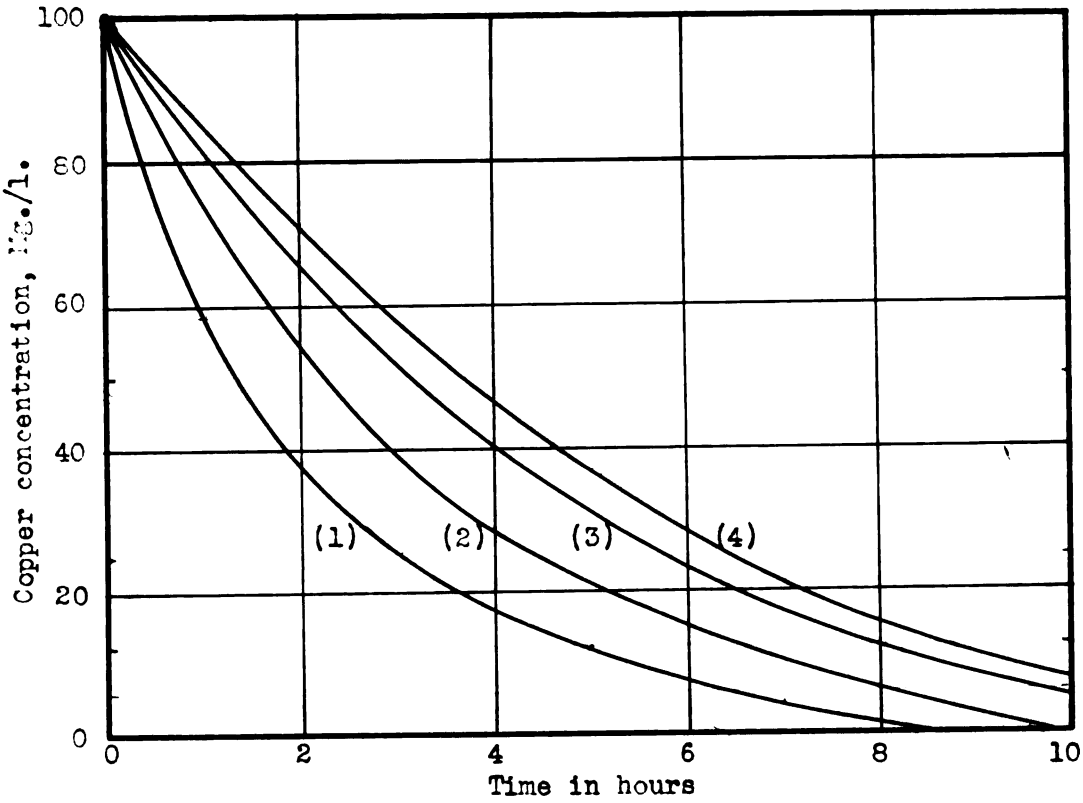


Figure 1

Depletion rate of copper at 40 amperes per square foot.
(1) Organic bath (2) Watts 5.2pH (3) Watts 2.2pH (4) Ni-Co.

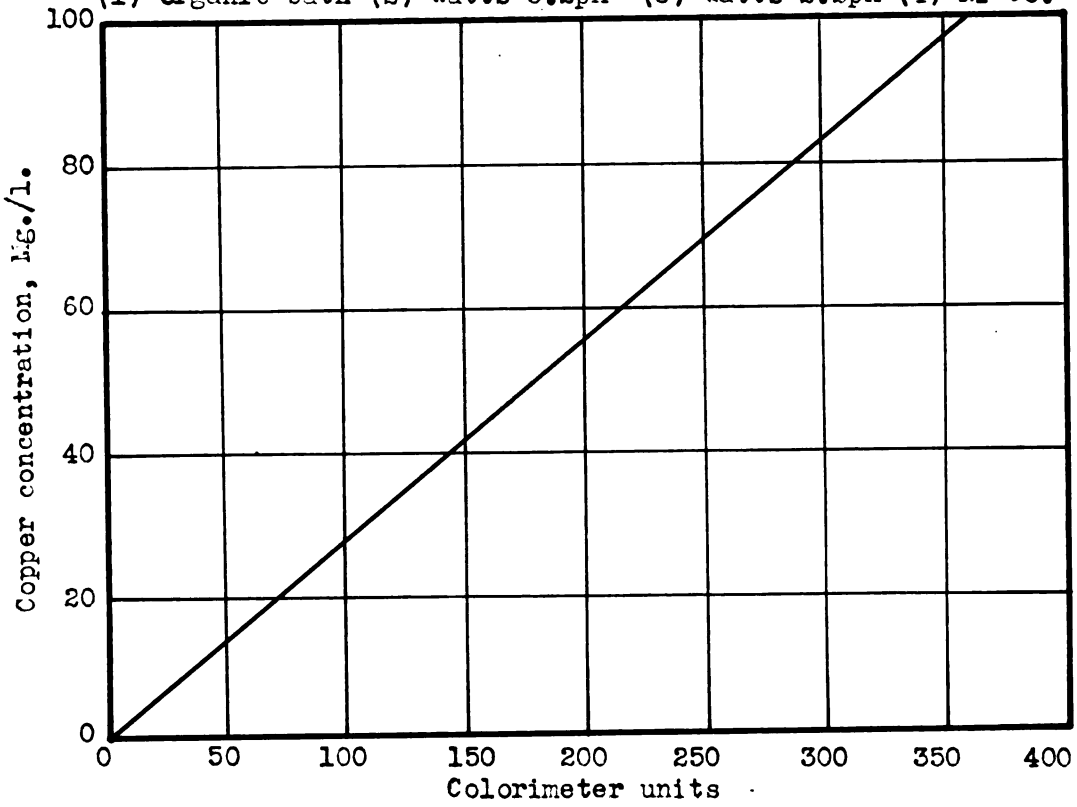


Figure 2

The calibration curve for the determination of copper in nickel solutions.

B. EFFECTS OF COPPER AS AN IMPURITY ON SOME PHYSICAL PROPERTIES OF NICKEL DEPOSITS

1. Appearance:

It has been generally known that small amounts of copper in nickel plating solutions effects the appearance or brightness of the deposits, especially in the recessed areas receiving low current densities. This physical property has been the criterion for the necessity of removal of copper because it is the most easily recognized as a source of rejection in the plating industry. Without a doubt the cathodic current density can change the numerical limits of copper contamination at which appearance may be effected. The limits stated hold true for the current densities found and the copper concentrations tested.

Two tests were used to describe the effects of copper on the appearance of 0.001" deposit thickness panels. First, for the gray nickel deposits, the panel was compared to the Eastman Gray Scale and assigned a value. Second, for the bright nickel deposits, the panels were classified in a range from mirror bright to a dull matte. Table 5 summarizes the effect of copper on appearance for the four nickel solutions.

Table 5.

Effect of copper as an impurity on the appearance of nickel deposits.

Cu. conc. Mg./l.	Watts 2.2pH	Watts 5.2pH	Ni-Co 3.75pH	Organic 3.2pH
0	2	2	mirror bright	dull luster
10	2	2	milky	dull luster
25	2	2	milky	dull luster
50	2	2-3	milky	milky
75	2	2-3	dull luster	milky
100	2	2-3	dull luster	milky

In general, brightness of deposits is effected when the copper concentration reaches 10 mg./l. in the solution, the effects being noticed in areas which received from 6-8 amperes per square foot. At 50 mg./l. practically all current densities are adversely effected. As appearance is the most easily evaluated physical property, it was not deemed necessary to give results in extreme detail. The deposits from all four baths were ultimately effected by increasing copper contents, with the Watts type deposit changing somewhat later than the bright deposits. Roughness of all the deposits, starting at the edges, became noticeable when the copper reached 50 mg./l. extending into other areas as the copper increased to 100 mg./l.

2. Adherence:

To evaluate the adherence of the nickel coating to the steel base, a simple test was devised which was the most adaptable and the nearest to the most satisfactory methods used in industry at this date. This test consisted of bending the lip of a 0,001" deposit thickness panel 180° around a 3/16" mandrel along the middle and across the short dimension. The deposit was observed for non-adherence along the bend with the aid of a magnifying glass. This allowed observation of non-adherence at a range of deposit thicknesses.

With increasing copper contents up to 100 mg./l., the adherence of the nickel coating to the steel base showed no discernible change in any deposit, at any current density area from the four nickel solutions.

3. Ductility:

It has been found that a nickel deposit on steel is not sensitive enough for a ductility test, and, therefore a stripped deposit was prepared for testing. This stripped deposit was made by plating a 0,001" deposit of nickel on a slightly oxidized surface. The deposit will cover the prepared surface completely but may still be readily peeled from the surface. The oxidized surface was prepared by repeating the cleaning and acid dip cycle on a steel panel plated with 0.0003" thickness of nickel

The stripped deposit was cut into 1"x2" strips and creased between the fingers across the short dimension. By alternately creasing and straightening, rupture will soon occur. By this method, the results were reported as an increase, a decrease, or no change in ductility in relation to the pure deposit.

Discernible changes in the ductility of the nickel deposits appeared with a copper content of the solution from 10 to 25 mg./l. Bending a plated steel panel or a stripped deposit showed definite and serious loss of ductility. As the copper content of the solution increased the ductility of the deposits decreased. No units for reporting this test are available and it is not possible to state where a deposit is useless because limits vary with the end use.

4. Hardness:

The vertical portion of the bent cathodes deposited to a thickness of 0.002" nickel was tested for hardness by means of a Knoop hardness tester. Table 6 gives the gain or loss in hardness of deposits from the four solutions.

Table No. 6.

Effect of copper as an impurity on the hardness of nickel deposits. *

Cu. conc. Mg./l.	Watts 2.2pH	Watts 5.2pH	Ni-Co 3.75pH	Organic 3.2pH
10	+ 2.8%	+ 27.2%	- 15.8%	+19.1%
25	+17.2	+ 22.6	-22.6	+12.3
50	+77.8	+13.6	+13.2	+18.2
75	+86.0	+10.1	+12.2	+20.5
100	+15.1	+14.3	-19.1	+ 7.8

* Expressed in percentage change from the pure deposit.

The deposits from the Watts bath showed an increase in hardness with the first addition of copper. The bright deposits followed this pattern but were slightly delayed. A degree of inconsistency was evidenced between hardness figures on the same type of deposit with various amounts of copper present in the plating solution. As the deposition of copper present with nickel is subject to great variations as a result of plating variables, it is not difficult to attribute the variations in hardness measurements to locale variations of copper content in the deposit.

The above test was conducted at the National Bureau of Standards, Washington D.C., through the courtesy of Dr. William Blum, Director of the Electrochemical Division.

5. Salt spray corrosion resistance:

The effects of the presence of copper in varying amounts upon the salf fog corrosion resistance of nickel coatings upon steel were determined using three thicknesses of nickel deposits, 0.0003", 0.001", & 0.0015". These panels were prepared in triplicate for each pure bath and each concentration of impurity, this enables observation of the effect of impurity upon increasing deposit thickness. The vertical portions were tested for corrosion resistance in a commercial type salt spray (fog) cabinet. The conditions followed are as described in the A.S.T.M. standards (13).

The breakdown was recorded after a condition was reached comparable to that of a set of standards previously prepared as a standard breakdown point.

Testing the deposit as plated produced results surprisingly consistent except for one or two of the 0.0015" deposits. Results were tabulated and recorded percentagewise using the hours of resistance to a salt fog atmosphere to a standard breakdown of a deposit from a clean plating bath as 100% performance. For all practical purposes, the results from the performance of the two thicknesses of nickel deposits, 0.001" and 0.0015" can be combined into one curve for each of the plating solutions. The 0.0003" deposits failed in less than one hour but revealed substantially the same trend as the heavier deposits.

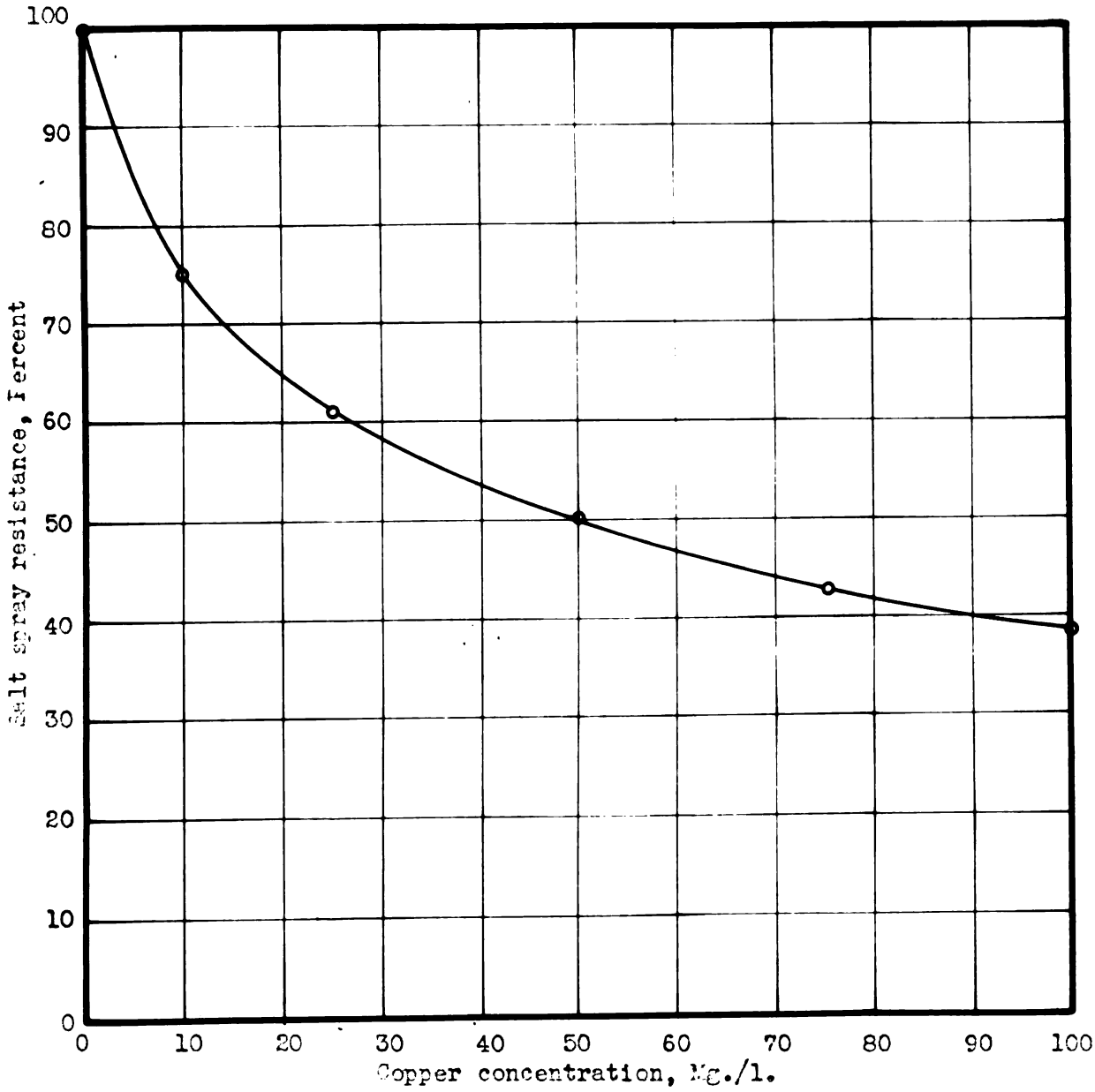


Figure 3

Percent salt spray resistance of 0.001" & 0.0015" nickel coating on steel vs. copper concentration in a Watts type nickel bath of pH 2.2 and 5.2 electrometric

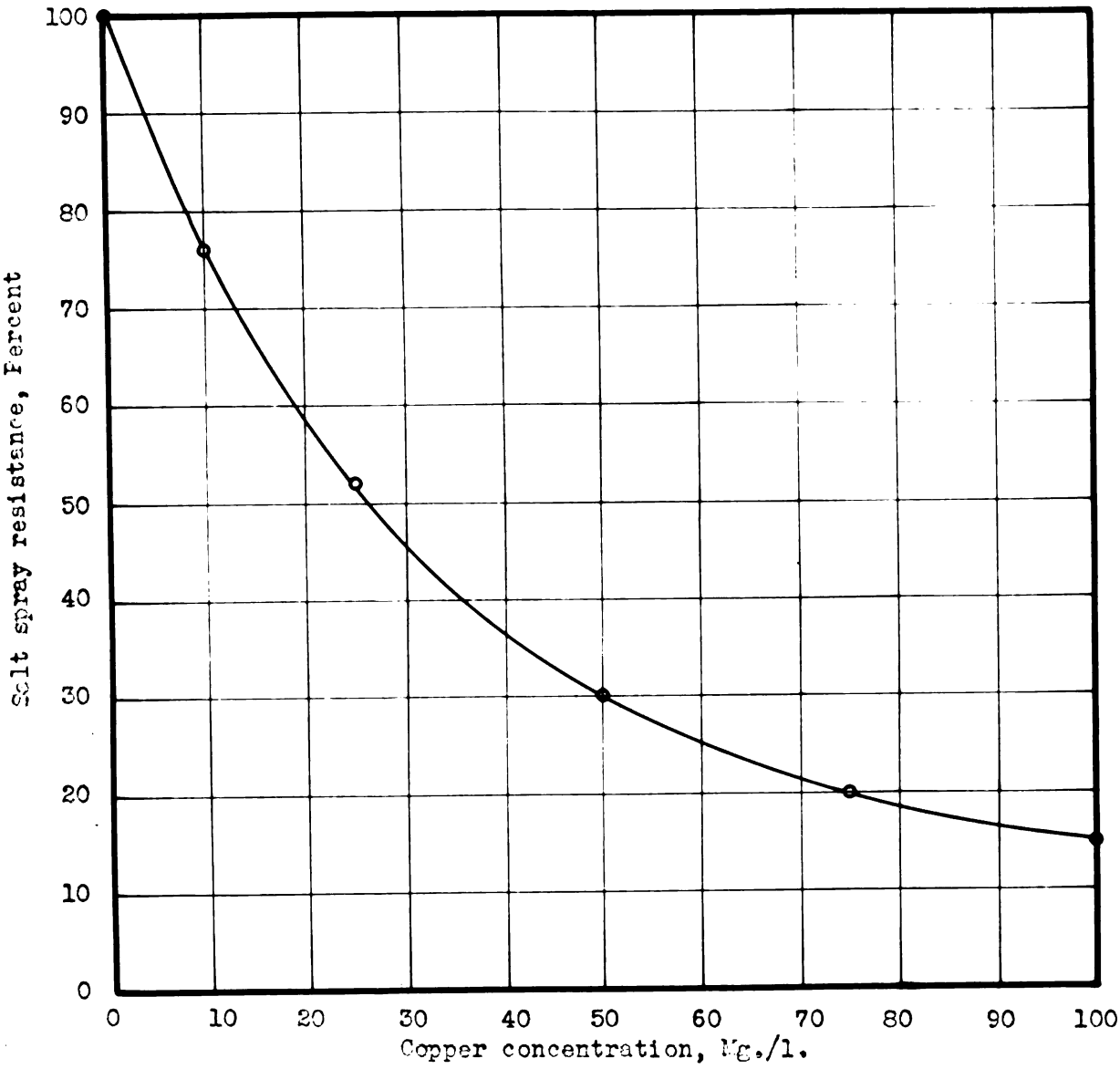


Figure 4

Percent salt spray resistance of 0.001" 0.0015" nickel coating on steel vs. copper concentration in a nickel-cobalt bath of pH 3.75 electrometri

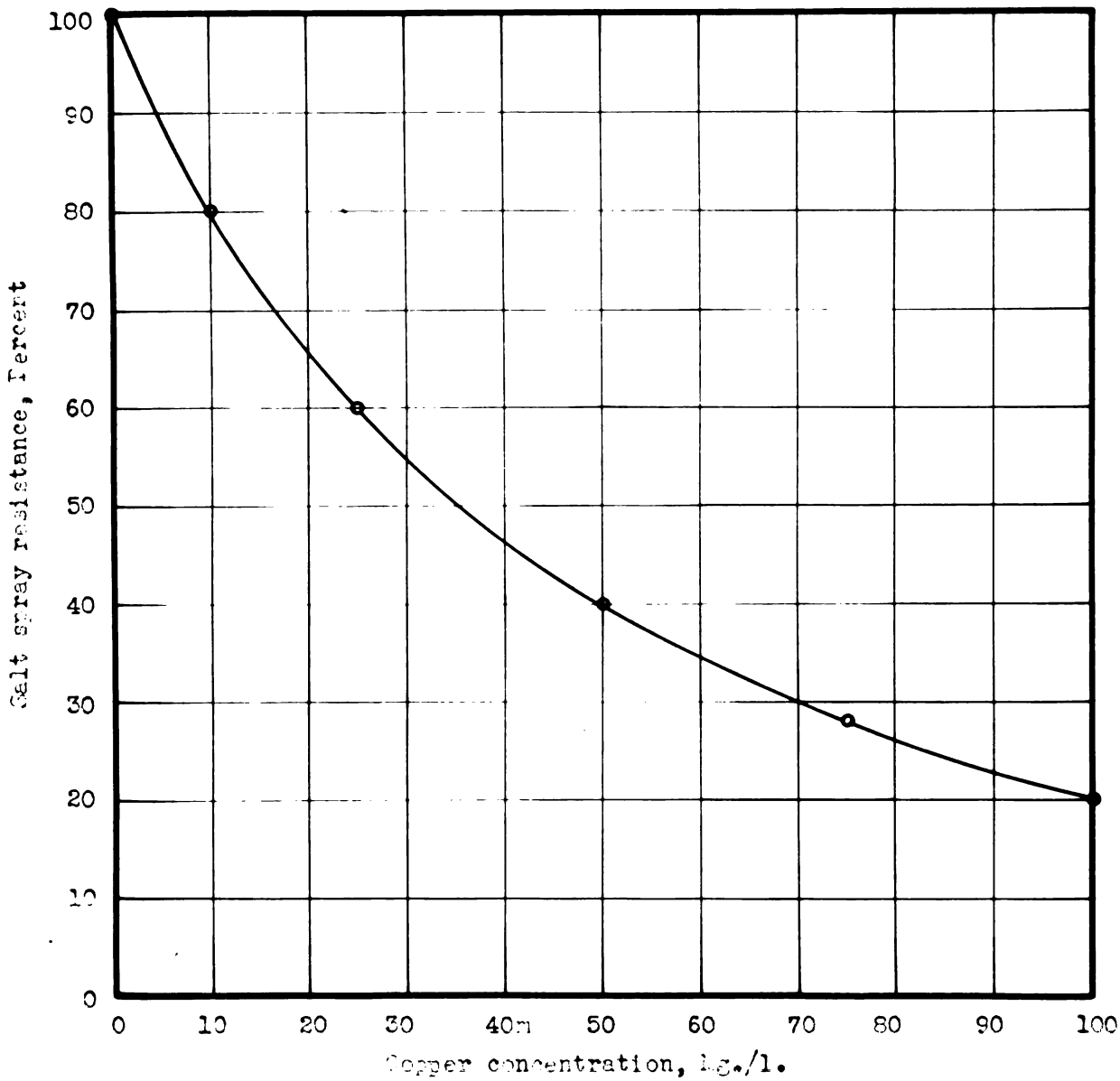


Figure 5

Percent salt spray resistance of 0.001" & 0.0015" nickel coating on steel vs. copper concentration in an organic type bright nickel bath of pH 3.2 electrometric.

The Watts type gray nickel deposits (Figure 3) were identical for both pH's and showed less loss in corrosion resistance, 60%/100 mg./l. of copper, than either of the bright nickel deposits. In the case of the nickel cobalt alloy type bright nickel deposits (Figure 4), and the organic type nickel deposits (Figure 5), these suffered the greatest loss in corrosion resistance, 80-85%/100 mg. of copper per liter.

6. Throwing power and efficiency:

Panels of 0.002" thickness were prepared from the four solutions covering the range of impurity concentrations. The lips of these panels were cut off and microscopic thickness tests were made on each series and were compared with the deposit from the corresponding pure baths.

The lips cut from the panels were carefully cut in half across the short dimension. These pieces were then clamped together, polished, etched with "nital", and examined on a Bausch and Lomb Research Metallographic microscope. The distance from the front edge of the steel to a deposit thickness of nickel of 0.002" was recorded for the four pure baths. On the succeeding panels the same distance was measured from the front edge of the steel, and the thicknesses of the deposits on the top of the lips were recorded.

Differences were reported as the net change in efficiency and throwing power. If no change in gassing is noted the change in thickness may be assumed to be due to a difference in throwing power.

The effects of varying amounts of copper in the four types of nickel solution did not disclose to the eye any change of gassing at the cathode, indicating little if any effect upon the cathode efficiency of that bath. In checking deposit thicknesses under controlled conditions and in pre-determined locations, any changes in thicknesses were therefore deemed the result of changes in the throwing power rather than in the cathode efficiency. Table 7 gives the gain or loss in throwing power and efficiency of the four nickel solutions.

Table 7.

Effect of copper as an impurity on the throwing power and efficiency of nickel plating solutions.*

Cu. conc. Mg./l.	Watts 2.2pH	Watts 5.2pH	Ni-Co 3.75pH	Organic 3.2pH
10	+2.7%	-3.0%	+7.0%	+1.5%
25	-0.8	-5.5	+8.6	+1.0
50	+7.5	-6.5	+7.0	+0.5
75	-2.0	-5.0	+2.5	+4.0
100	-2.7	-11.5	+1.5	+3.0

* Expressed in percentage change from the pure deposit.

The Watts type type of baths showed slight losses in deposit thicknesses indicating adverse effects upon the throwing power with increasing copper contents of the solution. The bright nickel deposits showed no substantial change in thickness at definite points of measurement, with increasing copper content indicating little effect from the presence of copper.

C. The removal of copper from nickel plating solutions:

1. Technique:

The laboratory investigation consisted of two methods for the removal of copper from nickel solutions: ie., the electrolytic method and the high pH treatment using nickel carbonate.

The procedure for the preparation and plating of the panels and apparatus used was as described previously. The current densities employed were 1,2,3,and 4 amperes per square foot, at the operating temperatures of the nickel baths. The agitation rate of four feet per minute of solution past the cathode. Flat lipless cathodes, 2" wide and $3\frac{1}{2}$" long were used.

Initial study indicated that in all cases the copper was rapidly lowered from 100 mg./l. to 25 mg./l., but for smaller concentrations the plating variables must be more carefully determined. Removal studies were therefore started at a copper concentration of 25 mg./l.

For the high pH removal of copper, nickel carbonate was added to a series of nickel solutions containing about 100 mg./l. of copper. The amounts of nickel carbonate added were such as were necessary to give a series of pH values ranging from 2.2 to 6.0. The pH values were taken only after equilibrium was established. The time of standing was a function of rate of agitation and temperature.

2. Evaluation of results:

In all cases studied of the electrolytic removal of copper, the current density of two amperes per square foot was the most effective for the fastest rate. If time is not a factor involved, a current density of one ampere per square foot can be utilized to remove the copper more economically from the standpoint of nickel deposited, at least up to 8000 ampere minutes per gallon. Figures 6-13 shows the rate of removal of copper from the four nickel solutions as removed electrolytically at the specified conditions.

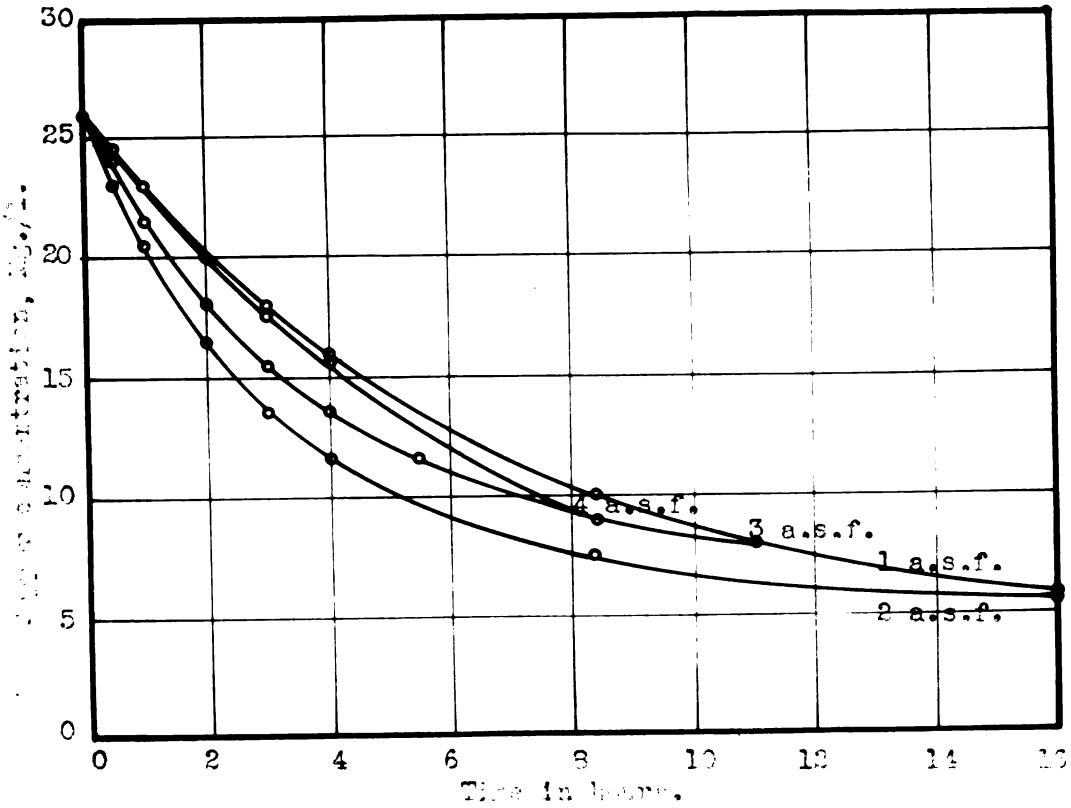
A study of the effect of temperature upon the electrolytic removal of copper was investigated, whereby a run was made on the Watts 5.2 pH bath at room temperature. The rate of removal was less as illustrated in Figures 14-15. An increase in the rate of agitation from four to twenty feet of solution past the cathode per minute

brought about an increased rate of removal of the copper impurity. This is shown in Figures 16-17.

To determine the effectiveness of removing copper by raising the pH, a measured volume of the Watts type gray nickel bath, at a pH of 2.5 and containing 87 mg./l. of copper was treated with nickel carbonate. The pH of the solution was raised slightly, sampled, and analyzed. This procedure was repeated until no further change in pH was observed. This treatment removed all but 13 mg./l. of copper. The solution was allowed to come to equilibrium before pH readings were taken. Figure 18 shows the curve obtained for this high pH treatment for the removal of copper.

In the case of the nickel-cobalt alloy bath and the organic bath, the above procedure was followed. The treatment reduced the copper content of the nickel-cobalt bath from 92 to 31 mg./l. at a pH of 5.85. The organic bath was treated at a pH of 5.95 and the copper content reduced from 92 to 15 mg./l. Figures 19-20.

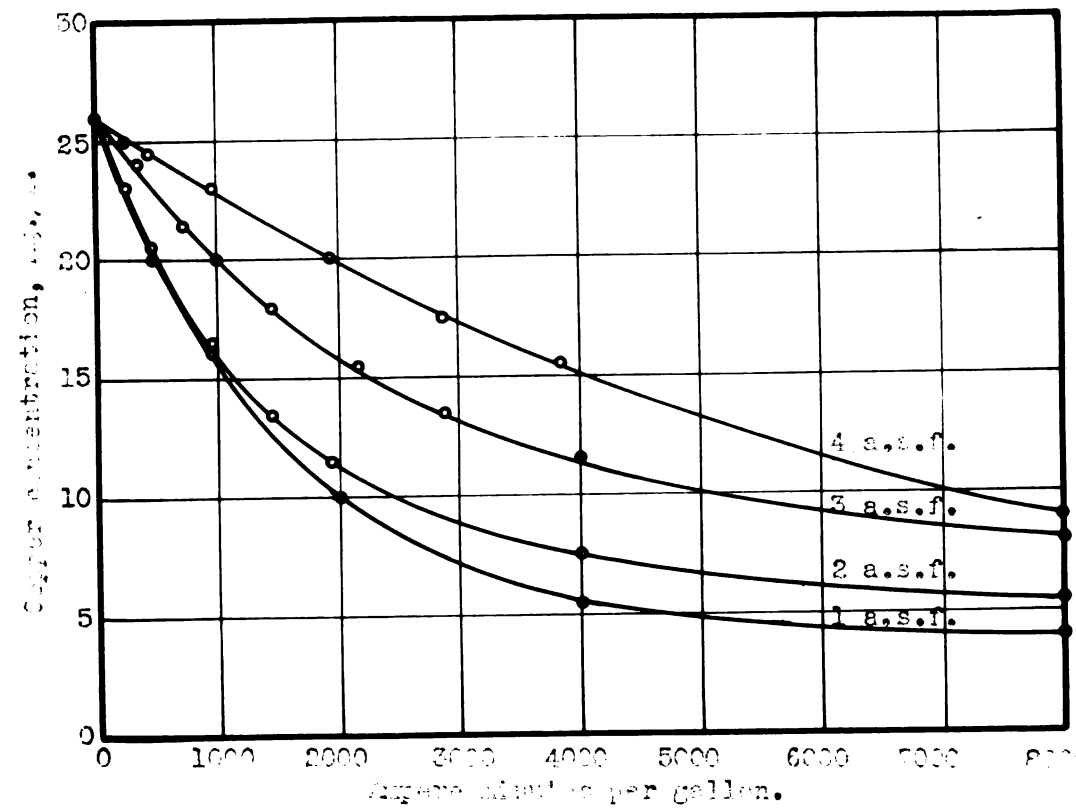
Nickel is also lost in this treatment as in the electrolytic method. In no case was the copper content reduced through high pH treatment to as low values as obtained through electrolysis at low current density.



Time in hours.

Figure 6

Rate of removal of copper by electrolysis at low current density from [illegible] type Nickel bath, 6 volts, 1-ohm rheostat. 50°F. agitation rate of 4 feet/min.



Ampere minutes per gallon.

Figure 7

Rate of removal of copper by electrolysis at low current density from [illegible] type nickel bath, 6 volts, 1-ohm rheostat. 50°F. agitation rate of 4 feet/min.

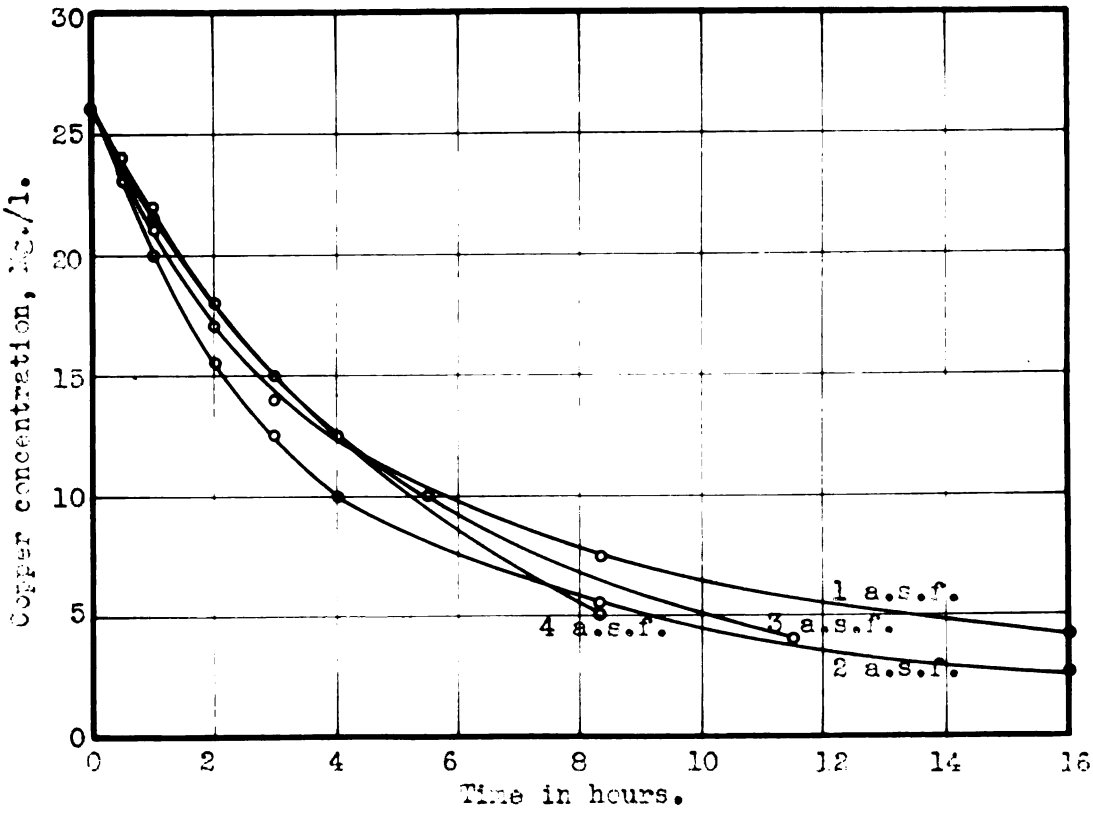


Figure 12

...te of removal of copper by electrolysis at low current densit... from an organic type bright nickel bath of pH 3.2 electrometric. 55°C. Agitation rate of 4 feet/min.

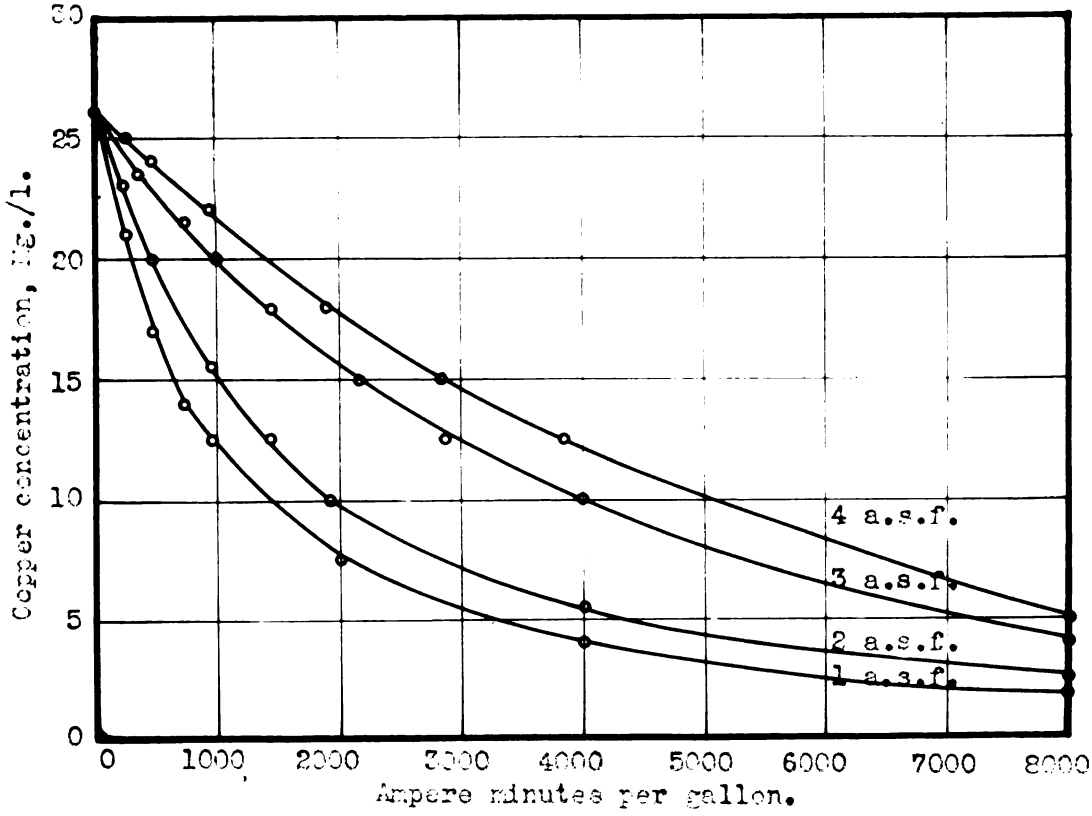


Figure 13

Rate of removal of copper by electrolysis at low current density from an organic type bright nickel bath of pH 3.2 electrometric.

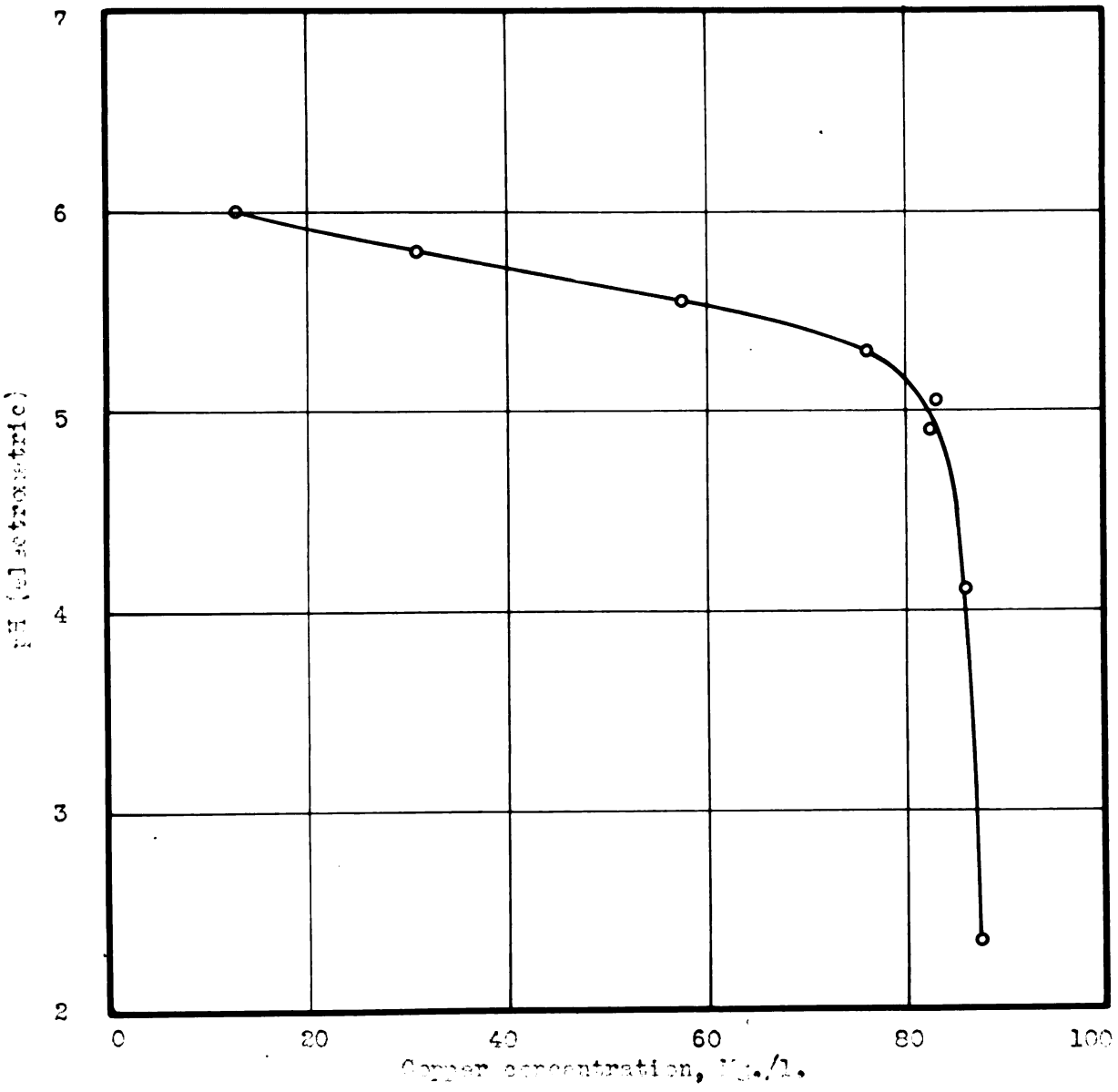


Figure 12

Rate of removal of copper from a Watts type nickel bath by reducing the pH with nickel carbonate.

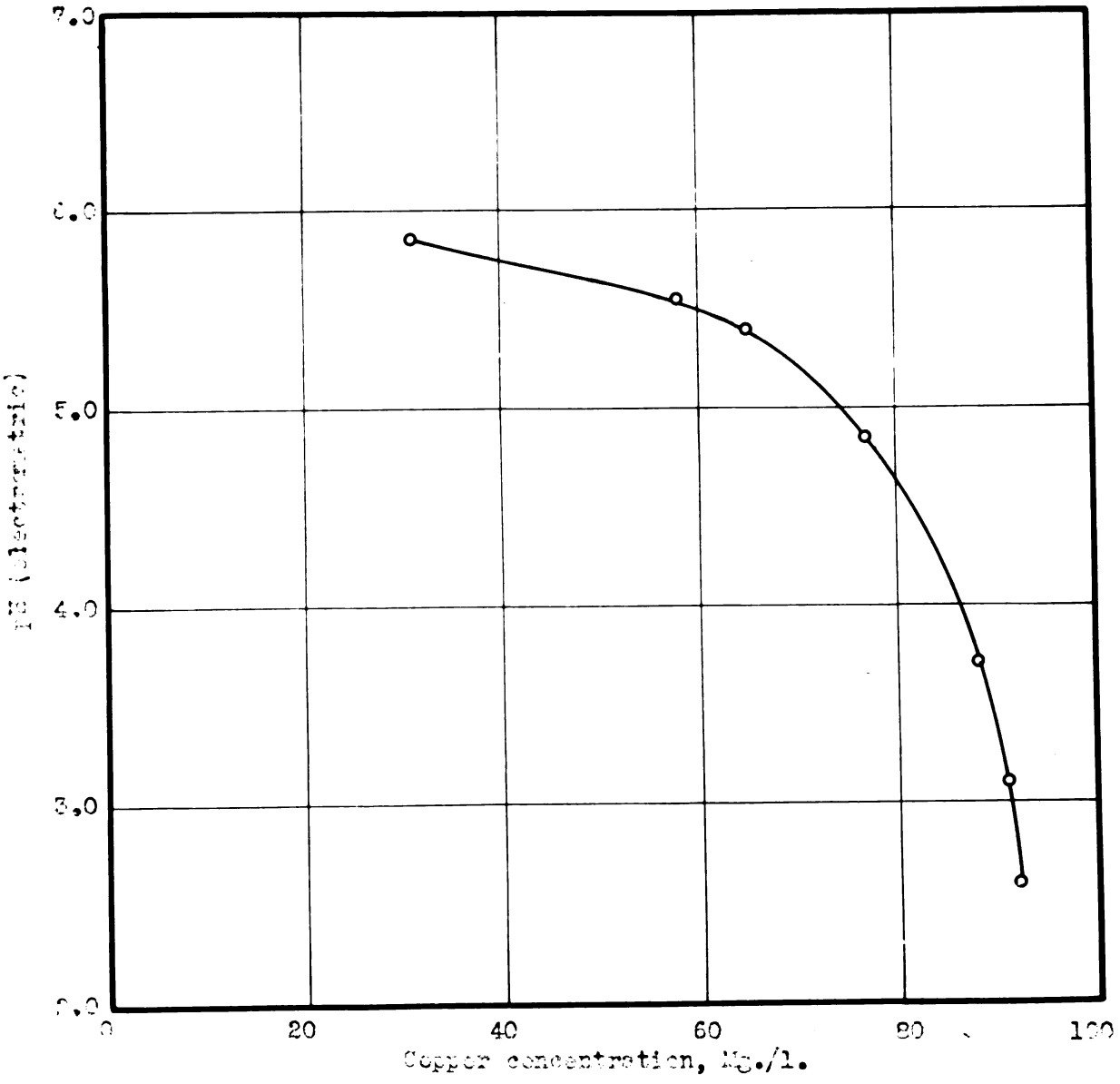


Figure 19

...t of removal of copper from a nickel-cobalt bright nickel bath by raising the pH with nickel carbonate.

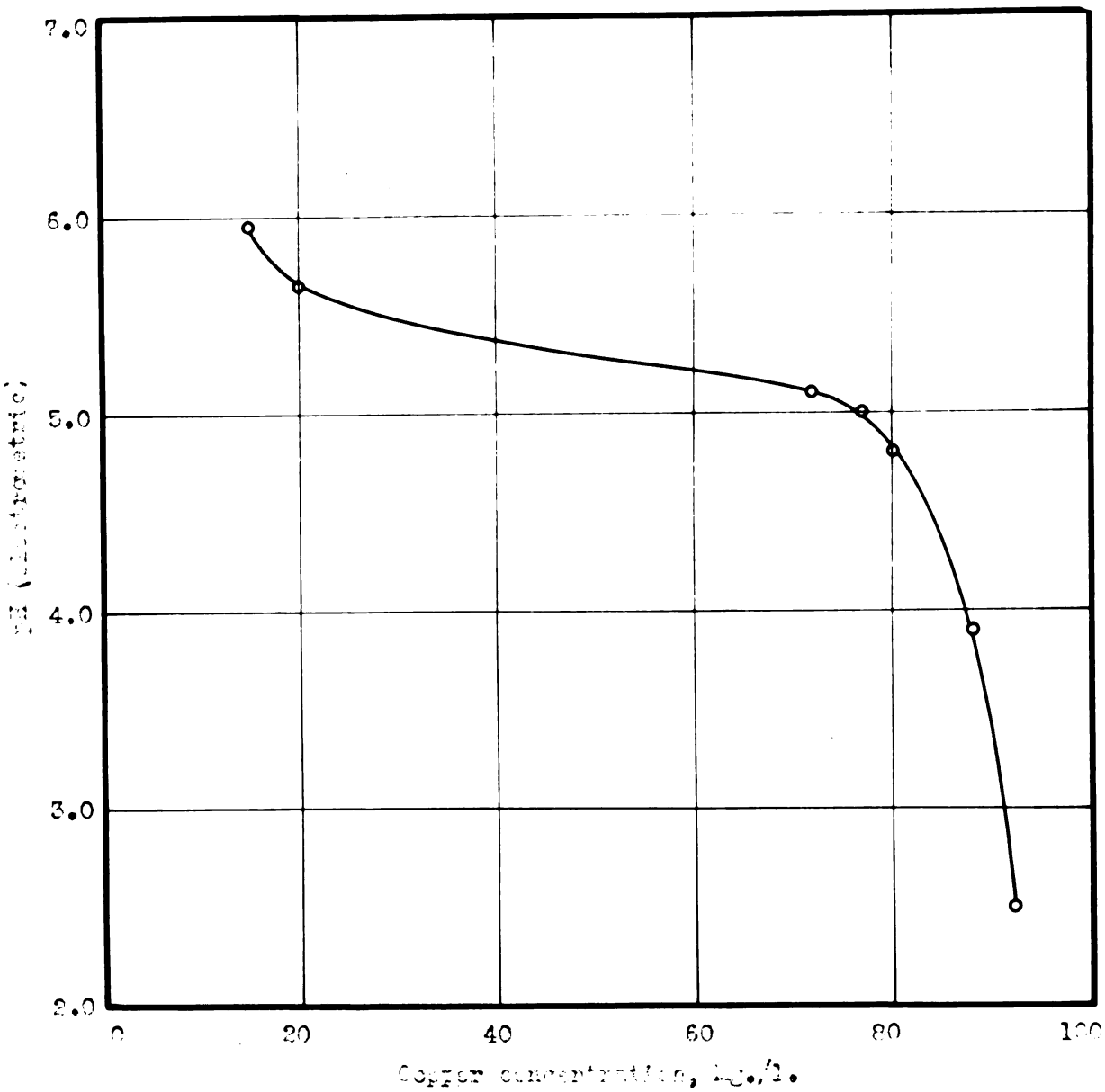


Figure 23

Rate of removal of copper from an organic type bright nickel bath by raising the pH with nickel carbonate.

CONCLUSIONS:

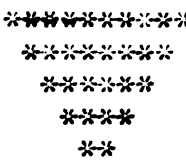
The results of this investigation of the effects and removal of copper from nickel solutions can be summarized as follows:

A. Effects:

1. Appearance - Amounts of copper in excess of 50 mg./l. produces a deposit tending to become rough and dark.
2. Adherence - Adherence of the nickel deposit on steel is not appreciably effected by concentrations of copper up to 100 mg./l.
3. Ductility - A loss in ductility results with an increase in copper concentration of the nickel solution in excess of 10-25 mg./l.
4. Hardness - In most cases the presence of copper as a contaminant increases the hardness of the nickel deposit over that of a pure bath.
5. Corrosion resistance - The corrosion resistance of nickel plated steel is decreased by increasing amounts of copper as an impurity in the plating bath, in concentrations usually considered to be traces.
6. Throwing power and efficiency - The nickel deposits from the Watts type nickel solution shows an adverse effect on the throwing power with increasing copper contents of the solution. The bright nickel deposits show no substantial change indicating little effects from the presence of copper.

B. Removal:

1. Copper can be removed electrolytically, at the optimum current densities of 1-2 amperes per square foot. This method permits reductions to the lowest concentrations.
2. Copper can also be removed by a high pH treatment to a minimum value of 15 mg./l. by means of nickel carbonate.



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