

# THE EFFECT OF LEAD AS AN IMPURITY ON THE PHYSICAL PROPERTIES OF ELECTROLYTIC NICKEL

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Charles James Owen

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## THE EFFECT OF LEAD AS AN INPURITY ON THE PHYSICAL PROPERTIES

OF ELECTROLYTIC NICKEL

by

Charles James Owen

### A THESIS

Submitted to the School of Graduate Studies of Michigan State College of Agriculture and Applied Science in partial fulfillment of the requirements for the degree of

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#### INTRODUCTION

This paper is the eighth in a series of investigations on the effect and removal of impurities in nickel electroplating solutions. Previous work by other investigators has covered copper, zinc, iron and chromium in a similar manner.

In this investigation lead, as the impurity, was added as lead chloride to each of the four representative nickel baths: Watts type pH 2.2 and 5.2, organic type pH 3.2, and nickel-cobalt alloy type pH 3.75. The impurity was added to each nickel bath in the concentration gradients: 0, 2.5, 5, 10, and 15 mg/l for the Watts pH 2.2; 0, 2.5, 5, 10, and 20 mg/l in the Watts pH 5.2; 0, 2.5, 5, 10, and 25 mg/l for the organic; and 0, 2.5, 5, 10, 20, and 35 mg/l in the nickel-cobalt bath. These gradients were interpolated from the equilibrium solubility tests where an excess of lead chloride was added to samples of each bath, allowed to stand at room temperature with periodic agitation, and the samples analyzed for lead content. The samples were then heated to  $60^{\circ}$ C. (140°F.), maintained at this temperature for 72 hours, and again analyzed. The room temperature solubility in the Watts pH 2.2 bath was 17 mg/l, in the Watts pH 5.2 bath 22 mg/l, in the organic bath 25.5 mg/l, and in the nickel-cobalt bath 34.5 mg/l. The solubility in each at the higher temperature was 55.5, 35, 43.3, and 64.5, respectively.

The deposits obtained, using the standard procedures outlined by Ewing, Rominski and King<sup>(1)</sup>, were analyzed qualitatively for appearance, adhesion, ductility, salt spray (fog) corrosion resistance and throwing power. These properties of the deposits prepared from solutions containing varying quantities of lead were determined by comparison with those prepared from solutions free of lead. Since trends were the objective instead of absolute data, deviations were reported as percent change from the properties of the pure deposit. Low current density electrolysis at 1, 3, 5 and 9 asf (0.1, 0.3, 0.5 and 1.0 amp/dm<sup>2</sup>) and high pH precipitation were also investigated as possible methods of lead removal.

According to a previous publication by Ewing and Gordon<sup>(2)</sup>, covering twenty references on the effect and removal of lead as an impurity in nickel solutions, the presence of lead resulted in the formation of bright, brittle deposits. Dark, streaked appearance and non-adhesion were also attributed to lead contamination. The solubility of lead was said to be negligible, dependent on pH, and increasing with temperature in chloride-containing baths. Removal by electrolysis at 2 to 3 asf (0.2 to 0.3 amps/dm<sup>2</sup>), a pH of 2, and a fairly high temperature was recommended. Removal by low pH precipitation was also suggested in a reference from the same publication which stated that, to remove lead from nickel baths, they should be strongly acidified with sulfuric acid, heated, cooled and filtered. In a later publication, Piontelli<sup>(3)</sup> advocated removal of metallic impurities, including lead, by adding Raney nickel to the bath, stirring for an hour at room temperature and filtering. Case<sup>(4)</sup> suggested electrolytic removal of lead at 2 to 4 asf (0.2 to 0.4 amps/dm<sup>2</sup>), agitation being an important factor in the rate of removal.

#### EXPERIMENTAL

# A. Preparation of Panels and Evaluation of Physical Properties

The pure solutions of each bath were prepared by standard methods described by Ewing, Rominski and  $\operatorname{King}^{(1)}$  in a previous publication. All testing and evaluation methods may be found in the same paper. Some changes in the standard procedures were found necessary and are outlined by Ewing, Brouwer and Werner<sup>(8)</sup>. The bath compositions are illustrated in Table I.

The colorimetric method of analysis used to determine the lead content in the various samples was suggested in a publication by Foulke, Meyer and  $Case^{(6)}$ , developed by the American Electroplaters Society, Research Froject No. 2 (7), and modified by the Engineering Division of Chrysler Corporation. The method involved the determination of a calibration curve, as shown in Figure 1, from

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		t a	Nickel- (18%) A	Cobalt Lloy	Organ	le
Component	g/1	oz/gal	g/1	oz/gal	g/1	oz/gal
Nickel Sulfate	240	32	240	32	262.5	35
Nickel Chloride	45	Q	45	Q	60	œ
Boric Acid	30	4	8	4	34	4.5
Nickel Formate	8	8	45	Q	8	ł
Cobalt Sulfate	ł	8	15	N	2 8 8	ţ
Armonium Sulfate	9 8 8	8 8 9	2•5 2	0.33	ł	ţ
Formaldehyde	i	8	2°2	0.33	a a	ł
Nickel Benzene Disulfonate		8	ł	8	7.5	1.0
Tri ami notolyldiphenylme thane	•	•			0•14	<b>1/1</b>
Temperature	50±2°C.	122 <mark>+</mark> 4 <sup>0</sup> F.	55 <u>+</u> 2°C .+	131 <b>-</b> 4°F.	55±2°C.	131 <b>+4<sup>0</sup>F.</b>
Current Density	4.3 amps/dm <sup>2</sup>	40 asf	4.3 amps/dm	a 40 asf	4.3 amps/dm <sup>a</sup>	40 asf
pH, electrometric	ਬ ਨਾ• ਨਾ	nd 5.2	З	75	3.2	

\*Changed to 60°C. in later publication by Ewing, Erouwer and Merner (5)



nickel solutions containing known amounts of lead. The samples were treated with a carbon tetrachloride solution of dithizone in the presence of potassium cyanide to separate lead from interfering ions by extraction. After removal of the excess dithizone with alkaline potassium cyanide, the transmittancy of the solution was measured with a Klett-Summerson colorimeter using a green, No. 54 filter and a 0.5 inch diameter cell. Scale reading was plotted against known lead content to give the calibration curve. Unknown samples from the removal baths were subjected to the same analysis, the transmittancy reading obtained with the colorimeter, and the lead content interpolated from the calibration curve.

The cathodes consisted of sheet steel strips, 2 by 5 inches (5.1 by 12.5 cm), cut from S.A.E. 1010 cold-rolled tin-can stock of 0.01 inch thickness and bent  $90^{\circ}$  at a distance 1.25 inches from one end. Cathodes for the removal baths were flat. The cathode surface defects, such as rolling seams, were not removed before use.

# B. Effects on Physical Properties

1. <u>Appearance</u> - The evaluation of surface appearance, described by Ewing, Rominski and King<sup>(1)</sup> and furthur supplemented by Ewing, Brouwer, Clark, Owen, Rominski and Werner<sup>(5)</sup>, was completed on the Watts pH 2.2 and 5.2 panels with the use of the Eastman Gray Scale. The lightest shade of gray on this scale was designated numerically as (1), the next darker shade as (2), and successively darker shades were assigned corresponding numbers. The method was essentially color comparison of the sample deposit to the scale. The evaluation of the surface appearance of the brighter deposits was more difficult. The classification range of dull to mirror bright was found to be insufficient for describing the appearance of the organic and nickel-cobalt deposits. However, in the absence of an accepted standard, all panels were classified as mirror bright and the increase in brightness with increasing lead concentration was attributed to an observed decrease in visible base motal defects. Table II summarizes the appearance

# Table II. The Effect of Lead on the Appearance of Nickel

Deposits

_				
Lead Conc. mg/1	Watts pH 2.2	Watts pH 5.2	Organic pH 3.2	Nickel- Cobalt pH 3.75
0	1-2*	1	Mirror Brt.	Mirror Brt.
2.5	1-2	1	<b>11</b> 11	n n
5	<b>1-</b> 2	1-2	n n	M (1
10	1-2	1-2		u n
15	1-2		n n	
20		1-2		#1 11
25			n n	
35				11 18

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Bath Types

evaluation of the vertical panel sections deposited from the four baths containing lead as an impurity at a current density of 40 asf (4.3  $\text{amps/dm}^2$ ).

The panels obtained from the Watts pH 2.2 bath showed a slight brightening effect in the low current density area (the bend in the panel) at the 5 mg/l lead concentration. This area increased in size and brightness with increasing lead concentration.

The Watts pH 5.2 panels showed a similar brightening effect in the low current density area, beginning at the 2.5 mg/l lead concentration and increasing in brightness and area with increasing lead concentration.

An increase in lead impurity in the organic bath resulted in a decrease in visible base metal defects on the panels and improved control over the brightener in the bath. This decrease in visible base metal defects gave a possible illusion of a brighter plate or increased reflectivity, depending on the definition of brightness.

The nickel-cobalt panels exhibited a similar effect in covering base metal defects and a similar ease of brightener control at the higher lead concentrations. In addition there was a blackening in the very low current density area on the back, center of the panels, beginning with the 20 mg/l lead concentration and becoming more pronounced on the 35 mg/l panels.

2. <u>Adhesion</u> - Representative samples of the horizontal panel sections obtained by deposition from the four nickel baths were cut from the bent cathodes and subjected to the qualitative test approved for this determination. This test consisted of bending each horizontal section longitudinally under pressure, upper side outward, and examining the bent edge under a <sup>4</sup>0 power microscope, evidence of flaking being considered indicative of poor adhesion. The results showed good adhesion of the nickel plate to the steel base metal in all cases. Flaking of the deposit was not observed on any panel tested.

3. Ductility - A 0.001 inch strip-deposit of nickel on the oxidized nickel surface of the panels was prepared from the pure solutions of the four nickel baths and from each bath at the successive lead concentrations, The

test consisted of bending and creasing two or three sections of each stripdeposit repeatedly in both directions, using the same crease line, until failure occurred. The extent of bending and creasing the strip-deposits obtained from the baths containing lead was compared with that of the strip-deposits from the pure solutions. This comparison was furthur classified according to percentage increase or decrease in the number of bends and creases of the strip-deposits from the impure solutions as compared with those from the pure baths. If a deposit failed after bending and creasing five times while the deposit from the pure bath failed after four, the former deposit was considered 25% more ductile.

The Watts pH 2.2 strip-deposits showed a maximum increase in ductility of about 1% at the 5 mg/l lead concentration, followed by a decrease to a negative 10% at the higher impurity concentrations, the upper limit being 15 mg/l. The Watts pH 5.2 strip-deposits showed a very definite increase in ductility, beginning with a 5% increase at the 5 mg/l lead concentration and increasing to 50% at the 20 mg/l concentration. The organic strip-deposits showed a slight decrease in ductility of about 8% at the 15 and 25 mg/l lead concentrations. The ductility of the nickel-cobalt strip-deposits was a constant for all lead concentrations.

4. <u>Salt Spray</u> (Fog) <u>Corrosion Resistance</u> - The corrosion resistance of the nickel deposits was determined in accordance with the A.S.T.M. Tentative Method of Salt Spray (Fog) Testing Specification B117-49T. Fanels No. 11 (before breakdown), No. 9 (at breakdown), and No. 7 (after breakdown) were used as a basis for evaluating the breakdown time of the deposits subjected to salt spray corrosion. The limited capacity of the salt spray apparatus necessitated the separation of the deposits into groups, such as the thin deposits (0.0003 inch) for the four baths and the heavy deposits (0.001 and 0.0015 inch) for each bath. In all five groupings the pure deposits for each were included in their representative group of panels and were used as a basis for determining the percentage deviation in corrosion resistance of the deposits containing

increasing lead impurity concentration, as discussed by Ewing, Rominski and King<sup>(1)</sup>. The approved procedure outlined in this publication was followed for checking panel breakdown. Comparison was based on the relative time exposed to salt spray corrosion before failure occurred. This was furthur classified according to percentage increase or decrease in corrosion resistance, based on the relative time before breakdown of the deposits from the impure baths as compared to the time before breakdown of the pure deposits. Surface film on the panels was removed by cleaning with magnesium oxide paste before testing.

The results of exposure of the Watts pH 2.2 panels to salt spray corrosion are shown in Figure 2. The 0.0003-inch deposits were apparently unaffected while the heavier deposits showed a slight increase in corrosion resistance with increasing lead concentration.

The thin Watts pH 5.2 panels showed a maximum decrease in corrosion resistance of a negative 16% at the 5 mg/l lead concentration. The heavier 0.001-inch deposits showed no deviation with increasing lead concentration and the 0.0015inch deposits exhibited a slight increase in corrosion resistance with increasing impurity to a maximum of 10% at the 5 mg/l lead concentration. Figure 3 illustrates the effect of lead on the corrosion resistance of the Watts pH 5.2 deposits.

The corrosion resistance of the thin organic deposits, shown in Figure 4, decreased to a negative 10% at the 15 mg/l lead concentration and the heavier deposits increased slightly in corrosion resistance.

The curves for the nickel-cobalt panels are shown in Figure 5. The thin deposits showed no deviation with increasing lead concentration while the heavier panels showed a slight increase in corrosion resistance as the impurity in the bath was increased.

5. <u>Throwing Power</u> - The throwing power determination consisted of a microscopic measurement of plate thickness of the 0.002-inch bent cathode horizontal sections. The cross-section of the upper plate thickness only was examined at a magnification of 410X under vertical illumination, using











representative sample sections from the four baths. Comparison of these measurements with those of the samples deposited from the pure solutions gave a measure of the throwing power. It was assumed that, since no gassing was observed at the cathode in any bath, any difference in deposit thickness was due to a change in throwing power and not current efficiency.

The results of the investigation of throwing power are summarized in Table III and Figure 6. The Watts pH 2.2 and 5.2 deposits showed a slight decrease in throwing power with increasing lead concentration while the organic panels showed a slight increase. The throwing power of the nickel-cobalt bath was erratic, exhibiting an initial increase of 8% at the 2.5 mg/l concentration followed by a decrease to a negative 3% at the 10 mg/l concentration and an increase to over 12% at the 35 mg/l concentration.

The cross-sections of the electrodeposited panels for the throwing power test were used to obtain a series of photomicrographs. These photomicrographs were taken to illustrate each deposit cross-section, possible evidence of porosity, and the lamellar deposition of organic and nickel-cobalt deposits.

Figures 7 through 13 illustrate possible formation of pores in organic nickel deposits. Of these photomicrographs all except Figures 8 and 9 are 1000X cross-sectional views of a 0.002-inch deposit from an organic bath containing 25 mg/l of lead as an impurity. The base metal may be found at the bottom of the photomicrograph in Figures 7, 10 and 11 and at the right in Figures 12 and 13. Figure 8 is a similar view of a deposition from a pure organic bath. The high current density tip of the bent cathode base metal shows in the lower left corner. The supposed pore in this case seems to start from a crack in the deposit. Figure 9 is a 100X photomicrograph of the organic deposit used to obtain Figures 7 and 10 through 13. Cracks, probably caused by previous sectioning with metal shears, are visible with the pores.

Figure 14 is a 50X view of the high current density tip of a bent cathode from an organic bath containing 25 mg/l lead.

Figure 15 is a 1000X view of the effect of occluded material on the



		Abas		
Lead Conc. mg/l	Watts pH 2.2	Watts Organic pH 5.2 pH 3.2		Nickel- Cobalt pH 3.75
		Percent Ch	ange	
0	0	0	0	0
2•5	-1.4	0.5	-1.2	8.0
5	-0.9	-1.2	0	6.8
10	-3.1	-4.2		-3.0
15	-5.9		1.7	
20		<b>-</b> 5 <b>.2</b>		0
25		an align	2.3	
35				12.2

Table III. The Effect of Lead on the Throwing Power of Nickel Solutions

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Bath Types



Figure 7

Organic Deposit

1000X



Figure 8

1000X

Figure 9 Organic Deposit 100X







Figure 11

Organic Deposit

1000X



Figure 14 Organic Deposit

Figure 12

1000%

50X

deposition from an organic bath containing 5 mg/l lead. All nine photomicrographs of organic deposits illustrate the typical lamellar deposit obtained from this type bath.

A 1000X cross-section of a pure Watts pH 5.2 deposit is shown in Figure 16 with the base metal at the right of the photomicrograph. The etch, a mixture of 50% nitric acid and 50% glacial acetic acid, has differentially attacked the nickel, creating a wormy appearance.

The photomicrographs in Figures 17 and 18 are 500X and 1000X views of a deposit from a pure nickel-cobalt solution. The lamellar deposition is clearly visible but not as profuse when compared with the nickel deposits from the organic baths. This lamellar effect is more noticeable near the surface and in the thicker nickel-cobalt deposits.

As a check on the possibility that the so-called porosity of the organic deposits illustrated in Figures 7 through 13 was or was not a separation under stress, the panel section used for these photomicrographs was reground, polished and removed from the clamp. An examination of the unetched surface revealed poor definition and continuous surface scratches at the higher magnifications necessary for microscopic detection of the porosity. Nickel flow, resulting from the grinding and polishing operation, obscured the microstructure. A relatively light etch removed these obstacles. The original porous surface was no longer evident and few possible pores could be found. After careful examination of both cross-sectional and surface views, a strong etch was applied and the pores were examined again. Three such structures are shown in cross-section in Figures 19, 20 and 21. The surface views of these possible pores are illustrated in Figures 22 and 23. The pore in Figure 19 is at the left in Figure 22. The pore in Figure 21 is visible only as a semi-circular cavity on the upper edge in Figure 23. The surface in Figure 23 also shows a shallow, scooped pit as a result of differential etching.

Since only the sectioned lip of the 25 mg/l organic panel in the entire set resulted in appreciable porous cross-section, it is possible that this



Figure 16 Watts pH 5.2 Deposit

100**0X** 



Figure 18

Nickel-Cobalt Deposit

1000X



Figure 20

Organic Deposit

1000X



Figure 20 Organic Deposit

1000X





400X







panel was set higher in the clamp and not ground as extensively. Cracks and separations in the deposit resulting from sectioning with heavy metal shears and extending inward from the cross-sectional surface only a few thousandths of an inch would not have been removed by grinding. Etching acid, seeping into these cracks, could not be completely washed out and would erode the walls. An examination of the depth of these so-called pores with the fine microscope adjustment revealed that the blackness was not trapped polishing compound, which would add weight to the pore theory, but was due to a lack of reflected light. As a result of this examination, the etching and enlarging of cracks remained a possibility. The heavy etch and subsequent examination over a period of time showed that the cavities were furthur etched by retained acid long after washing and drying the specimen. Two of the three possible pores viewed in cross-section were definitely proven to be cracks by the surface examination. The so-called pores are more probably cracks that have been attacked by minute quantities of etching acid retained in them.

# C. Removal of Lead

1. <u>Procedure</u> - The removal of lead was attempted by two methods: low current density electrolysis and high pH precipitation. Both are outlined by Ewing, Rominski and  $\operatorname{King}^{(1)}$  in a previous publication. Removal at the operating current density of 40 asf (4.3 amps/dm<sup>2</sup>) was also undertaken to obtain the data necessary for maintaining the baths at the specific lead concentrations used. For the operating current density depletion determinations, a liter of each bath type containing the maximum concentration of lead chloride was operated continuously, samples were taken at various time intervals and analyzed for lead content, and the analysis results were plotted against time. Additions of lead chloride solution (5 mg/ml concentration) were made to the baths used for preparing the test panels after ten percent of the lead concentration at each gradient had been removed during operation, as determined from these depletion rate curves.

Low current density electrolyses were conducted at current densities of 1, 3, 5, and 9 asf (0.1, 0.3, 0.55, and 1 amp/dm<sup>2</sup>) for each of the four bath types to clarify the inconsistency of the recommended optimum for lead removal. Samples were taken from the baths according to the time schedule shown in Table IV. The baths were run at operating temperature with an agitation rate of 4 ft./min. past the cathode.

Removal of lead by high pH precipitation was conducted according to the standard method, additions of nickel carbonate being used to raise the pH. Samples were taken periodically from each of the four representative baths containing varying quantities of lead in solution and, initially, not necessarily the maximum possible.

2. Evaluation of <u>Results</u> - The depletion rate curves obtained from the operating current density removal baths are shown in Figures 24 through 27.

The low current density removal of lead from the Watts pH 2.2 bath at current densities of 1, 3, 5, and 9 asf is illustrated by the curves in Figure 28. Assuming that a lead concentration of about 2.5 mg/l is the desired optimum, this concentration is reached in the 1 asf bath after about 6 hours (or about 1.2 amp. hrs./gal.), in the 3 asf bath after 10 hours (or 6 amp. hrs./gal.), in the 5 asf bath after 9 hours (or 9 amp. hrs./gal.), and in the 9 asf bath after 9 hours (or 17 amp. hrs./gal.). The curves in Figure 28 indicate that time is almost a constant for all four baths and since the lead:nickel removal ratio is greatest for the 1 asf bath, it is the most efficient for lead removal. Thus for the Watts pH 2.2 bath the 1 asf current density is the best to use for removal of lead from the bath. A single graph of mg/l of lead remaining in the bath versus time for all samples from the four different current density baths gave the equivalent of a single curve, which substantiated the inference that increased current density did not appreciably increase the rate of removal. If time is desired for any one removal, it may be obtained from Table IV.

Figure 29 shows the curves for the low current density removal of lead

	Sample No.	Amp. Hrs. per Gal.	l asi	E	3 as	sf	5 a.	3f	9 a <b>s</b>	f
•	0	0	0		0		0		0	
	1	0.5	2.5 h	rs.	0.83 1	n <b>rs.</b>	0.5	hrs.	0.28	hrs.
	2	1	5	N	1.67	n	1	hr.	0.57	11
	3	2	10	N	3•33	11	2	hrs.	1.09	11
	4	4	20	n	6.67	n	4	n	2.17	Ħ
	5	6	30	n	10	n	6	H	3.33	H
	6	9	45	n	15	H	9	n	5	n
	7	12	60	n	20	n	12	n	6.67	Ħ
	8	16	80	R	26.67	n	16	n	8.88	n
	9	20	100	11	33.33	n	20	n	11.1	H

Removal Baths

Table IV. Time Schedule for Sampling Low Current Density

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BATH UNDER OPERATING CONDITIONS



from the Watts pH 5.2 bath. The 1 asf curve shows that the optimum 2.5 mg/l lead concentration was reached after 10 hours (or 2 amp. hrs./gal.), the 3 asf after 10 hours (or 6 amp. hrs./gal.), the 5 asf after 12 hours (or 12 amp. hrs./gal.) and the 9 asf after 9 hours (or 16 amp. hrs./gal.). Again as in the Watts pH 2.2 bath, time is a constant for all four baths and therefore the 1 asf current density could be considered the best to use, the nickel saving being an additional factor qualifying this choice.

The curves in Figure 30 illustrate the low current density removal of lead from the organic type solution. The optimum lead concentration was reached in the 1 asf bath in 9 hours (or 1.8 amp. hrs./gal.), in the 3 asf bath in 10 hours (or 6 amp. hrs./gal.), in the 5 asf bath in 8 hours (or 8 amp. hrs./gal.), and in the 9 asf bath in about 12 hours (or over 20 amp. hrs./gal.). The 1 asf current density is recommended for the removal of lead from the organic bath because it not only removes lead to the desired concentration in less time and with greater efficiency but also with as little as one twelfth the amount of nickel expended.

The low current density removal from the nickel-cobalt alloy type bath is illustrated in Figure 31. The optimum lead concentration is reached in the 1 asf bath after 20 hours (or 4 amp. hrs./gal.), in the 3 asf bath after 20 hours (or 12 amp. hrs./gal.), in the 5 asf bath after 16 hours (or 16 amp. hrs./gal.), and in the 9 asf bath after 11 hours (or 19 amp. hrs./gal.). The question of which current density to use for removal becomes a problem of whether time or nickel is the least expendable. The 1 asf current density appears more practical when efficiency or the lead:nickel removal ratio is considered.

The high pH precipitation of lead from the four representative nickel baths is illustrated by the curves in Figure 32. The curve for the Watts pH 2.2 bath illustrates the removal of lead from this solution to a concentration of about 2 mg/l at a pH of about 5.8, above which nickel is also removed. The Watts pH 5.2 curve shows that the lead concentration follows the curve







${}^{\bullet}$	3	ASF	BATH
Θ	5	ASF	BATH
$\mathbf{a}$	•	ACE	OATH

SF. BATH





established for the Watts pH 2.2 bath and removal above a pH of 5.8 is shown to be more difficult. The curve for the organic type bath illustrates the removal of lead with increasing pH to a concentration of about 2 mg/l at a pH of 5.4. The curve for the nickel-cobalt bath shows that lead is difficult to remove below a concentration of about 7 mg/l at a pH of 5.7, the apparent maximum solubility of nickel carbonate in the bath. The pH limit reached in all cases was the highest possible and no visual loss of nickel was noted except possibly in the two Watts baths.

#### SUMMARY

A summary of the results of this investigation on the effect of lead as an impurity in nickel solutions on the physical properties of the nickel deposits and its removal from solution shows:

A general brightening effect of the deposits from the four baths with increasing lead concentration was noted. This was more noticeable on the Watts pH 2.2 and 5.2 panels in the low current density areas. In the absence of an accepted standard, the brightening of the organic and nickel-cobalt panels was attributed to a decrease in visible base metal defects with increasing lead concentration. In the latter two baths there was also improved control over the brightener with increasing impurity.

Adhesion was unaffected by increasing the lead concentration. In all cases the adhesion of the deposit to the base metal was good and no flaking of the deposit was observed.

The ductility test on the strip-deposits showed no general effect on ductility of the nickel with the exception of the Watts pH 5.2 deposits, where a definite increase in ductility with increasing lead concentration was observed.

The salt spray corrosion resistance of the 0.0003 inch, 0.001 inch, and 0.0015 inch panels from the four representative baths was only slightly affected by increasing lead concentration. No deviation was greater than 10%, with the exception of the 0.0003 inch Watts pH 5.2 panels, where a 15% decrease in corrosion resistance was noted at the 5 mg/l concentration. The thinner 0.0003 inch deposits, in general, exhibited a slight decrease in corrosion resistance or no effect was noted. The 0.001 inch deposits were either unaffected by increasing lead concentration in the bath or a slight increase in corrosion resistance resulted. The 0.0015 inch deposits showed a slight increase in corrosion resistance with increasing lead concentration.

Little effect was observed on the throwing power in the baths with the

exception of the nickel-cobalt bath where the throwing power proved erratic as the lead concentration was increased. The throwing power of the Watts baths decreased slightly and the organic increased slightly with increasing impurity.

Removal of lead by low current density electrolysis was generally more effective at the l asf current density. In all cases the efficiency, or the ratio of the amount of lead removed to the amount of nickel expended in removal, definitely favored the lower current density, as illustrated by the graphs. The nickel saving at the l asf current density was an additional factor in the choice. Lead was removed to the optimum 2.5 mg/l concentration in all baths, with the exception of the nickel-cobalt, in a constant time period, regardless of the current density used, eliminating time as a factor for consideration. In the nickel-cobalt bath the variation in time for optimum removal with current density was more apparent.

High pH precipitation was also effective in removing lead from all baths, with the exception of the nickel-cobalt, down to or below the optimum of about 2.5 mg/l before any appreciable amount of nickel was also removed, which occurred above a pH of approximately 5.8. The lead content of the nickel-cobalt bath could not be removed below a concentration of 7 mg/l by high pH precipitation.

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