COLZINC® ACF-II TRAINING MANUAL

Alkaline Non-Cyanide Zinc Plating Process

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INTRODUCTION

This training manual is meant as a service tool for evaluating and troubleshooting COLZINC® ACF-II plating baths and related processes. These simple techniques require only standard laboratory plating equipment such as a Hull cell, Duplicell, rectifier, thickness tester, lab scale and burettes for wet lab analyses.

The main focus of this manual will be the proper use of the Hull cell. Arguably the single most important service tool, the Hull cell is a miniature plating tank requiring only 267 mL (8.75 fluid ounces) of plating solution. By using different techniques, the Hull cell can evaluate:

- Deposit brightness & appearance over a very wide current density range of 0 - 200+ ASF (0 - 22+ ASD)
- Levels of Brighteners & other proprietary additives
- Bath chemistry such as zinc & caustic soda
- Plate distribution (throwing power)
- Covering power (ability to deposit zinc in the L.C.D.)
- Metallic & organic contamination
- Blister & adhesion characteristics of the deposit
- Effects of temperature
- Effects of time & current
- Effects of addition agents
- Effects of treatments such as carbon, peroxide, & potassium permanganate

Determination of cathode efficiency through use of a 400 mL Duplicell will also be covered in this manual.

HULL CELL OPERATING PROCEDURE

PURPOSE

This section will cover the techniques needed to properly prepare a Hull cell and steel panel for testing plate distribution, deposit appearance, deposit adhesion and COLZINC® ACF-II brightener content.

PROCEDURE

1. Thoroughly clean the Hull cell and zinc anode with warm tap water. If the zinc anode is dark or discolored, dip it in the 25 - 50% Hydrochloric Acid Pickle for 1 or 2 seconds and immediately rinse with tap water.

2. Add 267 mL of plating solution to the Hull cell.

3. Strip the zinc coated steel Hull cell panel in a 25 - 50% solution of Hydrochloric Acid Pickle. Immediately remove the panel when the gassing has stopped.

4. Holding the Hull cell panel along the top edge, place under cold tap water and thoroughly wipe with a clean paper towel to remove surface smut. A properly stripped and cleaned Hull cell panel should be water break free. Panels that are not properly cleaned will exhibit wipe marks, smears, fingerprints, and other blemishes.

   NOTE: Most steel Hull panels have a polished front side and a dull backside after stripping. The polished side should always be cleaned and face toward the anode. See figure 1.

Figure 1

5. Attach the red (positive) lead from the rectifier to the zinc anode and the black (negative) lead to the steel panel (cathode). Set the timer and amperage to the appropriate levels and turn on the rectifier.

   NOTE: Agitation is not required for Hull cell evaluation of alkaline cyanide free plating solutions. See figure 2.

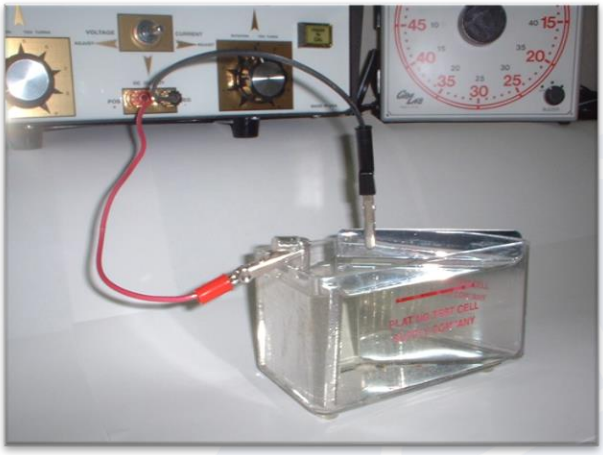


Figure 2

6. Upon completion of the test, remove the panel and thoroughly rinse under cold tap water. Next, half dip the panel for 4 seconds in a 0.5% solution of nitric acid to remove the yellow tarnished film on the zinc deposit surface. See figure 3.

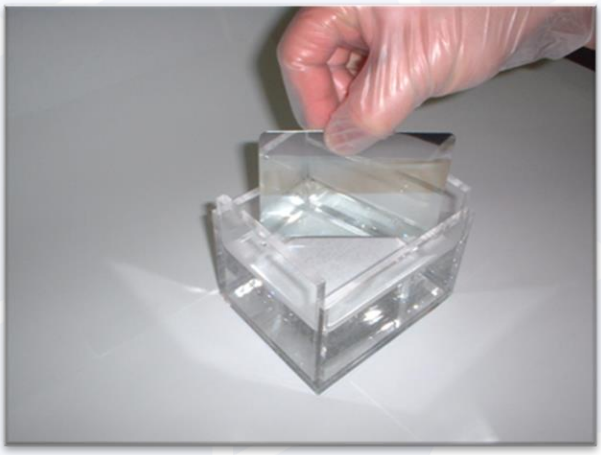


Figure 3

7. Immediately rinse the panel under cold tap water and dry. Panels can be dried using forced hot air from a blow dryer or by simply wiping with a clean paper towel. If using a paper towel, always wipe from right to left.

EVALUATING DEPOSIT BRIGHTNESS AND APPEARANCE

PURPOSE

The art of evaluating the general appearance of the zinc deposit on a Hull cell panel is critical for troubleshooting and determining the condition of the plating solution. This section covers one of the most important aspects of zinc plating, overall brightness and appearance of the deposit.

PROCEDURE

1. Prepare the Hull cell and panel by following the Hull Cell Operating Procedures in steps 1 through 5 on pages 4 and 5.

2. Heat the bath to 80° F (25° C) and run a 3 ampere-3 minute and 1 ampere-5-minute Hull cell panel (no agitation). Rinse, nitric dip and dry the panels by following the Hull Cell Operating Procedures in steps 6 and 7 on page 5.

3. Satisfactory panels should be uniform in brightness and free of mid to high current density striations and low to mid current density bands. See figure 4.

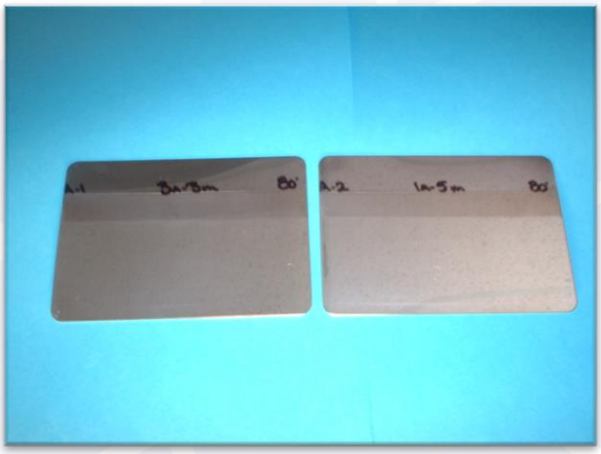


Figure 4

4. Refer to the COLZINC® ACF-II Laboratory Troubleshooting Section on pages 11 - 14 if the Hull cell panels exhibit dullness, bands, striations, etc.

PLATE DISTRIBUTION TEST FOR DETERMINING COLZINC® ACF-II BRIGHTENER CONTENT

PURPOSE

A very important and fairly unique characteristic of COLZINC® ACF-II alkaline cyanide free plating solutions is the ability to plate in deep recesses without over-plating high current density areas. Optimum plate distribution can be determined through this simple procedure.

PROCEDURE

1. Prepare the Hull cell and panel by following the Hull Cell Operating Procedures in steps 1 through 5 on pages 4 and 5.

   NOTE: Use a stripped 2 ½ in. x 2 ½ in. steel anode cut from a Hull panel in place of the zinc anode.

2. Heat the bath to 80° F (25° C) and run a 2 ampere-30-minute Hull cell panel (no agitation).

3. Do not nitric dip this panel (skip step 6 in the Hull Cell Operating Procedure); rinse and dry the panel by following step 7 on page 5.

4. Using a Hull cell ruler, take thickness readings at 4 ASF (0.43 ASD) and 80 ASF (8.64 ASD). See figure 5.

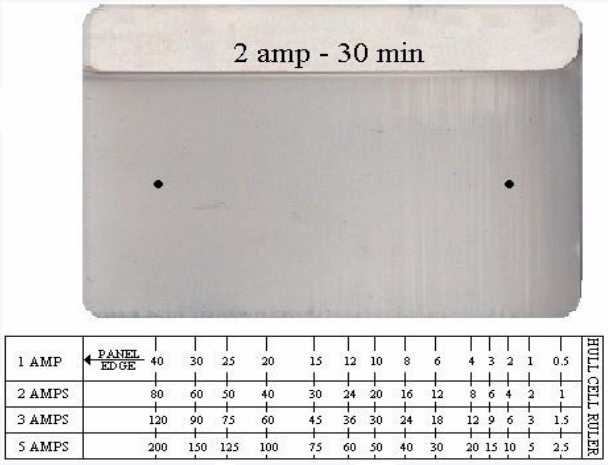


Figure 5

5. Calculate the high to low current density thickness ratio by dividing the thickness of the H.C.D. reading by the thickness of the L.C.D. reading.

   Example: 0.96 mil (24.4 microns) ÷ 0.53 mil (13.5 microns) = 1.8 ratio
   The optimum thickness ratio is 1.5 - 2.25. A thickness ratio above 2.25 is an indication of low brightener. A thickness ratio below 1.5 is an indication of high brightener.

   NOTE: This test should be used as a simple tool for evaluating the level of brightener relative to the zinc metal. Caustic soda content, chromium contamination, bath temperature, poor filtration, COLZINC® ACF Purifier overload, and organic contaminants can affect this test.

CATHODE EFFICIENCY TEST

PURPOSE

The deposition rate or the plating speed is directly related to cathode efficiency. A bath with a low cathode efficiency of 35% will plate much slower than bath with a moderate cathode efficiency of 80%. For testing and evaluation purposes, typical COLZINC® ACF-II plating baths operate in the 65 - 85% cathode efficiency range.

PROCEDURE

1. Thoroughly clean a 400 mL Duplicell.

   NOTE: Use a stripped 2 ½ in. x 2 ½ in. steel anode cut from a Hull panel in place of the zinc anode.

2. Add 400 mL of plating solution to the Duplicell.

3. Strip and clean a zinc coated steel Hull cell panel following the Hull Cell Operating Procedures in steps 3 and 4 on page 4.

4. Completely dry both sides of the panel with a clean paper towel or blow dryer to remove all excess water and moisture.

5. Weigh the panel on an electronic or mechanical balance and record the figure in grams as the panel net weight.

   NOTE: The scale/balance should be accurate to 0.01 grams.

6. Place the panel in the Duplicell, heat the bath to 80° F (25° C) and run at 1 ampere- 60 minutes.

   NOTE: Agitation is not necessary. See figure 6.

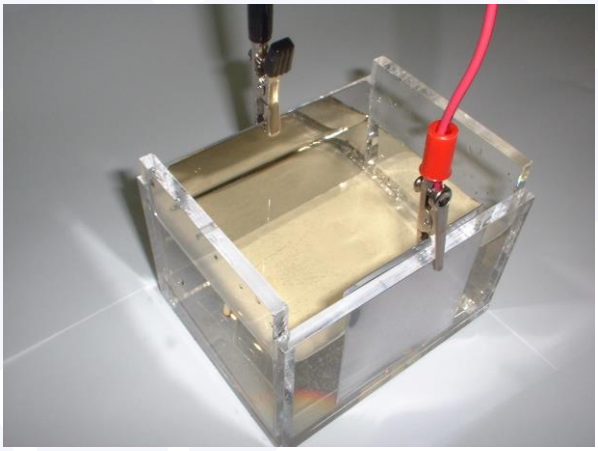


Figure 6

7. Rinse the panel with cold tap water and completely dry with a clean paper towel or blow dryer to remove all excess water and moisture.

8. Re-weigh the panel and record the figure in grams as the panel gross weight.

9. Calculate the weight of the zinc deposit by subtracting the panel net weight (weight of the steel panel) from the panel gross weight (weight of the zinc deposit and steel panel).

   Panel gross weight (grams) – Panel net weight (grams) = Weight of zinc deposit (grams)

10. Determine the cathode efficiency using the theoretical value of 1.219 grams of zinc deposited per 1 ampere-hour at 100% efficiency.

    Percent Cathode Efficiency Calculation: Weight in grams of zinc ÷ 1.219 x 100

    NOTE: Typically, COLZINC® ACF-II plating baths operate in at 65 - 85% efficiency range. Factors such as zinc metal, caustic soda, sodium carbonate levels, and brightener content will affect the cathode efficiency.

BLISTER TEST

PURPOSE

The ability to plate a thick, bright deposit that is ductile and free of blisters is critical to the zinc plating process. This test will determine the adhesion and ductility characteristics of the deposit by placing thermal stress on a zinc plated Hull panel.

PROCEDURE

1. Prepare the Hull cell and panel by following the Hull Cell Operating Procedures in steps 1 through 5 on pages 4 and 5.

   NOTE: Use a 2 ½ in. x 2 ½ in. steel anode cut from a steal Hull panel in place of the zinc anode.

2. Heat the bath to 80° F (25° C) and run a 2 ampere-30-minute Hull cell panel (no agitation).

3. Do not nitric dip this panel (skip step 6 in the Hull Cell Operating Procedure); rinse and dry the panel by following step 7 on page 5.

4. Place panel in a pre-heated oven and bake for 1 hour at 375° F (190° C)

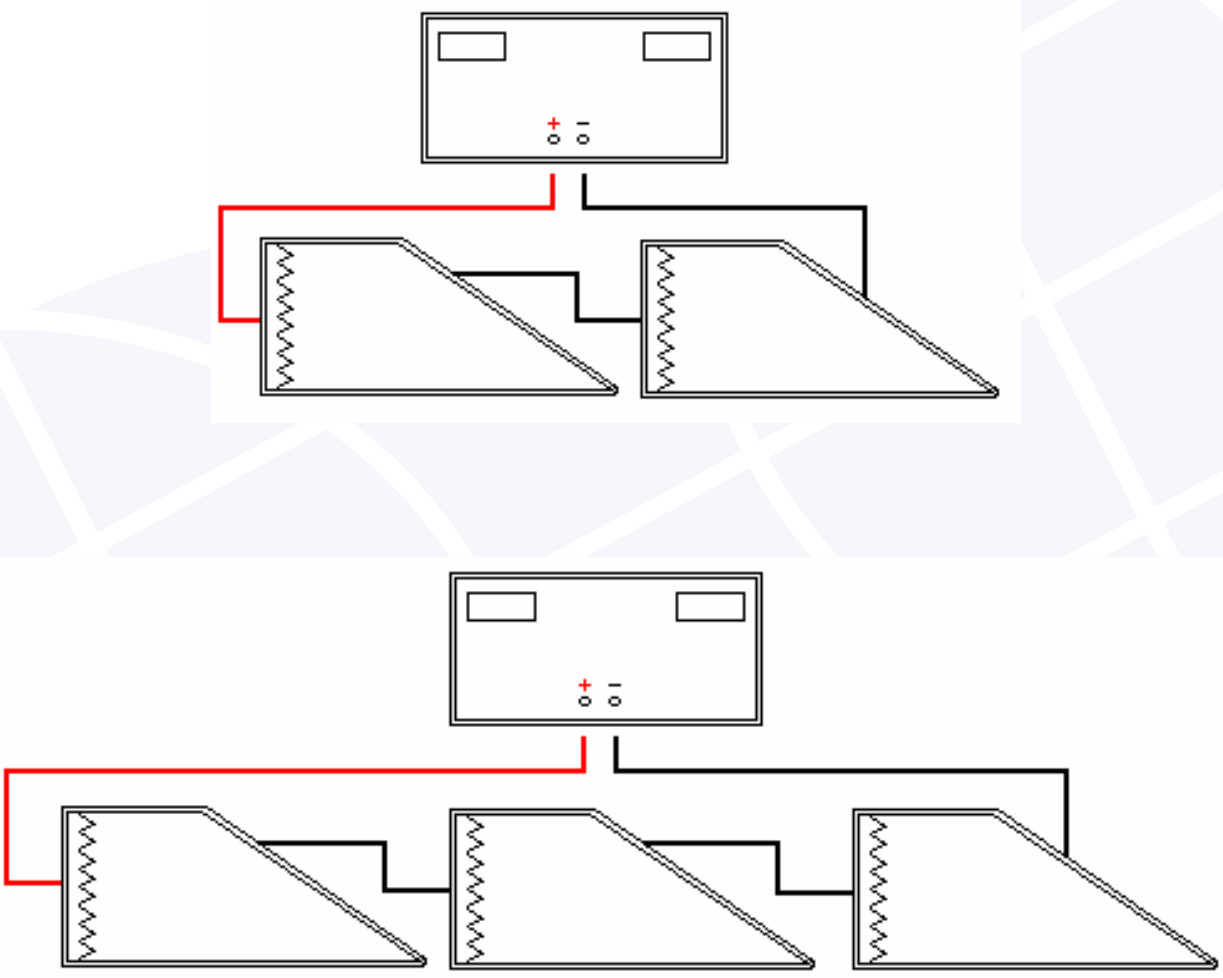
5. Remove panel and immediately quench in an ice water bath. Evaluate panel for blisters or adhesion problems.

   NOTE: Blistering of COLZINC® ACF-II baths is very rare. If the baked panel develops blisters, refer to the COLZINC® ACF-II Laboratory Troubleshooting Section on pages 11 - 14.

RUNNING MULTIPLE HULL CELLS OR DUPLICELLS ON A SINGLE RECTIFIER

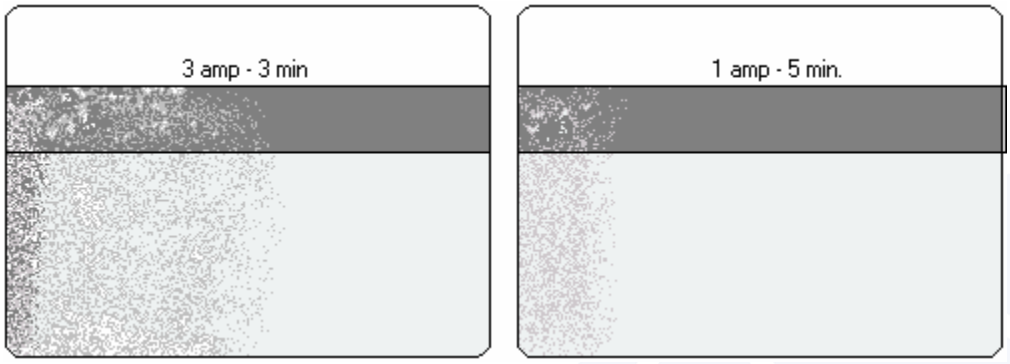
Multiple Hull cells can be wired in series to:

- Evaluate two or three baths in the time it takes to run one bath. This is especially important when running extended tests such as 2 amp-30-minute plate distribution panels.
- Evaluate two or three baths using identical current and time.
- Run side-by-side tests of a bath “as is” and a duplicate with addition agents or bath chemistry adjustments.



LABORATORY TROUBLESHOOTING GUIDE

1. Overall Dullness Caused by High COLZINC® ACF-II Brightener



- Add 2.7 mL Sodium Hypochlorite to the Hull cell and mix for 10 minutes to oxidize/destroy the excess Brightener.

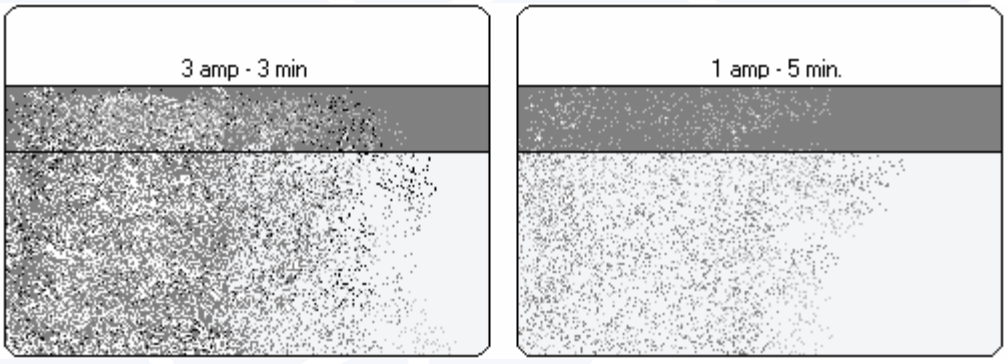
or

- Add 3 mL 1% Potassium Permanganate to the Hull cell and mix for 30 minutes to oxidize/destroy the excess Brightener.

or

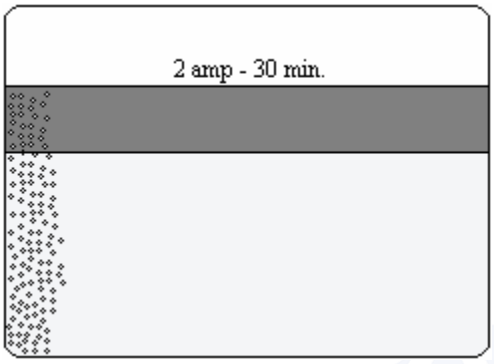
- Add about 0.1 grams Activated Carbon Powder to the Hull cell, mix for 30 minutes and filter well to remove the excess Brightener.

2. Overall Dullness/Grainy Mid to High Current Density Deposit from a Turbid Plating Solution



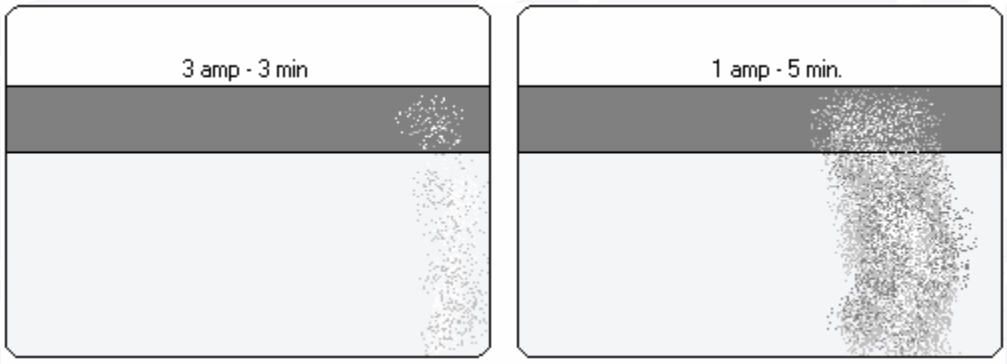
- Filter bath through triple 5-micron filter papers

3. **Blisters on Baked Panels from High Brightener, Organic Contamination or Chromium Contamination**



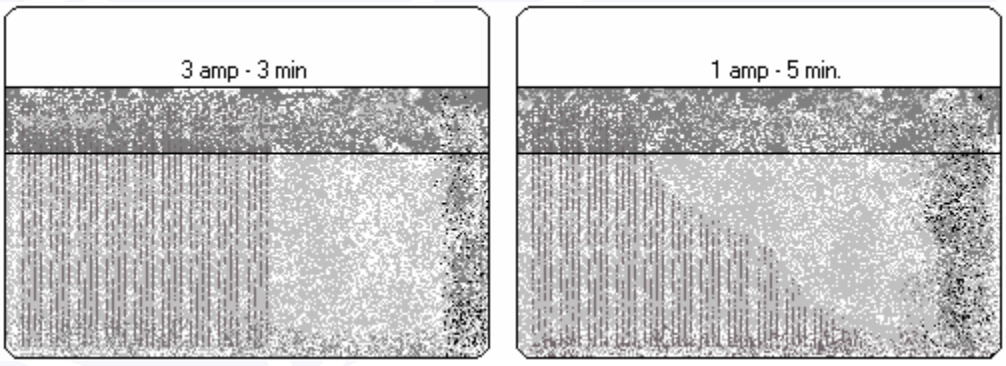
- See recommendations for (1) above
- Add 0.5 to 1 mL COLZINC® ACF Ductilizer to the Hull cell
- Add 3 mL of 1% Sodium Bisulfite solution to the Hull cell and mix for 5 minutes to reduce the hexavalent chromium to trivalent chromium

4. **Low Current Density Band and Dullness from Caustic Soda Impurities or Water Hardness**



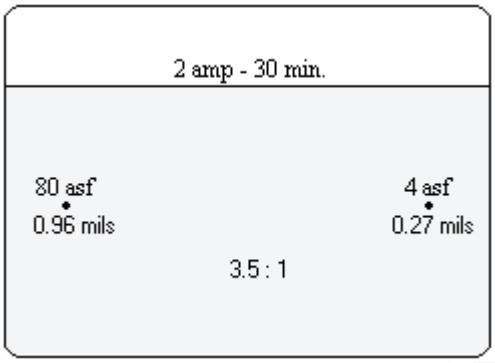
- Add 0.25 to 0.5 mL COLZINC® ACF Purifier to the Hull cell

5. **Mid to High Current Density Striations from Low Brightener**



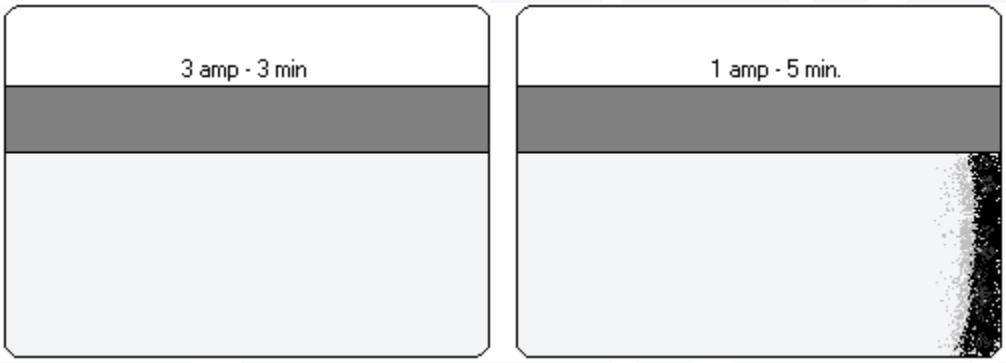
- Add 0.5 to 3 mL COLZINC® ACF-II Brightener

6. **Poor Plate Distribution (Ratio >2.25)**



- Add 0.5 - 3 m. COLZINC® ACF-II Brightener

7. **Gray to Black Discoloration of the Deposit after Nitric Dip from Metallic Contamination (>5 ppm Lead, Cadmium, and Copper)**

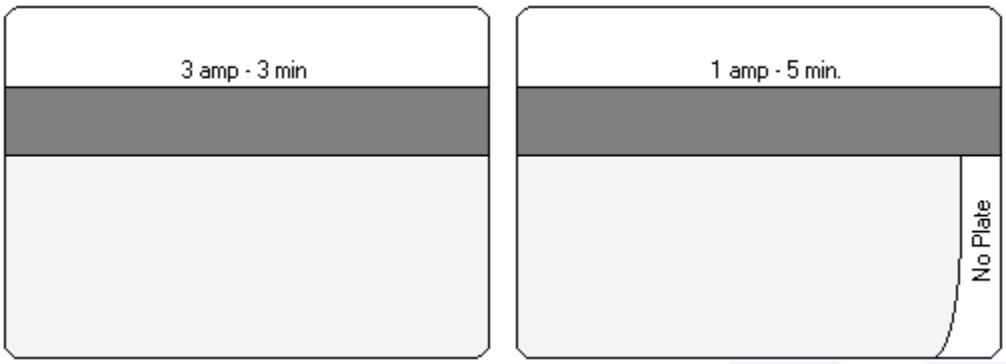


- Add 0.1-gram Zinc Dust to the Hull cell, mix for 15 minutes, allow to settle, and decant clean solution into another Hull cell.

8. **Overall Smoky/Cloudy Deposit with Underlying Brightness due to Organic Contaminants**

- Add 0.25 - 0.5 mL COLZINC® ACF Purifier to the Hull cell

9. Thin Zinc Deposit in the Low Current Density (Zinc quickly strips off in the nitric dip)



- Increase the zinc metal 0.5 - 1.0 opg (3.75 - 7.5 g/l) using Zinc Concentrate Solution. *Zinc Concentrate is made by dissolving 353 grams of zinc oxide in 2837 grams of Caustic Soda Liquid (50%) and diluting to 1 gallon. This will yield 10 opg zinc metal and 50 opg caustic soda. This solution is used at 26.7 mL to a Hull cell to raise the zinc 1.0 opg and the caustic soda 5 opg.*

- Freeze-out the carbonates by cooling the plating solution to 35° F (2° C) for 2 or more hours

COLZINC® ACF-II BATH ANALYTICAL PROCEDURES

ZINC METAL ANALYSIS

1. Pipette a 5 mL bath sample into a 250 mL Erlenmeyer flask and add 50 mL of distilled water.
2. Add 20 mL Ammonium Hydroxide Buffer solution.
3. Add approximately 0.2 grams Eriochrome Black T Indicator to turn solution to a medium purple.
4. Add 20 mL 8% Formaldehyde.
5. Immediately titrate with 0.0575M EDTA to a blue endpoint.

   CALCULATION: Milliliters 0.0575M EDTA X 0.1 (factor) = OPG Zinc Metal

CAUSTIC SODA ANALYSIS

1. Pipette a 5 mL bath sample into a 250 mL Erlenmeyer flask.
2. Add 10 mL of 10% Sodium Cyanide.
3. Add 2 mL Caustic Blue Indicator.
4. Titrate with 0.94N Sulfuric Acid to a color change of yellow-green to a blue endpoint.

   CALCULATION: Milliliters 0.94N Sulfuric Acid = OPG Caustic Soda

SODIUM CARBONATE ANALYSIS

1. Pipette a 5 mL bath sample into a 250 Erlenmeyer flask and add 100 mL of distilled water.
2. Add 2 mL Caustic Blue Indicator and titrate with 0.94N Sulfuric Acid to a color change of yellow-green to a blue endpoint.
3. Add 2 mL Methyl Orange Indicator and titrate with 0.94N Sulfuric Acid to a color change of gray-green to a gray-purple endpoint.

CALCULATION: Milliliters 0.94N Sulfuric x 1.3 (factor) = OPG Sodium Carbonate