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COLSID APD

TECHNICAL DATA
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COLSID APD

PROCESS FOR BRIGHT CHLORIDE ZINC PLATING BATHS CONTAINING AMMONIUM CHLORIDE AND POTASSIUM CHLORIDE

COLSID APD	produces a brilliant, level, ductile zinc deposit from acid chloride zinc plating electrolytes.
COLSID APD	is specially designed for mixed NH_4Cl/KCl baths.
COLSID APD	baths do not require boric acid, eliminating an extra expense, time consuming analysis, and crystallization on anodes in cold weather.
COLSID APD	baths can operate at extremely high temperatures of 125°F+ (52ºC+).
COLSID APD	contains a special ductilizer compound which ensures extremely ductile, bright deposits.
COLSID APD	operates with little or no foam and can be used with air agitation and evaporative recovery systems.
COLSID APD	is extremely tolerant to organic contaminants such as oils/cleaner surfactants and inorganic contaminants such as iron/copper.
COLSID APD	deposits readily accepts conventional and high corrosion trivalent chromate technologies.

OPERATING PARAMETERS

	RACK AND BARREL BATHS
Zinc Metal:	2.0 - 5.0 oz./gal. (15 - 37.5 g/l)
Chloride Ion:	16 - 22 oz./gal. (120 - 165 g/l)
COLSID APD CARRIER:	2.5 - 5% by volume
pH (Electrometric):	5.0 – 6.0
Temperature:	70º - 125º F (22º - 52º C)

SOLUTION MAKEUP

	100 LITERS	100 GALLONS
Zinc Chloride:	4.0 Kg	33 Pounds
Potassium Chloride:	19.0 Kg	158 Pounds
Ammonium Chloride:	3.5 Kg	29 Pounds
COLSID APD CARRIER:	4.0 Liters	4 Gallons
COLSID AP BRIGHTENER:	100 mL	385 mL

The bath is prepared by dissolving zinc chloride, potassium chloride, and ammonium chloride in hot water. The volume of the hot water should be roughly two-thirds the final volume of the plating solution. After all the salts have been thoroughly dissolved, **dilute the required amount of COLSID APD CARRIER with at least equal parts of water**; add to the bath and mix until dispersed. Follow the same procedure with the COLSID AP STARTER and the COLSID AP BRIGHTENER. Dilute the bath to the final volume and mix well.

Make-up of the bath as recommended above will yield the following bath chemistry:

Zinc Metal:	2.5 oz/gal (18.8 g/l)
Chloride Ion:	18.0 oz/gal (135 g/l)
Ratio $KCl:NH_4Cl$*:	5:1
pH (Electrometric):	5.6

*The ratio between potassium chloride and ammonium chloride is generally maintained at approximately 5:1, i.e. for every 100 pounds of chloride ion required, as determined by analysis, add 175 pounds potassium chloride and 34 pounds ammonium chloride.

EQUIPMENT

Filtration	Continuous filtration through polypropylene filter tubes of approximately 15 microns is recommended for routine operation. When carbon treatment or other bath purification is necessary, 5 - 10-micron filter tubes should be substituted.
Equipment	All plating tanks, racks, carriers, etc., which come into contact with COLSID APD solutions should be plastisol, polyethylene, hard rubber, or similarly coated to provide adequate protection from corrosion.
Agitation	Unlike many competitive processes, COLSID APD does not foam excessively and both mechanical and air agitation can be used.
Ventilation	The spray from COLSID APD solutions (not fumes) is inherently corrosive. The use of fiberglass, PVC, or polyethylene ventilation equipment and exhaust fans is recommended to prolong equipment life.

Cooling	Cooling coils made from Teflon are optimum, but titanium coils may be used as long as they are insulated from the electrical circuit. Lead or steel coils are not suitable.

MAINTENANCE ADDITIONS

COLSID AP BRIGHTENER is maintained in the plating bath at the rate of one gallon per 15,000 to 20,000 ampere-hours. Additions should be made every two to four hours of operation or continuously through the use of an addition agent pump.

COLSID AP-O BRIGHTENER is a highly concentrated solvent-containing brightener added to the plating bath at the rate of one gallon per 25,000 to 30,000 ampere-hours. Additions should be made every two to four hours of operation or continuously through the use of an addition agent pump.

COLSID APD CARRIER must be replaced in the plating bath as it is lost by drag-out. COLSID APD CARRIER can also be lost due to frequent treatment for iron. The most efficient and effective way to replace COLSID APD CARRIER is to add it along with COLSID AP BRIGHTENER additions, as follows:

LOW DRAGOUT/LOW IRON CONTAMINATION (Most Rack Baths):
add 1-gallon APD CARRIER per 4 to 5 gallons AP BRIGHTENER
(add 1-liter APD CARRIER per 4 to 5 liters AP BRIGHTENER)

MEDIUM DRAGOUT (Most Barrel Baths):
add 1-gallon APD CARRIER per 1-gallon AP BRIGHTENER
(add 1-liter APD CARRIER per 1-liter AP BRIGHTENER)

HIGH DRAGOUT/HIGH IRON CONTAMINATION (Some Barrel Baths):
add 2 gallons APD CARRIER per 1-gallon AP BRIGHTENER
(add 2 liters APD CARRIER per 1-liter AP BRIGHTENER

Maintenance of COLSID APD additives should be checked by periodic Hull cell evaluations.

TYPICAL CYCLE

PRE-PLATE TREATMENT

The following cleaning and pickling cycle is recommended when using the COLSID APD process:

1. Hot alkaline soak clean.
2. Hot alkaline electro-clean (anodic current / anodic cleaning).
3. Rinses.
4. 30% to 50% Muriatic Acid pickle with 1% to 2% COLUMBIA PICKLE PAL or PICKLE PAL PLUS.
5. Rinses.
6. Plate.

POST-PLATE TREATMENT

COLSID AP deposits are whiter and brighter than many competitive systems and provide surfaces that are highly receptive to most conventional hexavalent and high corrosion trivalent technologies.

ANALYTICAL PROCEDURE

ANALYSIS FOR ZINC METAL

1. Pipette 5 mL bath sample into a 250 mL Erlenmeyer flask and add 100 mL distilled water.
2. Add 10 mL ammonium hydroxide buffer solution.
3. Add approximately 0.2 grams Eriochrome Black T Indicator Mix.
4. Add 10 mL 8% formaldehyde solution.
5. Titrate immediately with Standard EDTA Solution 0.0575 M to a blue endpoint.

FACTOR: (mL Standard EDTA Solution 0.0575 M) x 0.10 = oz/gal zinc metal. (oz/gal x 7.5 = g/L)

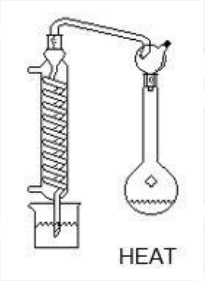
ANALYSIS FOR TOTAL CHLORIDE

1. Pipette 10 mL bath sample into a 250 mL volumetric flask. Dilute to 250 mL with distilled water and mix well.
2. Pipette 10 mL of above dilute solution into a 500 mL Erlenmeyer flask and add 100 mL distilled water.
3. Add 5 mL Sodium Chromate Indicator.
4. Titrate with Standard Silver Nitrate Solution 0.153 N to a reddish-brown endpoint. (The first permanent brown color is the endpoint.)

FACTOR: (ml Standard Silver Nitrate Solution 0.153 N) x 1.82 = oz/gal Chloride. (oz/gal x 7.5 = g/L)

ANALYSIS FOR AMMONIUM CHLORIDE

1. Pipette 25.0 mL of 1.00 N HCl into a 400 mL beaker and add 100 mL distilled water.
2. Add 25 mL 50% NaOH to a 250 mL distilling flask and add 100 mL distilled water.
3. Pipette 10 mL bath sample into the distilling flask and seal instantly so that no ammonia escapes.
4. Distill approximately 75 mL into the standard HCl solution, making sure that the tube from the condenser remains under the level of the standard HCl solution.
   Note: Do not allow the distillation flask to cool while the tube from the condenser is still in the solution.
5. Titrate the standard HCl solution with 1.00 N NaOH to a pH of 7.0.



FACTOR: $\frac{(25 - \text{ml } 1.00 \text{ N NaOH})}{1.4}$ = oz/gal NH_4Cl (oz/gal x 7.5 = g/L)

PREPARATION OF ANALYTICAL REAGENTS

EDTA 0.0575 M	Dissolve 21.6 grams C.P. Di-sodium EDTA salt in distilled water; dilute to exactly one liter.
8% Formaldehyde Solution	Dilute 200 ml 40% C.P. Formaldehyde to one liter with distilled water.
Silver Nitrate 0.153 N	Add 6 mL Nitric Acid to 26.0 grams C.P. Silver Nitrate. Dissolve in distilled water, dilute to exactly one liter.
Eriochrome Black "T" Indicator Mix	Grind together 1-part indicator and 100 parts sugar.
Sodium Chromate Indicator	Dissolve 10.0 grams Sodium Chromate in 100 mL distilled water.

HELPFUL HINTS

pH	The pH of the plating bath should be maintained within operating limits by the addition of dilute hydrochloric acid. Care should be taken during pH adjustment, as the pH changes rapidly with small additions of acid. Hydrochloric acid should be diluted with equal parts water prior to adding to the plating bath to avoid localized precipitation of addition agents. The pH should be checked using a meter, not pH paper. A high pH, over 6.0, causes low current density dullness. Low pH conditions are not normally encountered and occur only if excessive amounts of hydrochloric acid have been added during pH adjustment. If this occurs, diluted ammonium hydroxide or potassium hydroxide may be added to raise the pH to the proper operating level. Ammonium hydroxide is preferred because it is instantly soluble in the bath.
Ammonium Chloride/ Potassium Chloride	Ammonium Chloride and Potassium Chloride are the only chemical additions required in the plating bath during normal operation. They should be added on a regular basis based on periodic analysis for chloride. The following equivalents should be noted when maintaining the chloride content of the bath: Ammonium Chloride, NH4Cl, contains 66% chloride Potassium Chloride, KCl, contains 48% chloride Zinc Chloride, ZnCl, contains 52% chloride Therefore, to raise chloride content 1.0 oz/gal or 7.5 g/L requires: 0.34 oz/gal or 2.5 g/L Ammonium Chloride and 1.75 oz/gal 13 g/L Potassium Chloride
Zinc Metal	Zinc Metal is normally maintained by anodic dissolution during electrolysis. High grade zinc slab anodes with a minimum of 99.99% purity are recommended as an economical anode source. Anodes may be drilled and tapped or used in titanium anode baskets. Dissolution of the zinc anode does not occur during idle periods and anodes do not have to be removed from the bath during shutdowns. It is recommended to maintain as much anode area as possible to promote good current distribution. Anode baskets should be kept full. Acid resistant anode bags of cotton, dynel, or polypropylene are recommended for rack operation to reduce anode-caused roughness.
Operating Temperature	COLSID APD baths have a very wide range of bright operating temperatures, from approximately 70º F to over 120º F (22º C to over 48º C). The optimum temperature range for best brightness at minimum brightener consumption is 80º F to 85º F (25º C to 28º C), and the bath should be maintained at this temperature whenever economically feasible by cooling coils or refrigeration systems.

HANDLING & STORAGE

Columbia Chemical recommends referring to the specific product Safety Data Sheets for safety, handling, and storage precautions.

NON-WARRANTY

The data contained in this bulletin is believed by Columbia Chemical Corp. to be accurate, true, and complete. Since, however, final methods of use of this product are in the hands of the customer and beyond our control, we cannot guarantee that the customer will obtain the results described in this bulletin, nor can we assume responsibility of the use of this product by the customer in any process which may infringe the patents of third parties.