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ISO 9001:2015 CERTIFIED

NOVALYTE 370

CYANIDE-FREE CADMIUM PLATING PROCESS

INTRODUCTION:

The Aldoa Company has developed the Novalyte 370 process that utilizes a slightly alkaline electrolyte completely free of any cyanide bearing constituents. Initially the Novalyte 370-A addition agent was developed to produce a semi-bright to bright deposit for industrial application. The use of the addition agent markedly improved the throwing and covering power of the electrolyte. The deposits obtained have excellent base metal adhesion, good luster, and ductility. The process was limited to barrel plating only. The development of the Novalyte 370 B additive has extended the high current density range. Now it is possible to use the Novalyte 370 system for rack plating as well.

Aldoa's Novalyte 370 Cadmium Plating Process has a Boeing approval under BAC 5701, revision (J) 08-03-89.

SOLUTION COMPOSITION:

<u>Constituents</u>	<u>Concentration</u>	
Cadmium, metal	1.0 – 2.5 oz/gal	7.5 – 20 g/l
Chloride	2.0 – 3.5 oz/gal	15.0 – 30 g/l
Ammonium Sulfate	10 – 16 oz/gal	75.0 – 120 g/l
Novaplex*	10% by volume	
Novalyte 370-A	1.0 – 1.5% by volume	
Novalyte 370-B	0.5 – 1.0% by volume	
Novalyte 370-PR	0.01 – 0.10% by volume	

OPERATING CONDITIONS:

Temperature	60 - 90°F	16.0 – 32.0°C
pH	6.5 – 8.5	
Average Cathode Current Density	2 – 25 ASF	0.2 – 2.5 A/dm ²
Anode Current Density	5 – 40 ASF	0.5 – 4.0 A/dm ²
Voltage	6 – 12 Volts	

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OPERATING CONDITIONS (continued):

*Novaplex additive acts as a buffer, anode corrosion agent, and cadmium ions stabilizer at the pH of the electrolyte. Novaplex is added once in the beginning only. In case drag-out losses are significant, an appropriate amount of Novaplex must be replenished.

SOLUTION PREPARATION:

The Novalyte 370 solution should be prepared in a clean storage or treatment tank equipped with a mechanical mixer. If such tanks are not available, the solution can be prepared in the thoroughly cleaned plating tank. It is again desirable to have some means of mechanical agitation.

The Novalyte 370 solution can be prepared by the utilization of a prepared proprietary electrolyte concentrate, namely Novalyte 370-E, or from the basic chemical ingredients. These methods are described below:

Procedure

- A. Fill tank with water to one-half the final volume. Dissolved the requisite amount of Ammonium Sulfate. Then add an appropriate amount of Novalyte 370-E (10% by volume).
- B. To make 100 gallons of electrolyte containing, for instance, two ounces per gallon cadmium metal.
 1. Take 230 ounces of cadmium oxide and make slurry with 6 to 10 gallons of water in a PVC lined or stainless steel container.
 2. While stirring add slowly 220 to 230 fluid ounces of muriatic acid to get a clear solution.
 3. Fill the plating tank with water to one-half the final volume. Dissolve required amount of ammonium sulfate and Novaplex (1400 ounces and 10 gallons, respectively for 100 gallon solution in above example)/ finally add the cadmium chloride solution prepared in Step 2 above and mix thoroughly to obtain a clear solution.

FUNCTION OF CONSTITUENTS:

Cadmium Oxide

The Cadmium Oxide is used to produce cadmium ions in the plating solution. Chloride and Sulfate ions enable cadmium metal to produce cadmium ions at the anode.

Ammonium Sulfate

Ammonium Sulfate is used to complex cadmium ions as well as increase conductivity of the solution. Chloride and Sulfate ions enable cadmium metal to produce cadmium ions at the anode.

Novaplex

Novaplex is used in the original bath to stabilize the cadmium complex, to prevent anode sludge formation, and as a buffer at the cathode or work surface. Thereafter, it is required occasionally only. Increased throwing power of the bath is observed with increased concentration of Novaplex and Ammonium Sulfate.

Novalyte 370-A

The addition agent serves to produce grain refinement, to promote the throwing and covering power as well as the bright current density range of the deposit. The Novalyte 370-A has excellent stability during electrolysis as well as during the periods of idleness of the bath. Excess of Novalyte 370-A is not harmful, however, for economic consideration addition of Novalyte 370-A should be made in accordance with requirement.

The rate of brightening is excellent, and when normal addition of Novalyte 370-A fails to produce the desired results, it is recommended to check solution temperature and composition, cleaning cycle, and other factors rather than make unnecessary extraneous additions of brightener.

Novalyte 370-B

Novalyte 370-B increase the high current density bright range as well as helps uniform metal deposition on the entire surface of the parts in rack plating application. Excessive use of Novalyte 370-B may reduce the plating efficiency of the bath.

Novalyte 370-W

This is a wetting agent with a dual property of reducing surface tension of the plating solution as well as an anti-pitting agent. Novalyte 370-W is very stable in the electrolyte and requires only an infrequent addition as desired.

Novalyte 370 PR

A liquid additive which is used as a purifier to improve the micro-grain refinement of the deposit, especially at the low current density areas. An addition of one gallon of Novalyte 370-PR per 1 – 5 thousand gallons of plating bath is recommended.

SOLUTION OPERATION AND CONTROL:

Cadmium Concentration

The anode dissolution takes place at all specified current densities at efficiencies slightly higher than cathode efficiencies at similar current densities, thus compensating for the cadmium metal losses due to plating and drag out. Control of anode area is sufficient to maintain cadmium metal concentration.

Ammonium Sulfate

The depletion of ammonium sulfate as a consequence of drag out and slight decomposition to ammonia because the pH of electrolyte is between 7 and 8. These losses must be replenished on a regular basis to maintain optimum concentration of ammonium sulfate.

Temperature

The recommended range is 65° - 90°F (16.0 – 32.0°C). The optimum operating temperature within this range should be determined after operational experience and maintained thereat for consistent results.

Cathode Current Density

Current density in barrel work is difficult to determine due to many factors such as surface finish of the parts, bulk density of the parts, etc.

An average current density of 7 to 10 ASF is considered normal, and the range can extend from 2 to 15 ASF. For rack application the average current density of 10 – 25 ASF (1.0 – 2.5 A/dm²) is recommended.

Anode Current Density

The anode current density range varies from 5 to 40 ASF. Under normal operating conditions the anode have a smooth crystalline appearance. The optimum current density should be determined so as to maintain cadmium concentration in the operating range.

EQUIPMENT:

Tanks

PVC or similarly lined tanks are required.

Coils

Heating or cooling coils and exchangers made of stainless steel and titanium are highly recommended.

Anode Containers

Titanium anode containers should be used.

Filtration

Filtration equipment should be constructed of stainless steel, titanium or PVC lined. Brass, copper or bronze fittings should be avoided in the circulation system.

CONTAMINANTS AND THEIR REMOVAL:

Inorganic

The removal of inorganic impurities is necessary to maintain the quality of the deposit especially where bright dipping or chromate treatments are used. Low current density electrolysis will remove many of the metallic contaminants, which are soluble in the system. Insoluble metallic hydroxide build up should be removed by constant filtration.

Organic

When a build up of organic contamination occurs due to ineffective removal of oils, greases, etc., during the cleaning operation, a periodic treatment with activated carbon is usually effective in removing such organic contamination. Carbon treatment may remove some of Novalyte 370-A, which should be compensated for before resuming electroplating operation.

CLEANING AND PREPARATION OF BASIC METALS:

Cyanide based electrolyte are known to perform some cleaning of the parts when being plated. Because of the fact that Novalyte 370 system is non-cyanide, one should not expect any additional cleaning in the electrolyte. Therefore, before the work goes into the Cadmium plating tank, parts must be thoroughly cleaned as deemed necessary by the nature of soil, oxide film, etc., in an appropriate cleaning and pickling system.

NON-WARRANTY:

Due to numerous factors affecting results, all Aldoa products are offered to purchasers with no guarantee, expressed or implied, as to the results obtained or to the effects derived from such use. Aldoa guarantees only as to formulated quality upon shipment from our plant.

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NOVALYTE 370

ANALYSIS OF CYANIDE-FREE CADMIUM SOLUTION

Filter about 100 ml of the plating solution through a filter paper. Use the filtrate for subsequent analysis.

CADMIUM METAL:

1. Take 2.0 ml sample in a 250 ml Erlenmeyer Flask.
2. Add 50 – 75 ml distilled water.
3. Add 10 ml reagent grade Ammonium Hydroxide.
4. Add small amount of Eriochrome Black T indicator, 10 ml of 10% Formaldehyde, and titrate against 0.0575 M EDTA solution until the purple color change to blue.

Calculation

Cadmium Metal = $0.43 \times \text{ml of EDTA used} = \text{oz/gal}$

AMMONIUM CHLORIDE:

1. Take one ml sample in porcelain dish about 4 in. in diameter.
2. Add 25 ml distilled water and 5 ml of 5% $\text{K}_2\text{Cr}_2\text{O}_7$ solution.
3. Add 0.1N Silver Nitrate drop by drop while stirring solution with a thin glass rod. A brick-red color indicated end-point.

Calculation

$\text{NH}_4\text{Cl (oz/gal)} + 0.713 \times \text{ml of Silver Nitrate solution}$

AMMONIUM SULFATE

A. Gravimetric Method

1. Pipette one-ml sample (filtered) into a 400-ml beaker and add 150 ml distilled water.
2. Add 5 to 10 ml of concentrated hydrochloric acid and boil under the hood.
3. Add, while stirring 20 ml of 10% barium chloride solution, boil for another minute and allow to settle in a warm place for one hour.
4. Filter, using a quantitative ashless filter paper and wash with warm water.
5. Ignite the precipitate and filter paper in a weighed crucible. Cool in a desiccator and weigh.

Calculation

Ammonium Sulfate (oz/gal) = 75.4 X gain in weight of the crucible
in grams.

NOTES

Sulfate can be determined with a sufficient degree of accuracy for most control purposes by the centrifuge method, in which a sample placed in a calibrated tube is spun, and the sulfate read directly. The apparatus is low in cost, and a determination may be obtained in about 10 minutes.

B. Centrifugal Method

Equipment: Kocour sulfate test set, and a centrifuge.

Chemicals:

Solution A- 100 ml distilled water plus 180 mls hydrochloric acid (reagent grade).

Solution B- 160 g reagent grade Barium Chloride dissolved in
500 ml distilled water.

1. Take 10 ml solution in a 100 ml volumetric flask, add distilled water to the mark. Mix well.
2. Take 5 ml of above solution (10 ml if total ammonium sulfate is less than 10 oz/gal) and 5 ml distilled water (no water needed if sample is 10 ml) in the Kocour tube.
3. Add 5 ml solution A. Shake well.
4. Add 10 ml solution B, replace stopper and shake well for one to two minutes.
5. Centrifuge for 5 to 10 minutes.

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Calculation

Multiply the reading on the stem by 53.4 for a 5 ml sample, and by 26.7 for a 10 ml sample, taken in step 2. This gives ammonium sulfate in oz/gal.

C. Volumetric Method

Solution:

Indicator: Freshly prepared by dissolving 15 mg. Of sodium salt of Rhodizonic acid in 5 ml distilled water. Ten drops are used for each titration.

Barium Chloride: 0.2 N Prepare by dissolving 24.431g A.R. Grade $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$ in distilled water and making the volume to one liter.

Procedure:

1. Take 5 ml of Plating solution in a 300 ml Erlenmeyer flask and add one gram of reagent grade ammonium chloride and a known volume V_1 (20 or 25 mls) of 0.2 N barium chloride solution and 10 drops of the freshly prepared indicator.
2. Shake well and add 3 drops of 10% hydrochloric acid. (additions of 10 mls of ethanol will improve the end point).
3. Titrate against 0.2 N potassium sulfate solution until the color begins to fade. Then add the titrant drop wise, with vigorous shaking and waiting for a short time between the drops, until the red color has completely disappeared. Record the volume of 0.2 N potassium sulfate used as V_2 .

Calculation

$(V_1 - V_2) \times 5.28 = \text{g/l Ammonium Sulfate}$

or

$(V_1 - V_2) \times 0.704 = \text{oz/gal Ammonium Sulfate}$

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CHLORIDE, AS AMMONIUM CHLORIDE

1. Take one ml. sample in a Porcelain Dish about 4 in. in diameter.
2. Add 25 ml distilled water and 5 ml 0.1667 M. $K_2Cr_2O_7$ solution.
3. Add 0.1 N Silver nitrate drop by drop While stirring solution with a thin glass rod. A brick-red color indicates end-point.

CALCULATION

Total NH_4Cl = 0.713 X ml 0.1N Silver
Nitrate solution = oz/gal

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CYANIDE FREE CADMIUM (NOVALYTE 370)

EFFLUENT TREATMENT PROCEDURE

INTRODUCTION:

This treatment is recommended for an effluent containing less than 1,500-PPM (1.5 grams per liter) Cadmium metal. The sulfide used may be a solution of Sodium Sulfide or Sodium Polysulfide or Aldolyte ZP-471. Aquation CA-700 has been found to be an effective coagulant aid, that will enhance settling and aid in filtering the precipitated cadmium metal in the form of a mixture of cadmium sulfide and cadmium hydroxide.

If the solids are washed thoroughly after filtering, they may be converted to cadmium sulfate with sulfuric acid and may be added to the plating tank as cadmium replenishment. Consult with Aldoa Laboratory personnel for detailed explanation.

PROCEDURE:

It is suggested that a known quantity of the representative sample of the effluent be treated in the laboratory. The results obtained can then be translated to treat the known volume of the bulk effluent.

LABORATORY EQUIPMENTS AND CHEMICALS:

- One magnetic stirrer
- One 1500 ml glass beaker
- One 10 ml graduated pipette
- Two 1 ml graduated pipettes
- 20% by volume caustic soda solution
- Aquation CA-700
- Aldolyte SP-471

- Step 1. Take one liter (or one quart) of the effluent in a 1500 ml beaker and stir it either manually or on a magnetic stirring plate.
- Step 2. Using 1 ml graduated pipette, add; drop wise, Aldolyte ZP-471 until the solution becomes lightly yellow. Note the volume of Aldolyte ZP-471 used.
- Step 3. Slowly add 20% by volume caustic soda solution by means of graduated 10 ml pipette, till the pH is between 9 and 10. At this point, precipitate will appear. Note the volume of 20% caustic soda use.
- Step 4. While the solution is still being stirred slowly, add Aquation CA-700 drop wise by means of another 1 ml graduated pipette. Wait a few moments after the addition of each drop of Aquation CA-700.
- Step 5. The precipitate will appear to floc together. Stop adding when no fine precipitated remains suspended in solution. The coagulated solids will settle to the bottom very rapidly. Note the volume of Aquation CA-700
- Step 6. The supernatant liquid can be discarded after the solids are removed by filtration or by centrifuge. Each step 1 through 5 will provide sufficient information to treat a known volume of the effluent.

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ADDENDUM

NOVALYTE 370 – NON-CYANIDE CADMIUM PLATING PROCESS

BATH OPERATION AND CONTROL:

Novalyte 370 bath can be operated successfully at two pH ranges. Each range offers the operator certain advantages. The two pH ranges are:

- A. pH range 5.50 – 8.50
- B. pH range 4.80 – 5.20

The pH may be adjusted with ammonium hydroxide, glacial acetic acid (preferred) or sulfuric acid.

- A. pH range 5.50 – 8.50

This pH range offers the cadmium platers an alternative to the mildness of the cyanide type bath.

The chemistry of the bath being constant, the lower pH increases the high current density range by a factor of 10 – 50% without significantly affecting the coverage at low current densities. However, for a barrel type operation higher values of pH are recommended.

- B. pH range of 4.80 – 5.20

This pH range is recommended for those rack operations where a much wider current density range is more desirable.

1. All other variables being constant, the high current density range increases to more than 90 amperes/ft², while the low current density coverage is affected only slightly.
2. The conductivity of the bath at the pH range B is higher than that at pH range of A.

NOVAPLEX

1. As stated in the Technical Bulletin, Novaplex is added in the beginning only. It is true for cases where dragout is minimal and the metal concentration in the bath remains virtually constant. However, in the case of barrel and in some rack plating operations where dragout is significant, it may be necessary to make additions of Novaplex to compensate for the loss. It must be remembered that the Novaplex, as well as ammonium sulfate, help increase the throwing and covering ability of the bath.
2. It is advisable to maintain the concentration of Novaplex at a 6.0 ± 1 per cent by volume when the pH range A bath is used for rack plating application.

AMMONIUM CHLORIDE AND AMMONIUM SULFATE

The concentration of chloride ions (as ammonium chloride) should not exceed the recommended high limit. However, there is no such limit on the concentration of sulfate ions. Ammonium sulfate may be increased to more than 16.0 ounces per gallon concentration. Throwing and covering power of the bath increases with an increase in ammonium sulfate concentration.

NOVALYTE 370-PR

1. Ferrous iron build-up and certain other metallic impurities can cause darkening of cadmium deposit at the low current density areas on plated parts. Addition of one to two fluid ounces (diluted with water) of Novalyte 370-PR per one-hundred gallons of the bath will correct this problem. Make certain that the Novalyte 370-PR addition is thoroughly mixed throughout the plating bath.
2. Addition of Novalyte has demonstrated an increase in the throwing power of the bath, as well as brightening of the cadmium deposit.

AGITATION

It has been found that air agitation of the plating bath has reduced the problem caused by ferrous iron build-up. Moreover, due to air agitation of the bath, filtration of the plating solution becomes more efficient in the removal of ferric hydroxide and other suspended impurities, thus reducing their chance to co-deposit in the cadmium plating.

The air used for agitation should be furnished by means of turbo blower as opposed to a piston-type compressor. Details on the pressure and air volumes required for a particular installation can be obtained by contacting our Technical Staff.

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