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NiCOL BRIGHT INDEX

TECHNICAL DATA
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NICOL BRIGHT INDEX

INDEX BASED DUAL ADDITIVE SYSTEM FOR BRIGHT NICKEL PLATING

NICOL BRIGHT INDEX	two additives provide control of brightener a carrier individually.
NICOL BRIGHT INDEX	excellent physical properties such as leveling, brightness, and ductility for automotive applications.
NICOL BRIGHT INDEX	superb chromium coverage for plating complex geometries.
NICOL BRIGHT INDEX	can be used in duplex or multilayer nickel systems where extended corrosion resistance is required.
NICOL BRIGHT INDEX	can be used in air or mechanically agitated plating baths, as well as barrel plating.
NICOL BRIGHT INDEX	minimizes rejects from brittleness or cracking.

OPERATING PARAMETERS

	RANGE	OPTIMUM
Temperature:	120 - 150° F (50 - 65° C)	140° F (60° C)
pH:	3.5 - 4.4	4.2
Nickel sulfate ($NiSO_4.6H_20$):	110 - 375 g/L (15 - 50 oz/gal)	180 g/L (24 oz/gal)
Nickel chloride ($NiCl_2.6H_2O$):	38 - 150 g/L (5 - 20 oz/gal)	75 g/L (10 oz/gal)
Boric acid (H_3BO_3):	38 - 50 g/L (5 - 6.5 oz/gal)	45 g/L (6 oz/gal)
NICOL BN Index 00:	5 - 18 mL/L (0.5 - 1.8%/vol)	12 mL/L (1.2%/vol)
NICOL BN Carrier 10:	15 - 50 mL/L (1.5 - 5.0%/vol)	32 mL/L (3.2%/vol)
NICOL BN Brightener 30: w/air agitation w/mechanical agitation	 0.5 - 1.5 mL/L 0.75 - 1.75 mL/L	 0.75 mL/L 1.0 mL/L
Wetting Agents:	As required	
Current Density:	2.2 - 8.6 ASD (20 - 80 ASF)	4.3 ASD (40 ASF)

Agitation:	Air agitation from a low-pressure blower. Compressed air must not be used. Mechanical agitation is also suitable.
Anode Current density:	3.2 ASD (30 ASF) for air agitated solutions 1.8 ASD (17 ASF) for mechanical agitated solutions
Anode to Cathode Ratio:	1:1 - 2:1
Current – DC:	Less than 5% ripple
Voltage:	2 - 10 volts
Deposition Rate:	1.0 mil in 30 minutes at 40 Amps/ft^2 (25.4 microns in 30 minutes at 4.3 Amps/dm^2)
Cathode Efficiency:	94 - 96%
Filtration:	Continuous, 1 - 2 turnovers per hour, through a five-micron polypropylene cartridge or a horizontal plate filter packed with diatomaceous earth.
Ventilation:	Consult with an industrial engineer for recommendations.

SOLUTION MAKEUP

MATERIAL	**100 LITERS**	**100 GALLONS**
Nickel sulfate ($NiSO_4.6H_20$):	18 kg	150 lbs.
Nickel chloride ($NiCl_2.6H_2O$):	7.5 kg	62.5 lbs.
Boric acid (H_3BO_3):	4.5 kg	37.5 lbs.
NICOL BN Index 00:	1.2 liters	1.2 gallons
NICOL BN Carrier 10:	3.2 liters	3.2 gallons
NICOL BN Brightener 30: w/air agitation w/mechanical agitation	 75 mL 100 mL	 0.075 gallons 0.10 gallons
Wetting Agents:	As required	

SOLUTION PREPARATION

The plating solution should be made-up in a clean separate tank. Clean tank by filling with water then add 0.3% by vol sulfuric acid and 1 cc/L of the NICOL Wetter. Let solution leach overnight, then empty tank and rinse.

1. Fill the cleaned tank ½ full with water and heat to 140° F (60° C).
2. While stirring, add and completely dissolve the required amount of nickel sulfate and nickel chloride.
3. Raise the pH to 5.2 by adding nickel carbonate with vigorous stirring. When checking the pH obtain a 100 ml sample and filter or allow the sample to settle to get a true pH reading.

4. Add 3 mL/L of 30% hydrogen peroxide, well diluted with water.
5. Add 1 mL/L of NICOL Wetter.
6. Add 2.5 grams per liter of plating grade pulverized activated carbon. Stir for 1 hour, then keep heated overnight at 150° F (65° C) and allow the solution to settle.
7. Filter the solution into a clean plating tank that has been cleaned and leached like the storage tank.
8. Pack the filter with filter aid and carbon so there is approximately 0.2 g/L of each material.
9. With agitation add the required amount of boric acid.
10. Adjust the pH to 4.0 with 50% sulfuric acid.
11. Electrolyze the solution at a low current density, 0.54 ASD (5 ASF) using corrugated dummy cathodes. Usually this will take 8 hours, check low current density areas of the cathode for darkness. If darkness exists then continue dummy plating.
12. Clean filter then re-pack with filter aid.
13. Add the required amounts of addition agents.

EQUIPMENT

Tank or Liner:	CPVC, PVC, Koroseal-lined steel, or polypropylene.
Pumps:	Conventional plastic suitable for acid applications or high temperature.
Racks:	Plastisol-coated copper. Stainless steel should be avoided.
Heaters:	Quartz, titanium, or PTFE. The titanium should be grounded or anodically connected.
Filters:	1 - 2 turnovers per hour through a 5-micron polypropylene cartridge or horizontal plate filter packed with diatomaceous earth filter aid.
Anode Bags:	Dynel or polypropylene, washed then leached in a 5% by vol. sulfuric acid solution then flushed with hot water before use. Thread count should be about 38 x 29 per inch.
Anodes:	Sulfur depolarized nickel such as INCO S-Rounds, electrolytic squares or R-rounds.
Anode Hooks:	Monel or titanium.
Anode Baskets:	Titanium.

MAINTENANCE ADDITIONS

NICOL BN INDEX 00

NICOL BN Index 00 is used for make-up of new solutions. The NICOL BN Index 00 is synergistic with the NICOL BN Carrier 10, and the NICOL BN Brightener 30; together they produce bright leveled deposits. The proper amount of NICOL BN Index is in the NICOL BN Brightener 30. Normally extra

additions of the NICOL BN Index 00 are not required, unless high drag-out or extended high current density plating has occurred. NICOL BN Index 00 is not removed by carbon treatment.

NICOL BN CARRIER 10

NICOL BN Carrier 10 is added at solution make-up. NICOL BN Carrier 10 is synergistic with the NICOL BN Brightener 30 to produce brightness, leveling, and good ductility. Low concentrations of NICOL BN Carrier 10 will reduce the bright throw, decrease tolerance to impurities, lower the chromium acceptance, lower leveling, and decrease the ductility of the deposit. High concentration will not be harmful but may precipitate at concentration above 5.0%. Over the side additions may be necessary to maintain concentration from losses such as carbon treatment or drag out. Carbon treatments above 4 pounds/100gallons (480 grams/100 liters) may remove up to 50% of the NICOL BN Carrier 10. Solution can be analyzed using the procedure in this technical data sheet. NICOL BN Carrier 10 is added at 6340 amp-hr/L (24,000amp-hr/gallon) of operation.

NICOL BN BRIGHTENER 30

NICOL BN Brightener 30 contains the secondary brighteners, and the BN Index 00 in the consumptive proportions needed. High concentrations of NICOL BN Brightener 30 will reduce the ductility of the deposit. NICOL BN Brightener 30 is added at 5280amp-hr/L (20,000 amp-hr/gallon) of operation.

NICOL WETTER

Wetting agents are added at solution make-up and replenished by checking the surface tension. Wetting agents are readily removed with carbon. Concentration of wetting agents should be determined after any carbon is in contact with solution. As an approximate usage NICOL wetting agent should be replenished at 20,000 amp-hr/L (75,000 amp-hr/gallon). Carbon treatments such as 4 pounds/100gallons (480 grams/100 liters) will remove most of the wetting agents. Solution can be analyzed using the procedure in this technical data sheet.

NICKEL SULFATE

Complex shapes and/or the use of high current densities should have the higher nickel sulfate concentration. The nickel sulfate concentration contributes the nickel metal necessary to provide the latitude for the operating current density range. Lower nickel content can lead to high current density burning. Simple shapes and lower current densities may use concentrations of nickel sulfate as low as 15 oz/gal (112 g/L). Check nickel content to determine and maintain the correct nickel sulfate concentration.

NICKEL CHLORIDE

Nickel chloride provides approximately twice the conductivity of nickel sulfate. The nickel chloride concentration primarily contributes the chloride ion for proper anode corrosion and good bath conductivity. High concentrations can reduce ductility slightly.

BORIC ACID

The boric acid concentration contributes the cathode film buffering necessary for the bath to produce deposits with good ductility. High concentrations can cause sporadic pitting due to precipitation at lower temperatures. Air agitation pipes can also become clogged with high concentrations. Low concentrations can produce HCD burning, and reduced ductility.

pH

To lower the pH use dilute or 50% Sulfuric acid. It is not common to need to raise the pH, however if this is required add nickel carbonate to the slurry tank of the filter before exposing the nickel carbonate to the actual plating solution. Adding nickel carbonate directly to the solution can cause roughness.

TEMPERATURE

The temperature of the plating solution should be maintained between 120 - 160° F (50 - 70° C). Operating at higher temperatures will allow operation at higher current densities and improved solution conductivity and subsequent lower conductivity salts.

CURRENT DENSITY

The current density of the process should be maintained within the specified limits.

ANALYTICAL PROCEDURE

DETERMINATION OF NICKEL METAL

REAGENTS: Standard EDTA solution, 0.100 M
Ammonium Hydroxide, AR, concentrated
MUREXIDE INDICATOR - thoroughly grind about 0.25 grams of murexide powder with about five (5) grams of granulated table sugar. Transfer to a large wide mouth plastic capped jar and add about 95 grams sugar to the mixture. Shake vigorously to coat all sugar particles with the murexide mixture.

APPARATUS: 1 mL Pipette, volumetric.
10 mL Pipette, volumetric 10 mL Graduated cylinder. 250 mL Erlenmeyer flask.

PROCEDURE:

1. Pipette 1.00 mL of plating solution into a 250 mL Erlenmeyer flask.
2. Add about 50 mL DI or distilled water, mix.
3. Add about 10 mL of concentrated Ammonium Hydroxide.
4. Add about 0.15 grams of the murexide indicator mixture prepared as above.
5. While swirling, titrate with 0.100 M EDTA solution until the yellowish color sharply changes to purple.
6. Calculate the nickel concentration as follows:

    a. Nickel (oz/gal) = (mL EDTA) x M EDTA x 7.83 / mL sample
    b. Nickel (g/L) = Nickel (oz/gal) x 7.5

DETERMINATION OF NICKEL CHLORIDE

REAGENTS: Dichlorofluoresein indicator- (0.2% solution)
0.153N Silver Nitrate standardized solution

APPARATUS: 2 ml Pipette, volumetric
250 ml Erlenmeyer flask
50 ml Burette

PROCEDURE:

1. Pipette 2 ml of sample into a 250 ml Erlenmeyer flask.
2. Add 50 ml of DI water.
3. Add about 5 drops of Dichlorofluoresein indicator.
4. Titrate with the 0.153N Silver Nitrate solution to a definite light pink colored endpoint.
5. Calculate the total Nickel Chloride:

    a. $NiCl_2 \cdot 6\ H_2O$ (oz/gal) = (ml $AgNO_3$ used) x 1.23 b
    b. $NiCl_2 \cdot 6\ H_2O$ (g/L) = $NiCl_2 \cdot 6\ H_2O$ (oz/gal) x 7.5

DETERMINATION OF NICKEL SULFATE

REAGENTS: None required

APPARATUS: None required

PROCEDURE:

Calculate as follows:

a. Nickel Sulfate (g/L) = [(g/L total Nickel) - (g/L Nickel chloride x .247)] x 4.47

DETERMINATION OF BORIC ACID

REAGENTS: Mixed indicator- Dissolve 0.1 grams of Bromocresol Purple and 0.02 grams of Bromothymol Blue in 100ml methanol.
0.1N Sodium Hydroxide standardized solution
Mannitol

APPARATUS: 2 ml Pipette, volumetric
250 ml Erlenmeyer flask
50 ml Burette

PROCEDURE:

1. Pipette 2 ml of sample into a 250 ml Erlenmeyer flask.
2. Add 2 ml of DI water.
3. Add about 5 drops of Mixed indicator.
4. Add 5 grams of Mannitol.
5. Titrate with the 0.1N Sodium Hydroxide solution to a purple colored endpoint.
6. Calculate the Boric Acid concentration:

    a. Boric Acid (oz/gal) = (ml 0.1N NaOH used) x 0.414
    b. Boric Acid (g/L) = Boric Acid (oz/gal) x 7.5

DETERMINATION OF NiCOL BN Carrier 10

REAGENTS: Deionized or distilled water.
Ethyl Acetate
Methyl Alcohol
Hydrochloric Acid
0.1N Standard Sodium Hydroxide solution
0.1% Bromcresol Purple indicator solution

APPARATUS: 50 mL Pipette, volumetric.
300 mL Erlenmeyer flask
12 mL Separatory Funnel
50 mL Burette

PROCEDURE:

1. Pipette a 50 mL sample of plating solution into the separatory funnel.
2. Add 1.0 mL of Hydrochloric acid.
3. Add 50 mL of Ethyl Acetate to the separatory funnel. Shake vigorously for 1-2 minutes. Allow to separate for 1-2 minutes.
4. After separation drain off the lower layer and discard solution.
5. Rinse side of separatory funnel with 25 mL D.I. water. Allow separation to form and drain off lower layer again.
6. Repeat step #4 three to four times.
7. Transfer remaining Ethyl Acetate layer to 300 mL Erlenmeyer flask.
8. Add 10 mL Methyl Alcohol.
9. Add 15 drops 0.1% Bromcresol Purple indicator solution.
10. Calculate the NiCOL BN Carrier 10 as follows:

    a. NiCOL BN Carrier 10 (%/vol) = mL of 0.1N NaOH x 0.416
    b. %/vol x 10 = mL/L

DETERMINATION OF NiCOL BN Index 00

REAGENTS: 0.100 N Standard Sodium Thiosulfate solution.
0.100 N Standard Potassium Bromate/Bromide solution.
1:1 Sulfuric acid solution
10% w/v Potassium Iodide solution
1% Starch Indicator solution

APPARATUS: 10 mL Pipette, volumetric.
50 mL Graduated cylinder.
5 mL Graduated cylinder
250 mL Iodine flask.

PROCEDURE:

1. Pipette 10.00 mL of plating solution into a 250 mL Iodine flask.
2. Add 50 mL DI or distilled water and stir to mix.
3. Pipet 10.00 mL of 0.100 N Potassium Bromate/Bromide solution into the flask and mix again.
4. Add 10 mL of 1:1 Sulfuric acid and immediately stopper and seal with water. Mix thoroughly.
5. Allow to stand for 10 minutes.
6. Add 5 mL 10 % Potassium Iodide solution and immediately titrate with 0.100 N Sodium Thiosulfate solution.
7. With mixing, begin titrating with the thiosulfate solution until the solution turns a light-yellow color. Add 1-2 mL of starch solution and continue the titration until the dark blue starch/iodine color disappears for at least 30 seconds. Record this titration as “B”.
8. Run a blank using 10.00 mL DI water or distilled water in place of the plating solution. Record this titration as “A”.
9. Calculate the BN-Index 00 as follows:

    a. BN Index 00 (%/vol) = (mL A - mL B) X 0.36
    b. %/vol X 10 = mL/L

DETERMINATION OF SURFACE TENSION

APPARATUS: Stalagmometer: Available from Kocour

PROCEDURE:

1. The NiCOL Wetting agent concentrations can be determined by checking the surface tension of the solution. The stalagmometer number of drops delivered for a certain volume is determined by the specific gravity, surface tension, and the specific gravity of the solution.
2. The stalagmometer will supply directions with the instrument that should be followed.
3. Standards should be made with each stalagmometer using a plating solution that has no wetting agent.
4. Standards should be made at 0.1, 2.0, 3.0, 4.0, and 5 mL/L to prepare a concentration versus surface tension graph.
5. Take an average of three readings for each standard.
6. Calculate surface tension as:

$$\text{Surface Tension (Dynes /cm)} = \frac{SW \times NW \times D}{N \times DW}$$

D= Density of the Sample in grams/ml
DW= Density of the water in grams/ml
N= Counted number of drops of the sample
NW= water number engraved on the stalagmometer.
SW= Surface tension of the water (72.0 dynes/cm)

TROUBLESHOOTING GUIDE

PROBLEM	POSSIBLE CAUSE	CORRECTIVE ACTION
Dull deposits (LCD area)	1. Excessive BN Brightener 30 2. Metallic contamination (eg, Cu, Zn, Pb, Cd) 3. Low BN Carrier 10	1. "Dummy" solution at 4-5 ASF 2. "Dummy" solution at 4-5 ASF 3. Add BN Carrier 10
Cloudy deposits	1. Low BN Brightener 30 2. pH too low 3. Organic contamination 4. Metallic contamination (eg, Fe, Si, Al, Cr^{+3})	1. Add BN Brightener 30 2. Raise the pH 3. Carbon treat 4. High pH treat solution
High consumption of carrier	1. Drag-out is high 2. High carbon usage 3. Very high nickel concentration	1. Reduce drag-out 2. Reduce amount of carbon used 3. Reduce nickel concentration
Poor ductility	1. Excessive BN Brightener 30 2. Low BN Carrier 10 3. High pH 4. Metallic contamination (eg, Zn, Cd) 5. Organic contamination	1. “Dummy” solution at 4-5 ASF 2. Add BN Carrier 10 3. Adjust pH 4. “Dummy” solution at 4-5 ASF 5. Carbon treatment

Poor leveling	1. Low BN Brightener 30 2. Low BN Carrier 10 3. Low pH 4. Low BN Index 00	1. Add BN Brightener 30 2. Add BN Carrier 10 3. Adjust pH 4. Add BN Index 00
Burning	1. Low nickel salts/boric acid 2. High CD 3. Low temperature 4. Low agitation 5. Passivate contamination 6. Metallic contamination	1. Add nickel salts/boric acid 2. Reduce CD 3. Adjust temperature 4. Increase agitation 5. High CD "dummy" + high pH treatment 6. High pH + carbon treatment
Skip plating	1. High BN Brightener 30 2. Metallic contamination (eg, Pb, Zn, Cd)	1. "Dummy" solution at 4-5 ASF 2. “Dummy" solution at 4-5 ASF
Poor chromium acceptance	1. High BN Brightener 30 2. Low BN Carrier 10	1. “Dummy” solution at 4-5 ASF 2. Add BN Carrier

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.