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MARQUEE HP

TECHNICAL DATA
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MARQUEE HP

PROCESS FOR HIGH PHOSPHOROUS ELECTROLESS NICKEL PLATING
CUSTOMER SUPPLIED LIQUID NICKEL SULFATE

MARQUEE HP	has excellent stability.
MARQUEE HP	meets ASTM-B-733 and AMS-2404 C specifications.
MARQUEE HP	is a simple two component process.
MARQUEE HP	passes the nitric acid test.
MARQUEE HP	provides excellent corrosion resistance.
MARQUEE HP	produces a phosphorous (10.5 - 12%) nickel alloy.

OPERATING PARAMETERS

Nickel Metal	0.7 - 0.8 oz/gal (5.25 - 6 g/L)
Operating Temperature	190º - 197º F (88º - 92º C)
Ph	4.3 - 4.8 (4.6 optimum)
Bath Loading	0.20 - 1.0 ft^2/gal (0.5 - 2.50 sq.dm/L)
Typical Plating Rate	12.7 micron/hr. (0.5 mil/hr.)

SOLUTION MAKEUP

Liquid Nickel Sulfate EN grade (5#/gallon as Nickel Sulfate)	4.5 %/vol
MARQUEE HP-B-21	15.0 %/vol
MARQUEE HP-C-21	Not required for make-up

When making a new solution it is important to follow these steps. Taking shortcuts can increase the cost of your electroless nickel process.

1. Remove the old operating solution.

2. Remove the old filter bags or cartridge filters; if necessary, re-seal the filter unit. Fill the tank with 50%/vol. Nitric Acid.

3. Circulate the solution through the pumps and filter housings. Turn off air agitation. Allow the solution to contact all the surfaces that it would contact when in operation. Allow the solution to stand overnight to completely strip and passivate the tank.

4. Remove the Nitric Acid solution from the tank.

5. Rinse the tank/pumps and filter housings thoroughly.

6. Fill the tank with water higher than the level of the nitric solution and add approximately 1-liter Ammonium Hydroxide per 400 liters of volume. Allow this solution to circulate for 1 hour. While the solution is circulating, turn the agitation on and off. The Ammonium Hydroxide will neutralize the Nitric Acid.

7. Drain the system then rinse with clean water. Test the rinse water for nitrates (Fisher Scientific carries M-10020 EM quant test strips for testing for nitrates).

8. Install new filter bags or cartridges.

9. Equipment is now ready for a new operating solution.

10. Fill tank to half its volume with distilled or D.I. water.

11. Add the required amount of Liquid Nickel Sulfate and mix well.

12. Add the required amount of MARQUEE HP-B-21 and mix well.

13. Add distilled or D.I. water to volume and mix well.

14. Check pH and adjust to a pH of 4.8. Use dilute 1:1 Ammonium Hydroxide or Potassium Hydroxide to raise the pH. Use 20%/volume Sulfuric Acid to lower the pH.

EQUIPMENT

Tanks:	If the tank size is less than 500 gallons then tanks made of high density, natural, unpigmented, polypropylene is suggested. It is a good idea to have two tanks set-up so a spare tank is available if there is nickel build-up on the heaters or tank. Contact your Columbia Chemical representative for recommendations.
Heat:	316 Stainless Steel heat exchanger, PTFE steam coils, or electric immersion heaters fabricated from PTFE or 316 stainless steel is recommended.
Agitation:	Clean, mild air agitation from a low-pressure blower is recommended. Other means of agitation may be used such as mechanical or reciprocal.

MAINTENANCE ADDITIONS

MARQUEE HP-C-21 AND LIQUID NICKEL SULFATE

See Replenishment Schedule below.

Nickel Content by Analysis	% Nickel Activity	MARQUEE HP-C-21 to add	Liquid Nickel Sulfate to add
6.0 g/L (0.80 oz/gal) -	100%	0 mL/L (0.0 fl. oz/gal)	0 mL/L (0.0 fl. oz/gal)
5.7 g/L (0.76 oz/gal) -	95%	3 mL/L (0.38 fl. oz/gal)	2.25 mL/L (0.29 fl. oz/gal)
5.4 g/L (0.72 oz/gal) -	90%	6 mL/L (0.76 fl. oz/gal)	4.5 mL/L (0.57 fl. oz/gal)
5.1 g/L (0.68 oz/gal) -	85%	9 mL/L (1.15 fl. oz/gal)	6.75 mL/L (0.86 fl. oz/gal)
4.8 g/L (0.64 oz/gal) -	80%	12 mL/L (1.54 fl. oz/gal)	9 mL/L (1.16 fl. oz/gal)
4.5 g/L (0.60 oz/gal) -	75%	15 mL/L (1.92 fl. oz/gal)	11.25 mL/L (1.44 fl. oz/gal)
4.2 g/L (0.56 oz/gal) -	70%	18 mL/L (2.30 fl. oz/gal)	13.5 mL/L (1.73 fl. oz/gal)

Regular analysis of the nickel content should be the basis for additions of the Liquid Nickel Sulfate and the MARQUEE HP-C-21. Add back equivalent amounts of the MARQUEE HP-A-21 and MARQUEE HP-C-21 as needed from the chart.

For example: If the nickel metal analysis is 5.7g/L (0.76 oz/gallon) then add back 2.25mL/L (0.29 fl. oz/gal) Liquid nickel Sulfate and 3 mL/L (0.38 fl. oz/gal) MARQUEE HP-C-21.

Measure the pH after all other adjustment have been made. Make adjustments using dilute 1:1 Ammonium Hydroxide or Potassium Hydroxide to raise the pH. Use 20%/volume Sulfuric Acid to lower the pH.

pH

Use a calibrated pH meter to check the pH. Make measurements with the sample cooled to room temperature. The solution pH should be checked regularly to insure it is maintained within the operating parameters.

To raise the pH use 1:1 dilute ammonium hydroxide or potassium hydroxide. Use 20%/vol. Sulfuric Acid to lower the pH

Temperature

Maintain the temperature of the plating bath within specified limits. Derated heaters (low watt density) are highly recommended to maintain the solution temperature of the bath. High temperatures can spontaneously decompose the bath. Low temperatures will slow the plating rate of the bath.

ANALYTICAL PROCEDURE

DETERMINATION OF NICKEL METAL

REAGENTS: Standardized 0.1 M or 0.0575 M EDTA
Concentrated ammonia hydroxide
Deionized water
Murexide indicator – thoroughly mix approximately 0.25 grams murexide with 95 grams of table sugar.

PROCEDURE:

1. Pipette 5 mL bath sample into a 250 mL Erlenmeyer flask.
2. Add approximately 100 mL Deionized water.
3. Add 10 mL of concentrated ammonium hydroxide.
4. Add 0.2 grams murexide indicator.
5. Titrate immediately with the 0.1 or 0.0575 M standardized EDTA to a magenta endpoint.
6. Calculate the nickel metal content:

    a. Nickel metal (g/L) = mL 0.0575 M EDTA x 0.674 or mL 0.1 M EDTA x 1.174
    b. Nickel metal (oz/gal) = mL 0.0575 M EDTA x 0.090 or mL 0.1 M EDTA x 0.1565

HELPFUL HINTS

NICKEL DEPOSIT PROPERTIES

Phosphorous Content	10.5 - 12% (varies depending on operating pH and bath age)
Hardness	500 - 700 (VHN) as plated, 900 – 1,000 (VHN) heat treated
Appearance	Semi bright deposit
Density	Approximately 7.9 g/cc

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.

TROUBLESHOOTING GUIDE

PROBLEM	**CAUSE**	**SOLUTION**
Poor adhesion to substrate	Poor cleaning prior to plating	Improve the cleaning process
	Organic contamination	Dump bath
	Metallic contamination	Dummy plate
Bath plate out	Temperature too high	Lower the temperature
	pH too high	Lower the pH
	Additives out of balance	Adjust additives
	Nickel build-up on equipment	Strip and passivate tank
Slow plating rate	Temperature is too low	Increase the temperature
	pH is too low	Increase the pH
	Additives out of balance	Adjust additives
	Metallic contamination	Dummy plate
No deposit	Temperature is too low	Increase the temperature
	pH is too low	Increase the pH
	Metallic contamination	Try to dummy plate
	Organic contamination	Dump bath
	Additives out of balance	Adjust additives
Pitting	Low agitation	Increase agitation
	Organic contamination	Dump bath
	Metallic contamination	Dummy plate
Dull deposit	pH is too low	Increase the pH
	Organic contamination	Dump bath
	Metallic impurities	Dummy plate
	Additives out of balance	Adjust additives
Rough deposit	pH is too high	Lower the pH
	Particulate in bath	Improve filtration
	Additives out of balance	Adjust additives
Dark deposit	Metallic contamination	Dummy plate
	Organic impurities	Dump bath
Streaks and/or patterns	Metallic contamination	Dummy plate
	Organic contamination	Dump bath
	Low surface area	Increase surface area
Blisters	Poor cleaning prior to plating	Improve the cleaning process
	Metallic contamination	Dummy plate
	Organic contamination	Dump bath
	Temperature is too high	Lower the temperature