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TRIVECTA® BLACK 300

TRIVALENT BLACK LIQUID CHROMATE
TECHNICAL DATA
05-31-13

TRIVECTA® BLACK 300

TRIVECTA® BLACK 300	produces a uniform gloss-black, scratch-resistant finish.
TRIVECTA® BLACK 300	is a thick film (>400nm), trivalent chromate conversion coating for zinc and zinc alloy deposits.
TRIVECTA® BLACK 300	provides easy start-ups, is easy to use and has no break-in period.
TRIVECTA® BLACK 300	works equally well over acid chloride zinc (both ammoniated and non-ammoniated systems), cyanide zinc, alkaline cyanide zinc free and zinc-iron/cobalt alloy deposits.
TRIVECTA® BLACK 300	has excellent stability, does not degrade or precipitate during hard production runs or long shut-down periods.
TRIVECTA® BLACK 300	is tolerant to high levels of zinc contamination and is not affected by high concentration of iron contamination.
TRIVECTA® BLACK 300	yields a finish that consistently exceeds 120+ hours of neutral salt spray to first white rust.

SOLUTION MAKEUP: CHROMATE

TRIVECTA CHROMATE A-310	15% - 20% by volume
TRIVECTA CHROMATE B-320	0.4% by volume
TRIVECTA CHROMATE C-330	0.2% by volume
pH	2.0 - 2.6 (Optimum 2.3)
Temperature	75° - 85°F/24° - 29°C (Optimum 80°F/26°C)
Dip Time	30 to 60 seconds
Agitation	Mechanical or circulating pump Air agitation is NOT recommended for extended production periods and rack applications

SOLUTION MAKEUP: TRIVECTA SEALER 350

TRIVECTA SEALER 350	Rack - 12% - 15% by volume (Optimum 15% by volume) Barrel - 20% - 25% by volume (Optimum 20% by volume)
pH	4.4 - 5.0 (optimum 4.8) To raise the pH use Caustic Soda Liquid 50% To lower the pH use Sulfuric Acid or Hydrochloric Acid
Temperature	115° - 125°F / 46° - 52°C (Optimum 120°F/48°C)
Dip Time	20 - 30 seconds
Agitation	Recommended (mechanical or circulating pump Note: Turn agitation off when not in use

SOLUTION MAINTENANCE: CHROMATE

To maintain optimum appearance and performance of the TRIVECTA BLACK 300 CHROMATE, the following tests are recommended once per 8 hour shift.

1. **pH**, If necessary, adjust within optimum. (Optimum 2.0 to 2.6)

   High pH - slowly adjust with Sulfuric Acid or Nitric Acid within optimum range.
   Low pH - slowly adjust with Caustic Soda Liquid 50% within optimum range.

2. **Temperature**, Maintain temperature at the optimum of 75° to 85°F/24° to 29°C.

   High temperature - If possible, cool to within optimum range.
   Low temperature - Heat to optimum.

3. **Iron (Fe) and Chrome (Cr)**, Using Atomic Absorption or X-Ray Fluorescence analysis, determine content of iron (Fe) and chrome (Cr) in g/L or mg/L. See Chromate Analytical Procedure.

   Iron - Optimum range is 3,200 to 4,000 mg/L.
   Chrome - Optimum range is 2,200 to 2,400 mg/L.
   Iron to Chrome ratio (Fe:Cr Ratio) - Optimum ratio 1.2 to 1.5 : 1.

   Adjusting Iron to Chrome Ratio

   High Fe:Cr Ratio (1.6 - 2.0:1), Add 0.2% to 0.4% by volume TRIVECTA CHROMATE B-320.

   An addition of 0.1% by volume TRIVECTA CHROMATE B-320 will increase chrome content 80 mg/L.

   Low Fe:Cr Ratio (0.8 - 1.1:1), Add 1% to 5% by volume TRIVECTA CHROMATE A-310.

   An addition of 1.0% by volume TRIVECTA CHROMATE A-310 will increase chrome content 125 mg/L and iron content 220 mg/L.

   An iron to chromate ratio below 0.8:1 may require a large bath decant/dilution or complete dump of working solution. Excessive use of air agitation or poor routine maintenance addition practices can result in an extremely low iron to chrome ratio.

   NOTE: IRON CONCENTRATION WILL BE USED TO DETERMINE TRIVECTA CHROMATE A-310 CONTENT IN THE WORKING SOLUTION.

4. **Zinc (Zn) Contamination**, Using Atomic Absorption or X-Ray Fluorescence, determine content of zinc (Zn) contamination in g/L or mg/L. The maximum zinc contamination level is 15,000 mg/L or 15 g/L. When zinc contamination is equal to or greater than the maximum allowable limit, a 50% by volume solution cut or bath dump is recommended. A 50% bath cut will require the following actions.

   TRIVECTA CHROMATE A-310 - Determine volume to be added back to the cut solution by following the Chromate Analytical Procedure.
   TRIVECTA CHROMATE B-320 - 0.2% by volume
   TRIVECTA CHROMATE C-330 - 0.1% by volume

SOLUTION MAINTENANCE: SEALER

To maintain optimum appearance and performance of the TRIVECTA SEALER 350, the following tests are recommended once per 8 hour shift.

1. **pH**, If necessary adjust within optimum. (Optimum 4.4 to 5.0)
   High pH - slowly adjust with Sulfuric Acid within optimum range.
   Low pH - slowly adjust with Caustic Soda Liquid 50% within optimum range.

2. **Temperature**, Maintain temperature at optimum of 115° to 125°F/46° to 52°C.
   High temperature - If possible, cool to within optimum range.
   Low temperature - Heat to optimum.

3. **Chrome (Cr)**, Using Atomic Absorption or X-Ray Fluorescence, determine content of chrome (Cr) in g/L or mg/L. Follow the Sealer Analytical Procedure. Optimum range is 3,500 to 5,800 mg/L (12 to 20%).

   Adjusting TRIVECTA SEALER 350 Working Solution Concentration
   See TRIVECTA SEALER 350 Analytical Procedure and add back the required amount.

PRODUCT DESCRIPTION

TRIVECTA CHROMATE A-310	Provides base Trivalent Black Chromate film.
TRIVECTA CHROMATE B-320	Improves film hardness/uniformity and increases overall chromate film thickness.
TRIVECTA CHROMATE C-330	Improves blackening and uniformity of chromate film.
TRIVECTA SEALER 350	Increases chromate film hardness, gloss, scratch resistance and corrosion protection.

ADDITIONAL OPERATING INSTRUCTIONS

NITRIC ACID PRE-DIP

A nitric acid pre-dip of 0.2% to 0.4% by volume is recommended prior to the working TRIVECTA BLACK 300 chromate to polish the zinc deposit and to extend the life of the chromate solution.

RINSING

Optimum rinsing should be used between the TRIVECTA BLACK 300 and TRIVECTA SEALER 350 processes.

AGITATION

Maintain good "mechanical" agitation in the TRIVECTA BLACK 300 chromate to ensure good solution flow over the parts. Good solution flow is essential for optimum black color and film growth. Agitation is not recommended for the TRIVECTA SEALER 350 in rack applications. Shorter dip times and/or reduced barrel rotation are recommended for barrel applications. Air agitation is NOT recommended.

ALKALINE ZINC PLATED PART

Alkaline zinc plated parts may darken and turn black more quickly than acid chloride plated parts. Shorter dip times or lower concentrations of the TRIVECTA CHROMATE A-310 may be required for optimum black appearance.

ACID CHLORIDE ZINC PLATED PARTS

Acid chloride zinc plated parts may take longer to turn black than alkaline zinc plated parts. Higher concentrations of the TRIVECTA CHROMATE C-330 may be required for optimum black appearance. Incremental additions of 0.025% by volume TRIVECTA CHROMATE C-330 are recommended until black color is achieved. DO NOT INCREASE THE TOTAL TRIVECTA CHROMATE C-330 BY MORE THAN 0.2% BY VOLUME.

ANALYTICAL PROCEDURE: TRIVECTA CHROMATE A-310

The use of X-Ray Fluorescence or Atomic Absorption analysis is recommended to determine the working concentration of TRIVECTA CHROMATE A-310. TRIVECTA CHROMATE A-310 is the main additive used during day to day production, and the only product containing solubilized iron in the TRIVECTA BLACK 300 process.

Procedure

Using standard X-Ray Fluorescence or Atomic Absorption techniques, determine g/L of iron in the working chromate solution.

CALCULATION: Atomic Absorption Only

g/L iron x 4.8 = % TRIVECTA CHROMATE A-310

ANALYTICAL PROCEDURE: TRIVECTA SEALER 350

TITRATION PROCEDURE

Items Needed

1. DI Water
2. 20% Sodium Hydroxide Solution
3. 35% Hyrdrogen Peroxide Solution
4. 10% Nickel Chloride Solution
5. Conc. Hydrochloric Acid
6. Ammonium Bifluoride
7. 10% Potassium Iodide Solution
8. Starch Indicator Solution
9. 0.010N Sodium Thiosulfate Solution

Procedure

1. Pipette a 10 ml sample of working chromate into a 100 ml volumetric flask. Dilute to 100 ml with DI water.

2. Pipette 10 ml of the above diluted solution into a 250 ml Erlenmeyer flask and dilute to approx. 100 ml with DI water.

3. Add 5 ml of 20% Sodium Hydroxide solution and mix.

4. Add 1 ml of 35% Hydrogen Peroxide solution and mix.

5. Place solution on a hot plate and with mixing heat until boiling. Boil for 5 minutes.

6. Add 1 ml of 10% Nickel Chloride solution to the flask and continue boiling for an additional 2 minutes.

7. Remove solution from hot plate and cool to room temperature.

8. With mixing add the following in order; 10 ml of concentrated Hydrochloric Acid, 1 gram Ammonium Bifluoride, 10 ml Potassium Iodide, and 1 ml Starch Indicator.

9. Titrate solution with 0.010N Sodium Thiosulfate to a clear/slight greenish endpoint.

CALCULATION: mL of 0.010N Sodium Thiosulfate x 0.62 = % TRIVECTA SEALER 350

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.

TROUBLE SHOOTING GUIDE

FOR TRIVECTA BLACK 300

Important note: Prior to using this Trouble Shooting Guide, optimize the Trivecta Chromate 300 and Trivecta Sealer 350 solutions. Refer to Solution Maintenance section on pages 2-3.

Poor Color, Grey or uneven colored parts, No blackening

Step 1: Measure zinc deposit thickness to verify the part has a minimum of 0.3 mils / 8 microns. If thickness is below 0.3 mils / 8 microns, increase plating time.

Step 2: Increase chromate activity by adding 1% by volume A-310 and 0.2% by volume C-330.

Parts turn black too quickly

Step 1: Chromate solution is too active, raise pH to high end of the pH range (2.4) with Caustic Soda Liquid 50%.

Step 2: Chromate solution is too active, add 0.2% by volume Chromate B-320.

Step 3: Operate and maintain chromate solution at lower of temperature range (70°F/22°C)

Parts are smutty, dull, or easily scratched

Step 1: Reduce dip time to 30 seconds

Step 2: Raise pH with Caustic Soda Liquid 50% to high end of pH range (2.5).

Step 3: Chromate solution is too active, add 0.2% by volume Chromate B-320.

Step 4: Increase the Sealer to the high end of the concentration range (Barrel-25%, Rack 15%)

Poor Sealer adhesion, voids, uneven coverage

Step 1: Increase the Sealer temperature to the high end range (125°F/48°C)

Step 2: Raise pH with Caustic Soda Liquid 50% to high end of pH range (4.8).

Step 3: Optimize the chromate film- See **Parts are smutty, dull, or easily scratched** above.

Step 4: For barrel parts, spin drying may be too abrasive. Allow parts to heat for 30-60 seconds prior to turning on the spin dryer.

Sealer 350 leaves drip marks

Step 1: Reduce the Sealer concentration (Barrel-15%, Rack-10%)

Step 2: Reduce temperature of the dryers and increase the drying time..

Step 3: Use an air wand or compressed air to remove excess sealer that collects on part edges or recesses.

Step 4: Optimize the chromate film- See **Parts are smutty, dull, or easily scratched** above.

Finished parts are iridescent

Step 1: Optimize the chromate film- See **Parts are smutty, dull, or easily scratched** above.

Step 2: Increase the Sealer 350 to the high end of the concentration range (Barrel-25%, Rack 15%)

Step 3: For barrel parts, spin drying may be too abrasive. Allow parts to heat for 30-60 seconds prior to turning on the spin dryer.

Parts do not turn black after long shut-down period

Step 1: Activate the Chromate, add 1% by volume A-310, 0.1% - 0.2% by volume B-320, and 0.1% - 0.2% by volume C-330.

Step 2: Increase the Sealer temperature to the high end range (125°F/48°C)

Mold growth in the TRIVECTA SEALER 350

Step 1: With good agitation, treat working sealer solution with 0.1% to 0.2% by volume commercial grade sodium hypochlorite (12% Active) to oxidize/destroy mold growth.