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TriCOL™ Décor

TECHNICAL DATA
 02-11-20

TriCOL™ Décor

PROCESS FOR WHITE TRIVALENT CHROMIUM PLATING

- TriCOL Décor has excellent metal distribution across the entire current density plating range.
- TriCOL Décor is a state of the art, newest generation of trivalent chromium process that provides the deepest-lightest-whitest appearance.
- TriCOL Décor eliminates burning and whitewash associated with hexavalent processes.
- TriCOL Décor greatly reduces waste treatment costs.
- TriCOL Décor has excellent plating speed and allows easy conversion from existing hexavalent processes.

OPERATING PARAMETERS

	<u>RANGE</u>	<u>OPTIMUM</u>
Temperature:	27 - 43°C (80 - 110°F)	29 - 32°C (85 - 90°F)
pH:	2.3 - 2.9	2.6 - 2.8
Current Density:	8.5 - 13.4 Amps/dm ² (80 - 125 Amps/ft ²)	
TriCOL T-SALTS 100:	390 - 460 g/l (52 - 61.3 oz/gal)	415 g/l (55.3 oz/gal)
TriCOL REPLENISHER 300:	50 - 70 ml/l (5.0-7.0% by vol.)	60 ml/l (6.0% by vol.)
TriCOL ANTIMIST 400:	1.0 - 4 ml/l (0.1-0.4% by vol.)	1.5 ml/l (0.15% by vol.)
TriCOL CD PLUS 500:	2.0 ml/l (0.2% by vol.)	2.0 ml/l (0.2% by vol.)
Chromium (Cr+3):	20 - 23 g/l (2.7 - 3.1 oz/gal)	21 g/l (2.8 oz/gal)
Specific Gravity:	1.200*	
Agitation:	Air through low pressure blower recommended.	
Anode to Cathode Ratio:	1.5:1 to 2.0:1	

Current – DC:	Less than 10% ripple
Voltage:	6 - 15 volts
Deposition Rate:	Approximately 0.15 - 0.25 microns at 10.8 Amps/dm ² (6-10 micro inches/min at 100 Amps/ft ²)
Filtration:	Usually not required, proper Ion Exchange equipment is needed.

* - New bath specific gravity will start near this value, over years of use value may increase if low drag-out.

SOLUTION MAKEUP

	<u>100 LITERS</u>	<u>100 GALLONS</u>
TriCOL T-SALTS 100:	41.5 kg	346 pounds
TriCOL REPLENISHER 300:	6.0 Liters	6.0 gallons
TriCOL ANTIMIST 400:	150 milliliters	0.15 gallon
TriCOL CD PLUS 500:	200 milliliters	0.2 gallon

PREPARATION:

1. To a clean plating tank install the special graphite anodes and titanium hangers. It is recommended that the buss bars be heavy nickel plated before installation. After the titanium hangers are installed to the buss bar, wrap the bussing with plastic tape or cover with appropriate inert material to minimize copper contamination from the copper bussing.
2. Install the heating and cooling coils. If the cooling coils are titanium, connect the coils through some high impedance wire so they are anodically protected.
3. Regenerate the Ion Exchange resin and then connect the system to the plating tank.
4. Fill tank full with water.
 - o Check agitation to insure it is uniform.
 - o Turn on the Ion Exchange system and check flow rate then turn off, and turn flow valves off.
 - o Check heating and see what the heat up time of solution to 60 degrees Celsius is. Then turn on cooling if equipped to see if it works properly.
 - o Lower pH to 2.5 with hydrochloric acid and add 1ml/Liter of the TriCOL ANTIMIST 400.
 - o Allow solution to stand for eight hours at operating temperature of 32°C. This will leach the equipment and tank. Run the Ion Exchange system for 1 hour.
 - o Pump out the leach solution, drain the Ion Exchange system, and rinse the tank out.
5. Fill the plating tank ½ full with city water (D.I. water should be used if available).
6. Turn on the heating and air agitation and heat the solution to 60 - 65°C (140 - 150°F).
7. Using air agitation, slowly add 415 g/Liter of the TriCOL T-SALTS 100. Solution temperature will drop as the salts are added. Bring the temperature back up to 60 - 65°C (140 - 150°F). If the material is added too fast it may settle to the bottom and not dissolve properly. Check bottom of

tank to insure all the material is dissolved before proceeding to the next step. Usually it takes 2 hours at the high temperature to completely dissolve all of the TriCOL T-SALTS 100.

8. Add 60 ml/Liter (6.0%/vol) TriCOL REPLENISHER 300 then bring tank volume up to operating level and maintain heat for 4 hours.
9. Allow solution to cool to operating temperature.
10. Add 1.5 ml/Liter of the TriCOL ANTIMIST 400, then add 2 ml/Liter of the TriCOL CD PLUS 500.
11. Allow solution to mix for 10 minutes then check pH of the solution. Adjust if necessary.
12. Check the concentration of the chromium metal, and the TriCOL REPLENISHER 300. Adjust if necessary.
13. The solution will be ready to plate parts at this point.
14. If there are any HCD or LCD defects noted then the solution will need to be dummy plated for a short time. If dummy plating is needed then heavy nickel plate a steel corrugated panel and put enough area in the tank to equal approximately 25 – 50 m²/1000 liter or 1 - 2 ft²/100 gallons. Dummy plate at 9.7 Amps/dm² or 90 Amps/ft² for 1 hour, then plate parts to see if all defects have been removed.

RECOMMENDED EQUIPMENT

Tank or Tank Liner:	New PVC, polypropylene, polyethylene, ABS or Koroseal-lined steel.
Heaters/Cooling Coils:	Quartz, PTFE, and titanium (must be grounded or anodic).
Heaters/Chillers:	Quartz, PTFE, and titanium (must be grounded or anodic).
Racks:	Plastisol-coated copper.
Anodes:	Use TriCOL Anodes. Consult Columbia Chemical for recommendations.
Anode Hangers:	Use TriCOL Anode Hangers. Consult Columbia Chemical for recommendations.
Bus Bars:	Copper bus that is heavily nickel plated to avoid possible copper contamination. Bus bars that are exposed should be protected from solution by a plastic shield or by wrapping with plastic waterproof tape.
Ion Exchange:	Use Columbia TriCOL PURE Ion Exchange Systems.
Ion Exchange Resin:	Use Columbia TriCOL PURE Resin.

EQUIPMENT

TriCOL ANODES

Graphite anodes from Columbia Chemical must be used. Anode length should be approx. 2.5 - 3.75 cm (1 - 1.5 inches) shorter than the rack package at each end when measured from the top of the top

piece on the rack to the bottom of the bottom piece on the rack. The anodes should also be at least 5 cm (2 inches) below the solution level.

Generally, twice the area of anode area to cathode area should be used. The maximum current density on the anodes should be 540 amperes/m² (50 amperes/ft²). The graphite anode must be mounted below the surface of the solution. The copper bussing should be plated with a layer of nickel prior to installation; this will minimize the amount of copper contamination. Once the hangers are mounted to the buss bars, the buss bar should be wrapped with waterproof plastic tape or covered with other inert plastic material.

TriCOL ANODE HANGERS

Anode hangers are available from Columbia Chemical.

AIR AGITATION

Air agitation should be as mild and uniform as possible. High agitation can result in reduced coverage while low agitation can result in uneven metal distribution. Air must be provided from an oil free blower. Perforated plastic air lines must be installed to give uniform mild agitation below the cathode area.

AMPERE-HOUR METER

Product additions to the solution are made by ampere-hours. A suitable ampere-hour meter should be used to ensure proper control of the process. An automatic metering pump for the TriCOL REPLENISHER 300 is usually recommended.

RECTIFIERS

12- or 15-volt rectifiers are normally recommended however existing 6 - 9-volt rectifiers have been used in some installations. The TriCOL trivalent chromium process will not burn therefore higher voltages are normally used to provide superior covering power when compared to an existing hexavalent process.

TriCOL PURE ION EXCHANGE SYSTEMS

Ion Exchange equipment is available from Columbia Chemical. Consult your Columbia Chemical representative for proper sizing of equipment.

TriCOL PURE ION EXCHANGE RESIN

Obtain the special Ion Exchange resin from Columbia Chemical. Consult Columbia Chemical for specific volume recommendations.

MAINTENANCE ADDITIONS

TriCOL T-SALTS 100

TriCOL T-SALTS 100 is used for make-up and replenishment of the solution from drag-out. It provides conductivity to the bath. Additions of TriCOL T-SALTS 100 should be made slowly to minimize

undissolved salts in the bath. Additions should be made in several smaller increments if the additions are over 30 g/Liter (4 oz/gallon). Large additions can also be made by simply heating the solution after the addition. The heating will properly complex the chromium whenever very large additions are required.

The addition of 21 g/Liter (2.8 oz/gallon) of the TriCOL T-SALTS 100 will raise the specific gravity 0.01 units. Make the additions of TriCOL T-SALTS 100 prior to adding the TriCOL REPLENISHER 200.

High concentration of TriCOL T-SALTS 100 can result in crystallization if the solution temperature is too low.

Low concentration of TriCOL T-Salts will result in lower conductivity, and may require use of higher voltages to maintain the same current density.

TriCOL REPLENISHER 200

TriCOL REPLENISHER 200 is replenished on an ampere-hour basis and replaces the chromium that is plated out of solution. TriCOL REPLENISHER 200 is added at a rate of approximately 435 g/1000 ampere-hours (15.3 oz/1000 ampere-hours) and should be added at least once every 3.5 ampere-hours/Liter (13 ampere-hours/gallon) of operation.

Additions larger than 7.0 g/Liter (0.93 oz/gallon) should be made in several small increments. If a very large addition must be made then it may be helpful to heat the solution after the addition to properly complex the chromium.

TriCOL REPLENISHER 300

TriCOL REPLENISHER 300 allows the chromium to be plated out of solution. TriCOL REPLENISHER 300 is consumed by drag-out and electrolysis. High TriCOL REPLENISHER 300 can result in some precipitation of the salts. Low TriCOL REPLENISHER 300 can result in reduced plating rate. Maintain the concentration of the TriCOL REPLENISHER 300 within the specified ranges. TriCOL REPLENISHER 300 should be added at 1.52 liters (51 fl. oz.) per 1000 ampere-hours and should be added at least once every 4.5 ampere-hours/Liter (17 ampere-hours/gallon) of operation.

TriCOL ANTIMIST 400

TriCOL ANTIMIST 400 reduces the surface tension of the solution and improves the metal distribution of the deposit. Low concentrations can result in dark streaks. High concentrations can result in excessive foaming during electrolysis. TriCOL ANTIMIST 400 should be added at 18 - 36 ml/1000 ampere-hours (0.6 - 1.2 fl. oz./1000 ampere-hours) and should be added at least once every 4 ampere-hours/Liter (15 ampere-hours/gallon) of operation. Concentration can be determined by measuring the surface tension.

TriCOL CD PLUS 500

TriCOL CD PLUS 500 improves the current density range of the deposit. TriCOL CD PLUS 500 is added at solution make-up. Further additions should only be made when advised by your Columbia Chemical representative.

TEMPERATURE

The process operates at 27 - 43°C (80 - 110°F), heating will be required. If production is above 0.5 amperes/Liter or if ambient temperature is high, then cooling will be required. High temperature will reduce the covering power. Low temperature can result in precipitation of the salts.

pH

The pH should be maintained between 2.6 and 2.8. Raise the pH 0.1 unit with the addition of 2 ml/Liter (2 gallons/1000 gallons) ammonium hydroxide. Lower the pH 0.1 unit with the addition of 2 ml/Liter (2 gallons/1000 gallons) hydrochloric acid. pH adjustments will produce an artificially large change in pH that will equilibrate after a period of time. It is also recommended that pH buffers of 2.0 and 7.0 be used to calibrate the pH meter that will be used for analyzing the solution.

ANALYTICAL PROCEDURE

TRICOL DÉCOR ANALYSIS PROCEDURES

TRICOL REPLENISHER 300

REAGENTS: 5% Sodium Carbonate solution (Dissolve 50 grams Na_2CO_3 (AR) in DI water and dilute to 1 Liter).
10% w/v Potassium Iodide solution
1:1 Sulfuric acid solution (Dilute concentrated sulfuric acid 1:1 with DI water, use caution when mixing, always add acid to water.)
0.100 N Sodium thiosulfate solution.
0.100 N Potassium permanganate solution.
1% Starch Indicator solution

EQUIPMENT: 1 mL Pipet, volumetric
5 mL Pipet, volumetric
25 mL Pipet, volumetric
250 mL Erlenmeyer flask

PROCEDURE:

1. Pipette 1.0 mL of filtered solution into a 250 mL Erlenmeyer flask. Run a blank using water in place of the plating solution. It is suggested that a blank be run at least monthly until experience dictates otherwise.
2. Add 5 mL of 5% Sodium Carbonate solution.
3. Place the flask in a boiling water bath so that the flask is generally upright and the liquid in the flask is covered by the boiling water. Heat for 15 minutes. After this time, carefully remove the flask and check for the odor of ammonia. All ammonia should be driven off but if an odor of ammonia can still be detected then reheat for another 2-3 minutes. In any event, all ammonia must be removed before continuing.
4. Remove the flask from the water bath and wash down the sides of the flask with 5 mL DI water. Cool to room temperature.
5. Pipet 25 mL of 0.100 N KMnO_4 solution into the flask. Swirl to coat any precipitate on the flask walls with the mixture.

6. Again, place the flask into the boiling water bath for 5 minutes. The purple permanganate should change to brown shortly after heating.
7. Cool to room temperature.
8. Add 5 mL of 10% w/v Potassium Iodide solution.
9. Add 5 mL of 1:1 H₂SO₄ and immediately titrate with 0.100 N Sodium Thiosulfate solution until the solution turns a light yellow. Add 10 drops of starch indicator and titrate to the point where the dark iodine/starch color disappears and the solution is a clear light blue.
10. Calculate as follows:

TriCOL Replenisher 300 = 0.606 X (mL B - mL A) where A = mL 0.1N Sodium Thiosulfate for the sample, B= mL 0.1N Sodium Thiosulfate for blank.

TRIVALENT CHROMIUM

REAGENTS: 30% Hydrogen Peroxide solution, AR (see Note)
50% Sodium Hydroxide Solution
10%w/volume Potassium iodide solution
1:1 Sulfuric acid solution (Dilute concentrated sulfuric acid 1:1 with DI water, use caution when mixing, always add acid to water.)
1% Starch Indicator solution
0.100 N Sodium Thiosulfate solution

EQUIPMENT: 5 mL Pipet, volumetric
100 mL Volumetric flask
250 mL Erlenmeyer flask

PROCEDURE:

1. Pipette 5.0 mL of filtered solution into a 100 mL volumetric flask and dilute to volume.
2. Pipette 5.0 mL of the solution from the volumetric flask directly to the Erlenmeyer flask.
3. Add 6 drops of 30% Hydrogen Peroxide.
4. Add 4 drops of the 50% Sodium Hydroxide solution.
5. Swirl lightly and insert into the boiling water bath. Maintain heat for 10 minutes. All traces of gassing should be removed.
6. Add 5 mL 10% Potassium Iodide solution and mix.
7. Add 10 mL 1:1 Sulfuric acid and immediately titrate with 0.1 N Sodium Thiosulfate solution until the solution turns a light-yellow color.
8. Add 1 mL of 1% Starch Indicator solution and again titrate to the disappearance of the dark blue starch/iodine color.
9. Calculate the concentration as follows:

$$\text{oz/gal Cr}^{+3} = \text{mL } 0.1 \text{ N Na}_2\text{S}_2\text{O}_3 \times 0.904$$

$$\text{g/L Cr}^{+3} = \text{oz/gal} \times 7.5$$

DETERMINATION OF SURFACE TENSION

EQUIPMENT: Stalagmometer: Available from Kocour.

PROCEDURE:

The TriCOL Antimist 400 concentration 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.

The stalagmometer will supply directions with the instrument that should be followed.

Standards should be made with each stalagmometer using a plating solution that has no TriCOL Antimist 400.

Standards should be made at 0.0, 1.0, 2.0 and 3 ml/Liter of TriCOL Antimist 400 to prepare a concentration versus surface tension graph. Take an average of three readings for each standard.

Calculate surface tension as:

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

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)

HELPFUL HINTS

TriCOL plating solutions utilize a weak complex to deposit trivalent chromium. Trivalent chromium solution must be properly maintained to provide the correct amount of complexed chromium. Additions should be made frequently to provide consistent concentration of the constituents.

TRICOL DÉCOR ADDITION AGENT CONSUMPTION

TriCOL plating solutions are consumed by electrolysis and by drag-out. Drag-out affects all the addition agents but electrolysis only affects certain addition agents.

ELECTROLYSIS AND DRAG-OUT

TriCOL Replenisher 200

TriCOL Replenisher 300

TriCOL Antimist 400

DRAG-OUT

TriCOL T-Salts 100

Addition agents that are lost by electrolysis can be replaced by ampere-hour determination. Materials that are lost by drag-out can be replaced by checking the specific gravity of the plating bath.

Use the log sheet provided on the following page to determine the appropriate additions to the TriCOL plating solution. High drag-out or low drag-out may require adjustment to the addition rates for the TriCOL process.

TROUBLESHOOTING

DARK STREAKS

CAUSE

Low TriCOL REPLENISHER 300

Low specific gravity

Metallic contamination

Contaminated nickel-plated surface

Organic contamination

Low TriCOL Antimist 400

REMEDY

Add TriCOL REPLENISHER 300

Add TriCOL T-SALTS 100

Turn on TriCOL PURE or dummy-plate at 20 - 40 Amps/ft²

Improve rinsing after nickel or delay plating in the TriCOL bath

Carbon Treat

Add TriCOL ANTIMIST 400

POOR COVERAGE

CAUSE

Low specific gravity

Low pH

High temperature

Low TriCOL CD PLUS 500

Low Current Density

High agitation

REMEDY

Check and adjust with TriCOL T-Salts100

Adjust with ammonium hydroxide

Reduce temperature to 85 - 90°F (29 - 32°C)

Add TriCOL CD PLUS 500

Increase Current Density

Reduce agitation

PATCHY WHITE DEPOSIT

CAUSE

Organic contamination

Dry-on of nickel-plating solution

Organic contamination

Contaminated nickel rinses

Zinc contamination

REMEDY

Carbon Treat

Transfer racks faster, increase rinsing time, lower nickel temperature
Delay current initiation in TriCOL plating bath

Carbon treat

Dump and refill rinse tanks

Dummy plate at 20 - 40 Amps/ft² or purify

LOW PLATING RATE

CAUSE

Low current density

Low specific gravity

Anodes coated

High pH

Low TriCOL REPLENISHER 300

REMEDY

Increase current applied

Add TriCOL T-SALTS1

Check anodes and clean

Lower pH in 0.2 pH unit increments

Add TriCOL REPLENISHER 300

NON-UNIFORM THICKNESS

CAUSE

Low agitation

Low TriCOL ANTIMIST 400

Low TriCOL REPLENISHER 300

High TriCOL REPLENISHER 300

Poor anode spacing

REMEDY

Increase agitation

Add TriCOL ANTIMIST 400

Add TriCOL REPLENISHER 300

Allow concentration to fall

Check length to anode length and adjust

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.