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TriCOL® RECLAIM

TECHNICAL DATA
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TriCOL® RECLAIM

PROCESS FOR RECLAIMING TRIVALENT CHROMIUM SOLUTION

TriCOL RECLAIM	allows reclamation of trivalent chromium solutions and eliminates chromium dust exposure to operators.
TriCOL RECLAIM	can save up to 60% on chemical costs with proper reclamation system and also reduce waste treatment costs.
TriCOL RECLAIM	has excellent metal distribution across the entire current density plating range.
TriCOL RECLAIM	is a state of the art “newest generation” trivalent chromium process providing the deepest-lightest-whitest appearance.
TriCOL RECLAIM	eliminates burning and whitewash associated with the hexavalent process.
TriCOL RECLAIM	has excellent plating speed and allows easy conversion from existing hexavalent chromium plating.

OPERATING PARAMETERS

PARAMETER	RANGE	OPTIMUM
Temperature	27 - 43º C (80 - 110º F)	29 - 32º C (85 - 90º F)
pH	2.3 - 2.9	2.4 - 2.8 optimum
Current Density	8.5 - 13.4 ASD (80 - 125 ASF)	
TriCOL RECLAIM BASE SALTS 111	279 - 329 g/L (37.2 - 43.9 oz/gal)	300 g/L (40 oz/gal)
TriCOL RECLAIM 200L	80 - 120 mL/L	100 mL/L
TriCOL RECLAIM REPLENISHER 333	50 - 65 mL/L	60 mL/L
TriCOL ANTIMIST 400	1.0 - 4 mL/L (0.1 - 0.4%/vol.)	1.5 mL/L (0.15%/vol.)
TriCOL CD PLUS 500	2.0 mL/L (0.2%/vol.)	
Chromium (Cr+3)	20 - 23 g/L (2.7 - 3.1 oz/gal)	21 g/L (2.8 oz/gal)

Specific Gravity	1.170 - 1.210	1.190
Agitation	Air through low pressure blower recommended.	
Anode to Cathode Ratio	1.5:1 - 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.	

SOLUTION MAKEUP

MATERIAL	**100 LITERS**	**100 GALLONS**
TriCOL RECLAIM BASE SALTS 111	30 kg	247.8 pounds
TriCOL RECLAIM 200L	10 L	10 gal
TriCOL RECLAIM REPLENISHER 333	6 L	6 gal
TriCOL ANTIMIST 400	150 mL	0.15 gal
TriCOL CD PLUS 500	200 mL	0.2 gal

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 then connect the system to the plating tank.

4. Fill tank full with water.
   - Check agitation to ensure it is uniform.
   - Turn on Ion Exchange system and check flow rate then turn off, and turn flow valves off.
   - Check heating and see what the heat up time of solution to 60º C is. Then turn on cooling if equipped to see if it works properly.
   - Lower pH to 2.5 with hydrochloric acid.
   - Allow solution to stand for eight hours at operating temperature of 32º C. This will leach the equipment and tank. Run the Ion Exchange system for 1 hour.
   - Pump out the leach solution, drain the Ion Exchange system, and rinse the tank out.

5. Fill the plating tank 1/3 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 - 65º C (140 - 150º F).

7. Using air agitation, slowly add 300 g/L of the TriCOL RECLAIM BASE SALTS 111. Solution temperature will drop as the salts are added. Bring the temperature back up to 60 - 65º C (140 - 150º F). If the material is added too fast it may settle to the bottom and not dissolve properly. Check bottom of tank to ensure all the material is dissolved before proceeding to the next step. It takes approximately 2 hours at the high temperature to completely dissolve all the powder salts.

8. Add 100 mL/L TriCOL RECLAIM 200L.

9. Add 60 mL/L of the TriCOL RECLAIM REPLENISHER 333 then bring volume up to operating level, maintain heat for 4 hours.

10. Allow solution to cool to operating temperature.

11. Add 1.5 mL/L of the TriCOL ANTIMIST 400 then add 2 mL/L of the TriCOL CD PLUS 500.

12. Allow solution to mix for 10 minutes then check pH of the solution. Adjust if necessary.

13. Check the concentration of the chromium metal, and the TriCOL RECLAIM REPLENISHER 333 and adjust if necessary.

14. The solution will be ready to plate parts at this point.

15. 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 0.25 – 0.50 meters squared per 1000 L or 1 - 2 square feet/100 gal. Dummy plate at 9.7 amps/dm^2 or 90 amps/ft^2 for 1 hour, then plate parts to see if all defects have been removed.

RECOMMENDED EQUIPMENT

Heaters or cooling coils:	Quartz, PTFE, and titanium (must be grounded or anodic).
Tank or Tank Liner:	New PVC, polypropylene, polyethylene, ABS or Koroseal-lined steel.
Heaters/Chillers:	Quartz, PTFE, 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.
Buss Bars:	Recommend copper buss that is heavily nickel plated to avoid possible copper contamination. Buss bars that are exposed should be protected from solution by a plastic shield or by wrapping with plastic waterproof tape.
Ion Exchange:	Use Columbia TriCOLPURE ION EXCHANGE system.
Ion Exchange Resin:	Use Columbia TriCOLPURE Resin.

EQUIPMENT

TriCOL ANODES

Graphite anodes from Columbia must be used. Anode length should be approx. 2.5 -3.75 cm (1 - 1 ½ 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 amps/meter squared (50 amps/square foot). 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

The additions to the solution are made by ampere-hours. A suitable ampere/hour meter should be used to ensure proper control of the process. Automatic metering pump for the TriCOL RECLAIM REPLENISHER 333 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 over existing hexavalent process.

TriCOLPURE ION EXCHANGE SYSTEMS

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

TriCOLPURE ION EXCHANGE RESIN

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

MAINTENANCE ADDITIONS

TriCOL RECLAIM BASE SALTS 111

TriCOL RECLAIM BASE SALTS 111 is used for make-up and replenishment of the solution from drag-out. It provides conductivity to the bath. Additions of TriCOL RECLAIM BASE SALTS 111 should be made slowly to minimize undissolved salts in the bath.

The addition of 21 g/L (2.8 oz/gal) of the TriCOL RECLAIM BASE SALTS 111 will raise the specific gravity 0.01 units.

High concentration of TriCOL RECLAIM BASE SALTS 111 can result in crystallization if the solution temperature is too low.

Low concentration of TriCOL RECLAIM BASE SALTS 111 will result in lower conductivity and may require use of higher voltages to maintain the same current density.

TriCOL RECLAIM 200L

TriCOL RECLAIM 200L is used for make-up or when the solution is low in chromium concentration.

Additions larger than 6 mL/L should be made in several small increments. If a very large addition must be made then it may be helpful to heat the solution for 4 - 6 hours after the addition to properly complex the chromium.

To raise chromium by 0.1 oz/gallon (Multiply tank volume in gallons) X (0.00386) = (amount in gallons of TriCOL Reclaim 200L to add).

OR

38.6 mL/Liter TriCOL Reclaim 200L = 7.5 g/L chromium or 1 ounce/gallon chromium

TriCOL RECLAIM REPLENISHER 333

TriCOL RECLAIM REPLENISHER 333 is proportioned and precomplexed and contains appropriate amounts of the chromium and complexer and other proprietary chemistry to allow simple control of the chromium complex to be plated out of the solution. TriCOL RECLAIM REPLENISHER 333 is consumed by drag-out and electrolysis. High TriCOL RECLAIM REPLENISHER 333 can result in some precipitation. Low TriCOL RECLAIM REPLENISHER 333 can result in reduced plating rate. Maintain the concentration of the TriCOL RECLAIM REPLENISHER 333 within the specified ranges. TriCOL RECLAIM REPLENISHER 333 should be added at 1588 mL per 1000 amp-hrs. Solution and is precomplexed so large additions are not detrimental compared to competitive processes.

1 mL/L will raise the chromium concentration by approximately 0.0375 g/L and increase TriCOL RECLAIM REPLENISHER 333 by 0.1%/vol.

OR

1mL/Liter TriCOL RECLAIM REPLENISHER 333 = 0.0375 g/Liter chromium or 0.0050 ounce/gallon chromium

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

amp-hr. (0.6 - 1.2 fl.oz./1000 amp-hr.) and should be added at least once every 4 amp-hrs./L (15 amp-hrs./gal) 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 amp/L 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 2.3 and 2.9. Raise the pH 0.1 unit with the addition of 2 mL/L (2 gal/1000 gal) ammonium hydroxide. Lower the pH 0.1 units with the addition of 2 mL/L (2 gal/1000 gal) hydrochloric acid. pH adjustments will produce an artificially large change in pH that will equilibrate after a period of time. It is also recommended pH calibration buffers bracket the pH being measured.

ANALYTICAL PROCEDURE

TriCOL RECLAIM ANALYSIS PROCEDURES

TriCOL RECLAIM REPLENISHER 333

REAGENTS: 5% Sodium Carbonate solution (dissolve 50 grams Na_2CO_3 (AR) in DI water and dilute to 1 L)
10% w/v Potassium Iodide solution
1:1 Sulfuric acid (cautiously dissolve 100 mL of concentrated H_2SO_4 (AR) in 100 mL of DI water)
0.100 N Sodium thiosulfate solution
0.100 N Potassium permanganate solution
1% Starch Indicator solution

EQUIPMENT: Pipette 1.0, 5.0, and 25.0 mL volumetric
Flask 250 mL Erlenmeyer

METHOD:

1. Pipette 1.0 mL of the 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 5.0 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 smell the flask for ammonia. All ammonia should be driven off but if an odor of ammonia can be detected, reheat for another 2 - 3 minutes. In any event, all ammonia must be removed before continuing.

4. Remove from the water bath, wash down the sides of the flask with 5 mL DI water. Cool to room temperature.

5. Pipette 25.0 mL of approx. 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 KI solution.

9. Add 5 mL of 1:1 H_2SO_4 and immediately titrate with 0.100 N sodium thiosulfate solution until the solution turns a light yellow. Add approx. 10 drops 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 RECLAIM REPLENISHER 333 volume% = 0.5757 X (mL B - mL A) where A = mL 0.1N sodium thiosulfate for the sample, B= mL 0.1N sodium thiosulfate for blank.

<u>TRIVALENT CHROMIUM</u>

REAGENTS: 30% Hydrogen Peroxide solution, AR (see Note)
50% Sodium Hydroxide Solution
10% w/vol 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 solution
0.100 N Sodium Thiosulfate solution

EQUIPMENT: Pipette - 5 mL volumetric
Volumetric Flask - 100 mL
Flask - 250 mL Erlenmeyer flask

METHOD:

1. Pipette 5.0 mL of the filtered solution into a 100 mL volumetric flask and dilute to volume.

2. Pipette 5.0 mL of the solution from the 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 five (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 straw color.

8. Add 1 mL 1% starch solution and again titrate to the disappearance of the dark blue starch/iodine color.

9. Calculate the concentration as follows:

a. oz/gal Cr+3 = mL 0.1 N $Na_2S_2O_3$ X 0.904
b. g/L Cr+3 = oz/gal X 7.5

DETERMINATION OF SURFACE TENSION

APPARATUS: Stalagmometer: Available from Kocour

PROCEDURE:

The TriCOL ANTIMIST 400 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.

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, 1, 2, and 3 mL/L to prepare a concentration versus surface tension graph.

Take an average of three readings for each standard.

Calculate surface tension as:

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

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)

HELPFUL HINTS

OVERVIEW

TriCOL RECLAIM 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 ADDITION AGENT CONSUMPTION

TriCOL RECLAIM 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 RECLAIM 200L
TriCOL RECLAIM REPLENISHER 333
TriCOL ANTIMIST 400

DRAG-OUT

TriCOL RECLAIM BASE SALTS 111

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 a log sheet to determine the appropriate additions to the TriCOL RECLAIM plating solution. High drag-out or low drag-out may require adjustment to the addition rates for the TriCOL RECLAIM process.

TROUBLESHOOTING GUIDE

DARK STREAKS

CAUSE	REMEDY
Low TriCOL RECLAIM REPLENISHER 333	Add TriCOL RECLAIM REPLENISHER 333
Low specific gravity	Add TriCOL RECLAIM BASE SALTS 111
Metallic contamination	Turn on TriCOLPURE or dummy plate at 20 – 40 amps/ft^2
Contaminated nickel-plated surface	Improve rinsing after nickel or delay plating in the TriCOL bath
Organic contamination	Carbon treat
Low TRICOL ANTIMIST 400	Add TriCOL ANTIMIST 400

POOR COVERAGE

CAUSE	REMEDY
Low specific gravity	Check and adjust with TriCOL RECLAIM BASE SALTS 111
Low pH	Adjust with ammonium hydroxide
High temperature	Reduce temperature to 85 - 90º F (29 - 32º C)
Low TriCOL CD PLUS 500	Add TriCOL CD PLUS 500
Low Current Density	Increase Current Density
High agitation	Reduce agitation

PATCHY WHITE DEPOSIT

CAUSE	REMEDY
Organic contamination	Carbon Treat
Dry-on of nickel-plating solution	Transfer racks faster, increase rinsing time, lower nickel temperature
	Delay current initiation in TriCOL RECLAIM plating bath

Organic contamination	Carbon treat
Contaminated nickel rinses	Dump and refill rinse tanks
Zinc contamination	Dummy plate at 20 - 40 amps/ft^2 or purify

LOW PLATING RATE

CAUSE	REMEDY
Low current density	Increase current applied
Low specific gravity	Add TriCOL RECLAIM BASE SALTS 111
Anodes coated	Check anodes and clean
High pH	Lower pH in 0.2 pH unit increments
Low TriCOL RECLAIM REPLENISHER 333	Add TriCOL RECLAIM REPLENISHER 333

NON-UNIFORM THICKNESS

CAUSE	REMEDY
Low agitation	Increase agitation
Low TriCOL ANTIMIST 400	Add TriCOL ANTIMIST 400
Low TriCOL RECLAIM REPLENISHER 333	Add TriCOL RECLAIM REPLENISHER 333
High TriCOL RECLAIM REPLENISHER 333	Allow concentration to fall
Poor anode spacing	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.