



1000 Western Drive
Brunswick, OH 44212
PHONE: 330-225-3200
FAX: 330-225-1499
www.columbiachemical.com

TriCOL® BlackJack

TECHNICAL DATA
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TriCOL® BlackJack

PROCESS FOR BLACK TRIVALENT CHROMIUM PLATING

TriCOL® BlackJack	Provides the deepest darkest color in the industry.
TriCOL® BlackJack	Color can be adjusted from a light smoky color to a deep dark black color.
TriCOL® BlackJack	Eliminates burning and whitewash associated with hexavalent processes.
TriCOL® BlackJack	Greatly reduces waste treatment costs.

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	45 - 75 mL/L (4.5-7.5% by vol)	55 mL/L (5.5% by vol)
TriCOL® Antimist 400	2 - 6 mL/L (0.1-0.6% by vol)	4 mL/L (0.4% by vol)
TriCOL® CD Plus 500	1.0 mL/L (0.1% by vol)	1.0 mL/L (0.1% by vol)
TriCOL® BLACK 600	1 - 32 mL/L (0.1 - 3.2%/vol)	
Chromium (Cr+3)	20 - 23 g/L (2.7 - 3.1 oz/gal)	21 g/L (2.8 oz/gal)
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 0.05 - 0.10 microns at 10.8 Amps/dm ² (2 - 5 microinches/min at 100 Amps/ft ²)	
Filtration	Usually not required, proper Ion Exchange equipment I needed.	

SOLUTION MAKEUP

	<u>100 LITERS</u>	<u>100 GALLONS</u>
TriCOL® T-Salts 100	41.5 kg	346 pounds
TriCOL® Replenisher 300	6.5 L	6.5 gal
TriCOL® Antimist 400	400 mL	0.4 gal
TriCOL® CD Plus 500	100 mL	0.1 gal
TriCOL® BLACK 600	Dependent on color desired	

SOLUTION 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.
 - a. Check agitation to insure it is uniform.
 - b. Turn on the Ion Exchange system and check flow rate then turn off, and turn flow valves off.
 - c. 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.
 - d. Lower pH to 2.5 with hydrochloric acid and then add 1 mL/L of the TriCOL® Antimist 400.
 - e. 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.
 - f. 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/L 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 55 mL/L (5.5%/vol) TriCOL® Replenisher 300 and the TriCOL® BLACK 600 then bring tank volume up to operating level and maintain heat for 4 hours.
9. Allow solution to cool to operating temperature.
10. Add 4 mL/L of the TriCOL® Antimist 400, then add 1 mL/L 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. 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 L or 1 - 2 ft²/100 gal. 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.

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).

Racks

Plastisol-coated copper.

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.

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.

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.

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® Replenisher300 and the TriCOL® Black 600 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.

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/L (4 oz/gal). 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/L (2.8 oz/gal) 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/L (13 ampere-hours/gal) of operation.

Additions larger than 7.0 g/L (0.93 oz/gal) 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 L (51 fl. oz) per 1000 ampere-hours and should be added at least once every 4.5 ampere-hours/L (17 ampere-hours/gal) 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/L (15 ampere-hours/gal) of operation. Concentration can be determined by measuring the surface tension.

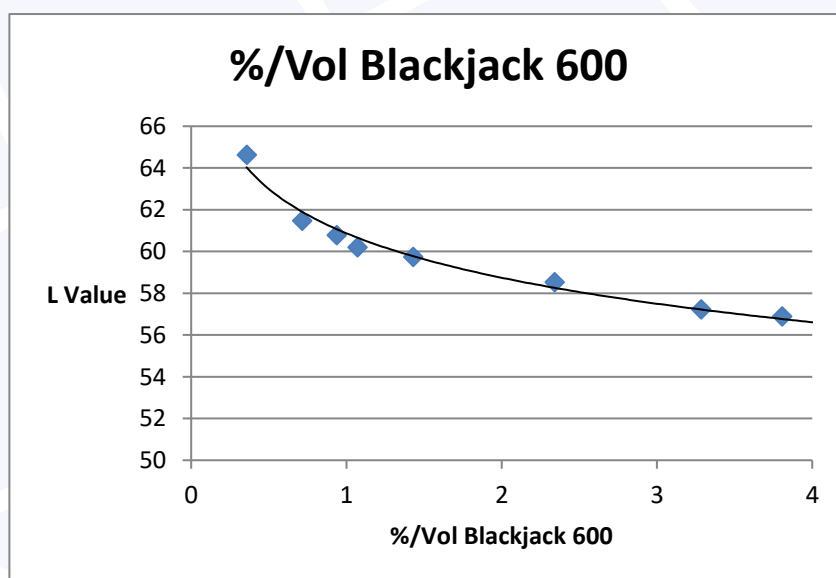
TriCOL® CD Plus 500

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.

TriCOL® BLACK 600

TriCOL® BLACK 600 produces the black color in the deposit. TriCOL® BLACK 600 is used for make-up and replenishment. The degree of darkness can be increased by increasing the concentration of the TriCOL® BLACK 600. Concentration of the TriCOL® BLACK 600 can be determined by measuring the degree of darkness of the deposit. The chart below gives the L-Value versus the %/volume concentration of the TriCOL® BLACK 600. However, the degree of darkness does not increase substantially over the concentration of 32 mL/L (3.2%/vol). It is there recommended that the upper limits for concentration should be 32 mL/L.

Consumption of the TriCOL® BLACK 600 depends on the concentration of TriCOL® BLACK 600 maintained in the operating solution. Lower concentrations of TriCOL® BLACK 600 such as 1 mL/L (0.1%/vol) will require an addition rate of 78 mL /1000 AH (2.6 fl. oz/1000 AH). Higher concentration of TriCOL® BLACK 600 32 mL/L (3.2%/vol) will require an addition rate of 113 mL/1000 AH (3.8 fl. oz/1000AH).



Temperature

The process operates at 27 - 43° C (80 - 110° F), heating will be required. If production is above 0.5 amperes/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 2.6 - 2.8. Raise the pH 0.1 unit with the addition of 2 mL/L (2 gal/1000 gal) ammonium hydroxide. Lower the pH 0.1 unit 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 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**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. **CALCULATION:** $\text{oz/gal Cr+3} = \text{mL } 0.1 \text{ N Na}_2\text{S}_2\text{O}_3 \times 0.904$
 $\text{g/L Cr+3} = \text{oz/gal} \times 7.5$

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.

CALCULATION:

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

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)

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.