

PLATING PRODUCTS IND PVT LTD

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Technical Data Sheet

Trivalent Black Chromium (Type of Sulfuric Acid)

I. Descriptions

It is a black trivalent chromium plating process. It is usually plated on bright nickel or satin nickel to produce a bright chrome layer. It operates with a lower chromium concentration than the hexavalent system, significantly reducing the cost of wastewater treatment. It uses a simple additive and anode system to make process control simple and easy.

II. Operating conditions

Item	Range	Optimal Value
Conductive salt	220-280g/l	250g/l
A	100-150ml/l	150ml/l
C	30ml/l	
Bath opening additive	10ml/l	
D	20-35ml/l	30ml/l
Wetter	2ml/l	
Temperature	50-60° C	55° C
PH value	3.4-3.8	3.6
Anode current density	5-10A/dm ²	7A/dm ²
Voltage	MAX: 12v	
Stirring	Gentle air stirring or light solution stirring	

III. Bath opening

1. Add the following chemicals per 1L solution:

1)Conductive salt: 250g/l

2)A: 100ml/l

3)C: 30ml/l

4)B: 5ml/l

5)Wetter: 2ml/l

6)Bath opening additive: 10ml/l

2. Add 60% volume of pure water to the plating bath and heat to 55 °C. Add the required amount of conductive salt and stir until completely dissolved.

3. Add the required amount of A.

4. Well mixed, heat to the operating temperature and leave the solution for 8 hours.

5. Adjust the PH value to 3.5 with 25% caustic soda. Check again a few hours later, adjust the PH value to 3.5. Repeat until the PH value stabilizes at 3.5. If the local PH value is too high, it will be harmful to the bath solution, add 25% sodium hydroxide for adjustment and add it slowly and stir strongly. Note: PH value is not allowed to be higher than 3.8. 6. Add the required amount of wetter, Bath opening additive, C and B.

7. Electrolytic cell solution with new nickel-plated cathode sheet, at least 2 Ah/L. After every 90 minutes of electrolysis, remove the plating layer, re-plate the nickel, and re-electrolyze, and the PH value change of the bath solution needs to be closely monitored during the initial operation, and 25% sodium hydroxide is slowly added in time to adjust, accompanied by strong stirring.

IV. Maintenance of bath solution

A, B, C, D and E can be added and maintained by ampere-hours. Chromium and conductive salt concentrations should be regularly maintained and adjusted accordingly.

1)A: up to 0.5l/KAh.

Its role is to maintain the chromium concentration in the bath, and the actual amount of plating depends on the plating time and the solution brought out.

2)B: 0.4-0.6l/KAh.

Its role is to maintain the color of the plating layer.

3)C: up to 1.0l/KAh.

Its role is to maintain the color of the plating layer.

4)D: 50-100ml/KAh.

Its role is to maintain the color of the plating layer.

5)E: 0-100ml/KAh.

Work together with D to maintain the black color of the plating layer.

6) Bath opening additive: initial preparation requires 10ml/l for optimal performance.

7) Conductive salt: depends on brought-out consumption and can be maintained by analysis.

8) Wetter: when the surface tension exceeds 40mN/m, wetter needs to be added. 0.5 to 1.0ml/l is sufficient to correct the surface tension.

V. PH value

The PH value of the solution should be kept between 3.4 and 3.8. During general production operations, the PH value may have a downward trend. Slowly add 10% sodium hydroxide to the bath with strong stirring to adjust the PH. If the PH value raises to higher than 3.8 abnormally, 10% sulfuric acid can be slowly added to adjust. Any additions should be done slowly and with strong stirring. If the PH value is higher than 3.8, it can cause the solution to fail. After measuring PH, the PH meter probe must be thoroughly cleaned with 50% hydrochloric acid. Failure to do so will result in an incorrect PH reading.

If a shutdown is required, dilute sulfuric acid is recommended to reduce the PH value to 2.0. The bath temperature needs to be raised to operating temperature and held for at least 24 hours before re-production, which ensures that any precipitated solids are redissolved. Before plating, a 25% solution of sodium hydroxide can be used to adjust to the working PH value. It is recommended to use a normal current density to electrolyse the solution for a period of time before production.

Purification Metal

Metal impurities such as nickel, iron, zinc and copper are often brought into the solution, please refer to the following methods to remove these impurities:

Precipitation (weak electrolysis)

Similar to other plating methods, metal contamination can be removed by weak electrolysis at low current densities. The recommended current density is 2A/dm². After 30 minutes, the plating layer is removed, nickel plated again on the cathode sheet, and electrolysis is performed again.

The bath solution is maintained according to normal per-amperes during weak electrolysis.

Organic matter

Needs to be filtered through an irregular activated carbon filter element to remove organic impurities brought in from the nickel bath, especially poor washing between nickel and chromium.

Organic impurities can cause the plating layer to darken and have poor coverage.

Temperature

The temperature should be maintained between 50 - 65°C. Higher temperature increases plating rates but also reduces electrolyte coverage.

Current density

It is suggested not to exceed the recommended anode current density (5A/dm²) and to be aware of the risk of scorching in high current density areas.

Standard process

- 1.Plating in bright or satin nickel.
- 2.Recovery bath (if needed)
- 3.Washing. Before plating with the solution, the workpiece should be thoroughly washed with cleaned and running water. Especially when electroplating tubular workpieces or any other workpieces that are not easily washed out, poor cleaning can lead to contamination of the bath with nickel or iron which results in dim plating and reduced plating rates.
- 4.The workpiece "stands" in the plating bath for 10-30 seconds before applying the current.
- 5.Wash thoroughly in a clean alkaline solution, for example: 5-10g/l sodium carbonate solution.
- 6.Passivation. Trivalent chromium doesn't have the self-passivation as hexavalent chromium, so passivation is required.
- 7.Hot water to clean and dry the workpieces.

Passivation

Trivalent chromium bath solution doesn't have the passivation characteristics as hexavalent chromium. The surface of the chromium layer remains "active" until it is plated with a passivation layer, which can also be passivated by air oxidation, but it takes a longer time. Unplated substrates (e.g. inside steel pipes) also require corrosion protection, and different substrates require different types of passivation.

Bath

PVC lining or PP plating bath.

Heating

Titanium coil, titanium coating, silica jacket or Teflon immersion electric heater. Thermostatic control is the key point.

Filtration

It is recommended to continuously filter the bath solution.

PH

Accurate PH meter.

Stirring

Double row PVC or ABS air mixing pipe.

Automatically adding

It is strongly recommended to add A, B, C, D and E by ampere-hour.

Anode: Titanium + composite metal oxide (MMO) anode.

Anode area: The ratio to cathode area is 2:1.

Note: Inert anodes must be handled carefully to prevent chips or cracks in the plating. These anodes can't be operated at anode current densities higher than $5A/dm^2$.

Analytical and control

Chromium content determination

Reagents:

Sodium peroxide

Sodium hydrogen fluoride

Potassium iodide

Concentrated hydrochloric acid

0.1N sodium thiosulfate standard solution

Soluble starch indicator (if no starch can be used instead)

Methods

1. Pipette 5ml of plating solution into a 250ml Erlenmeyer flask and add pure water until approximately 100ml.

2. Add 2g of sodium peroxide to the solution and put some anti-zeolite.
3. Boil the solution and keep boiling for 30 minutes, adding pure water to maintain the capacity.
4. Cool the solution to room temperature. Add 1g sodium hydrogen fluoride, 1g potassium iodide, and 15ml of concentrated hydrochloric acid.
5. Titrate with 0.1N sodium thiosulfate standard solution until the color becomes light, add soluble starch indicator and continue the titration. **Counting** ml of titration $\times 5.18 = \text{ml/l A}$ ml of titration $\times 0.36 = \text{g/l chromium}$

Chromium content determination (instrumental analysis)

Reagents:

10% caustic

30% hydrogen peroxide

Methods

1. Pipette 5.0ml of plating solution into a 100ml beaker, dilute with 10ml of pure water and add 20ml of 10% caustic solution.
2. Add 5.0ml of 30% hydrogen peroxide after 10 minutes. Heat slowly to 80°C and heat continuously for 30 minutes.
3. After cooling to room temperature, dilute the solution with pure water in a volumetric flask to 100ml.
4. Dilute 10ml with pure water in a volumetric flask to 500 ml. Use pure water as a reference solution and determine the absorption of the final dilution at 374nm by using a 10mm glass cuvette. **Counting**

Absorption $\times 157 = \text{ml/l A}$

Absorption $\times 11.0 = \text{g/l chromium}$

The chromium content is maintained within the range of 5 - 8.5g/l by adding A.

The addition of 10ml/l A increases 0.7g/l of chromium. For every 1g/l of chromium that needs to be increased, 15 ml/l A should be added.

Determination of conductive salt

Reagents:

Phenol red indicator (0.1% phenol red indicator added into 20% ethanol)

Mannitol

0.1N NaOH

Methods:

1. Pipette 2ml of solution sample into a 250ml flask.
 2. Add 40 - 50ml of water.
 3. Add a few drops of phenol red indicator and the solution turns turbid yellow.
 4. Add 0.1N NaOH until the solution turns clear red.
 5. Add about 5g of mannitol powder and stir to mix, the solution will turn turbid yellow.
 6. Titrate with 0.1N NaOH until the red endpoint (same red as before mannitol is added) **Counting**
- Titration x 13.46 = g/l conductive salt

Composition

1. Conductive salt
2. A
3. B
4. C
5. D
6. E
7. Bath opening additive
8. Wetter