

SurTec® 864

Cyanide-Free Alkaline Copper Process

Properties

- makes the use of cyanide electrolytes with their high risk for environment and health superfluous
- can be used directly on steel and brass, and in rack processes even for zincated aluminium and zinc die cast
- suited for barrel, rack and continuous strip lines
- forms a good basis for acid copper and nickel
- no toxic decomposition products in case of drag-out into acid copper bathes
- tolerates carbonate (no need to freeze out)
- excellent coverage and throwing power
- produces fine-granular, ductile und non-porous deposits
- IMDS-number: 736943

Application

The process SurTec 864 includes the following products:

- SurTec 864 Electrolyte Concentrate supplies copper in the necessary matrix and a little amount of complexing agent
- SurTec 864 I Corrector contains the complexing agent of the system

make-up values:

SurTec 864 Electrolyte Concentrate	30 %vol	(25-60 %vol)
SurTec 864 I Corrector	6.5 %vol	
KOH solution (45 %)	to adjust the pH	

analytical values:	copper	9 g/l	(7.5-18 g/l)
	phosphorus as complex	36 g/l	(35-40 g/l)
	ratio Cu : P	1 : 4	

make-up:

Steps for make-up:

1. Remove residual cyanide by treating the tank and other material (filter pumps, anode baskets etc.) with 2 % solution of sodium hypochlorite for 24 h, then rinse with water, 2 % sulfuric acid, again with water and finally with 5 % potash lye.
2. Fill the cleaned tank with max. 1/3 water (depending on the make-up values).
3. Add SurTec 864 I Corrector.
4. Adjust the pH-value with 45 % KOH solution.
5. Add SurTec 864 Electrolyte Concentrate.
6. Correct the pH-value once again.
7. Fill up with water to the final volume.

temperature: 55°C (50-70°C)

pH-value: 9.5 (9.2-9.8) *for iron*

9.2 (9.0-9.4) *for zinc dye cast and aluminium*

lower with SurTec 864 I Corrector or rise with 45 % KOH solution

covering current: 1-2 A/dm² applied for 5 min

cathodic
current density: 0.1-1 A/dm² *for barrel application*
0.5-1.5 A/dm² *for rack application*
depending on the make-up concentration,
higher current density also is possible

rectifier: 18-20 V

current efficiency: approx. 95 % at 0.4-2 A/dm²

deposition rate: approx. 0.3 µm/min at 1.5 A/dm²

conductivity: approx. 70 mS/cm *at 20-25°C*
approx. 60 mS/cm *at 55-60°C*
according to the make-up concentration, higher values are possible

anodes: OFHC-copper (Oxygen Free High Conductivity)
as bar anodes or pellets in baskets of titanium

ratio
anode/cathode: approx. 1.5 : 1

tank material: steel with plastic coating

agitation: air agitation (optimal: at anode and cathode area);
barrel rotation with approx. 2-6 rpm

filtration: continuously

heating: stainless steel or Teflon

exhaust: recommended

hint: Zinc die cast and zincate treated aluminium can only be plated in
rack process, contacting the parts before immersing into the bath.
A pre-dip in 1 %vol SurTec 864 I is strongly recommended therefore.

recommended process sequence:

1. **soak cleaning:** since the alkaline cyanide-free electrolyte gives unlike cyanide no cleaning effect, the parts must be perfectly pretreated with well rinsable cleaners:
zinc die cast: SurTec 151 Alkaline Cleaner
steel: SurTec 188 + SurTec 091 resp. SurTec 092
2. **pickling:** with well rinsable acid cleaners and inhibitors:
steel: SurTec 424 or SurTec 425 Inhibitor
3. **electrolytical cleaning:** free of surfactants and without or just with soft complexing agents:
zinc die cast: SurTec 177 Electrolytical Cleaner
steel: SurTec 419 + SurTec 188 Cleaner
4. **activation:** after using an electrolytical silicate-containing cleaner (e.g. SurTec 177), a fluoride activation is necessary:
SurTec 481 Salt for Acid Dip
5. **pre-dip:** for best adhesion the parts can be pre-dipped in 10 ml/l SurTec 864 I (especially recommended for barrel plating and zinc die cast)

Between each step there has to be rinsed thoroughly, in order to prevent any drag-in of silicate, inhibitors or surfactants into the electrolyte. Circulating rinse water should be of appropriate good quality (in case of doubt send a specimen for evaluation).

Technical Specification

(at 20°C)	Appearance	Density (g/ml)	pH-value (conc.)
SurTec 864	liquid, blue, clear	1.380 (1.32-1.43)	9.2 (8.0-11.5)
SurTec 864 I	liquid, colourless, clear	1.146 (1.13-1.16)	approx. 1.0

Maintenance and Analysis

Compensate drag-out losses and correct the pH-value with SurTec 864 I and 45 % KOH. Replenish evaporation losses only with deionised water. After longer periods of working interruption, the bath should be filtrated through an active carbon cartridge.

All values (e.g. content of complexing agent or current density) have to be adjusted according to the current copper concentration.

SurTec 864 Electrolyte Concentrate supplies copper in the necessary matrix and a little amount of complexing agent. During the plating process, the deposited copper will be replenished by anode corrosion. Replenish drag-out losses of the complexing agent matrix by adding SurTec 864 I. An addition of SurTec 864 Electrolyte Concentrate is necessary just in case of make-up, or if an impoverishment is determined (see analysis). If this happens frequently, the anode/cathode ratio should be raised.

SurTec 864 I Corrector contains the complexing agent of the system. Additions of SurTec 864 I are necessary in order to compensate drag-out losses and electro-chemically build-in. A lack of SurTec 864 I leads to adhesion problems, a slight excess has no negative effect. The corrector solution is strongly acidic, so add it carefully and correct the pH-value afterwards with 45 % KOH solution.

Mainly in case of combines treatment of zinc dye cast with aluminium, a very regulated dosage, best with automatic control by pH-electrodes (measuring by bypass), must be ensured.

Sample Preparation

Take the sample at a homogeneously mixed position and let it cool down to room temperature. If dull, allow to settle and decant or filter.

Copper (Cu) – Analysis by Titration

reagents:	ammonium persulfate ammonia (conc.) 0.1 mol/l EDTA solution (Titrplex III) PAN-indicator (1-(2-pyridylazo)-2-naphthol), 1 g/l in ethanol
procedure:	<ol style="list-style-type: none">1. Pipette 5 ml bath sample into a 250 ml Erlenmeyer flask.2. Dilute with approx. 25 ml deionised water.3. Add 2-3 g ammonium persulfate.4. Stir the solution for about 15 min.5. Add 5 ml conc. ammonia solution (colour turns to dark blue).6. Add another 50 ml deionised water.7. Add 4-6 drops PAN-indicator (do not add more: the colour changing point will not be clearly observable).8. Titrate with 0.1 mol/l EDTA from dark blue to greenish-grey.
calculation:	consumption in ml · 1.27 = g/l copper
correction:	rise by 1 g/l Cu = addition of 33 ml/l SurTec 864 (with this addition also the content of P rises by 3 g/l)

Phosphorous and SurTec 864 I Corrector – Analysis by Cuvette Test

equipment: Dr. Lange Cuvette Test LCK 350
(measuring range: 6-60 mg/l phosphate or 2-20 mg/l phosphorous)
50 ml and 100 ml volumetric flask

procedure: Dilution **a**) 1:200
Pipette 0.5 ml bath sample into a 100 ml volumetric flask, fill up to the final volume with deionised water and mix well.

Dilution **b**) 1:5000

Pipette 2 ml of dilution **a**) into a 50 ml volumetric flask. Fill up with deionised water and mix well.

The phosphorous content of these two dilutions has to be determined. Therefore, make the analysis according to the description of Dr. Lange, with two exceptions (see bold text).

A) without heat treatment (**free phosphorous (P)**, result **A**):

1. Remove the white DosiCap from one of the cuvettes.
2. Pipette 0.4 ml of dilution **a**) into the cuvette.
3. Add 0.5 ml of reagent B.
4. Close the cuvette with DosiCap C, mix well and measure it after **30 minutes** (= result **A**).

B) with heat treatment (**total phosphorous**, result **B**):

5. Remove the white DosiCap from the cuvette and remove the protecting film.
6. Pipette 0.4 ml of dilution **b**) into the cuvette and close it with the same Cap, but with the riffled end of the Cap down to the glass.
7. Mix well and leave it for **heat treatment 2 h** at 100°C in a thermostat to de-compose the phosphorous compound.
8. Cool down to room temperature.
9. Add 0.5 ml reagent B, close the cuvette with DosiCap C, mix again.
10. Measure after 10 minutes reaction time at 850 nm (= result **B**).

calculation: result **A** · 0.2 = g/l free phosphorous

result **B** · 5 = g/l total phosphorous

total phosphorous (in g/l) - free phosphorous (in g/l)
= g/l phosphorous in the complexing agent

desired value: (P-complexing agent in g/l) / (Cu in g/l) = 4 or more

correction: rise by 1 g/l phosphorous = addition of 12 ml/l SurTec 864 I

rise by 1 g/l phosphorous = addition of 4 ml/l SurTec 864 I K

attention: SurTec 864 I is acidic. The pH-value of the bath has to be controlled and adjusted with 45 % KOH solution, after addition of the correction solution.

Hull Cell Test

For evaluation of the deposition, perform all tests in a standard 250 ml Hull cell. Plate copper at 60°C with 0.5 A for 15 min on a pretreated steel panel (pickled and anodic electrolytic degreased). The first panel shows the current state of the bath. Adjust the desired basic values according to the analysis (2. panel). With the next panels correct the organic and the bath conditions with the aid of the trouble shooting table (see annex).

To check the adhesion properties of the layer, blend the panels especially in the lcd.

Bath Impurities

Cyanide

Produces dark layers with bad adherence in the lcd.

Remedy: Addition of 5-10 ml/l H₂O₂ (1:10 pre-diluted)

Lead

Above a content of approx. 50 mg/l lead, it causes dark copper layers in the hcd with bad adherence, the higher the Pb content is.

Remedy: Work-out at high current densities on panels with cathode bag. It is essential to find and eliminate the source of lead.

Iron

Above a content of approx. 2 g/l iron, the adherence of the layer in the hcd decreases.

Remedy: The addition of SurTec 864 I Corrector firstly masks the excessive iron. At long sight, the iron has to be worked out at high current densities.

Organic Impurities

Dragged-in organic produces matt depositions.

Remedy: Treatment with H₂O₂ (1:10 pre-diluted) and successive active carbon filtration.

Copper(I)

Copper(I) is formed at the anodes at too low anodic current density. At higher concentrations, it will lead to greenish electrolyte colour and the adherence of the deposit decreases.

Remedy: Copper(I) can be oxidised to Copper(II) by working with air agitation. So air agitation is generally recommended. Copper(I) can also be oxidised by adding H₂O₂ (1:10 pre-diluted) in regular intervals.

Consumption and Stock Keeping

The consumption depends heavily on the drag-out as well as on cathodic build-in. To determine the exact amounts of drag-out, see [SurTec Technical Letter 11](#). The following values per 10,000 Ah can be taken as estimated average consumption:

SurTec 864 I	2 l	(1.5-4 l)
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In order to prevent delays in the production process, per 1,000 l bath the following amounts should be kept in stock:

SurTec 864	100 kg
SurTec 864 I	140 kg
KOH 45 %	100 kg
H ₂ O ₂	1-5 kg

Product Safety and Ecology

The safety instructions and the instructions for environmental protection have to be followed in order to avoid hazards for people and environment. The Material Safety Data Sheets (according to European legislation) contain explicit details for this.

The following hazard designations and classifications into water hazard classes (WHC) have to be taken into account:

<u>product</u>	<u>hazard designation</u>	<u>water hazard class</u>
SurTec 864	Xn - Harmful	WHC 2
SurTec 864 I	Xi - Irritant	WHC 2

Warranty

We are responsible for our products in the context of the valid legal regulations. The warranty exclusively accesses for the delivered state of a product. Warranties and claims for damages after the subsequent treatment of our products do not exist. For details please consider our [general terms and conditions](#).

Further Information and Contact

In our forum, you can discuss topics of the surface technology:
<http://forum.SurTec.com/>

If you have any questions concerning the process, please contact your local technical department: <http://SurTec.com/International.html>

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Trouble Shooting

problem	possible cause	remedy
burnings	a) too low complexing agent	add SurTec 864 I
	b) current density is too high	reduce the current density
	c) insufficient bath agitation	improve the bath agitation
	d) metal impurities (Fe, Pb)	work out at high current density
	e) metal content is too low	increase the anode area or add SurTec 864
	f) too much Cu(I) in the bath	increase the air agitation (especially at the anodes) or add H ₂ O ₂ (1:10 diluted)
	g) bath conductivity is too low	add SurTec 864 I and 45 % KOH
cloudy or hazy deposition (especially in lcd)	a) insufficient pretreatment	check the pretreatment, improve the rinsing process
	b) insufficient rinsing after a fluoride containing acid dip	additional pre-dip in SurTec 864 I (1 %vol) prior to the SurTec 864 electrolyte
	c) organic impurities	treat with H ₂ O ₂ (1:10) and filtrate with active carbon
	d) insufficient agitation	improve the bath agitation
bad coverage	a) too low complexing agent	add SurTec 864 I
	b) metal impurities (esp. Fe)	add SurTec 864 I or work-out
	c) bath conductivity is too low	add SurTec 864 I and 45 % KOH solution
electrolyte turns green	a) too low complexing agent	add SurTec 864 I
	b) too much Cu(I) in the bath	increase the air agitation (especially at the anodes) or add H ₂ O ₂ (1:10 diluted)
	c) metal impurities (esp. Fe)	add SurTec 864 I or work-out
	d) organic impurities	treat with H ₂ O ₂ (1:10) and filtrate with active carbon
too low layer thickness	a) metal content is too low	increase the anode area or add SurTec 864
	b) current density is too low	increase the current density
	c) contacting problems	control / clean the contacts
	d) too much complexing agent	work-out, stop the addition of SurTec 864 I for a short time
	e) plating time is too short	increase the plating time
	f) bath conductivity is too low	add SurTec 864 I and 45 % KOH solution
rough deposit	anode sludge in the bath	check anodes and anode bags, improve the filtration
adherence problems or blister	a) too low complexing agent	add SurTec 864 I
	b) too low current density / too low covering current	increase the covering current nearly to the burning limit
	c) metal impurities	work out at high current density
	d) bath agitation is insufficient	improve the bath agitation
	e) insufficient pretreatment (esp. important for Zn and Al)	check the pretreatment, improve the rinsing process; insert Zn and Al parts under current
	f) bulk material	test other bulk material in comparison