

SurTec® 714

Alkaline Cyanide-Free Zinc/Iron Process (Electrolyte based on Potassium)

Properties

- for rack and barrel plating, with excellent metal distribution
- good covering and throwing power, appropriate for profiled parts
- iron content adjustable from 0.3 to 0.7 % (higher than 0.7 % is not recommended)
- deposits semi bright to brilliant zinc/iron layers, according to the requirement
- blister-free, ductile and well chromatable coatings
- high hydrogen permeability, suited for plating hardened parts
- very good corrosion protection with yellow chromate SurTec 671 and the silver free black chromate SurTec 698, also after heat treatment of several hours at 120°C
- completely temper resistant corrosion protection with SurTec 680 Chromiting
- both trivalent black Chromiting SurTec 695 and SurTec 694 are possible
- IMDS-number: 213579

Application

The process SurTec 714 includes the following products:

- SurTec 714 I Carrier is responsible for the very good metal distribution
- SurTec 714 Fe-C Iron Complex is necessary for replenishing iron and complexing agent during the normal plating process
- SurTec 714 C Complexing Agent is necessary for a new bath make-up and if the content of complexing agent decreases (after analysis)
- SurTec 714 Fe Iron Solution is only necessary, if the complexing agent is too high and iron is missing
- SurTec 700 L LCD Booster is used as a secondary brightener as required and works at low current density
- SurTec 700 EK Potassium Zincate Electrolyte, 3x Concentrate provides zinc, potassium hydroxide and potassium carbonate for the bath make-up (as an alternative to the make-up out of the salts)
- SurTec 700 S Fume Suppressant is optional to reduce aggressive alkaline fumes resulting from the gas evolution

make-up values:

| | |
|---------------------|-----------|
| zinc oxide | 12.5 g/l |
| potassium hydroxide | 170 g/l * |
| potassium carbonate | 40 g/l |

or using the electrolyte concentrate:

| | | |
|--|-------------|------------------|
| SurTec 700 EK | 33 %vol | |
| SurTec 714 I Carrier | 6 ml/l | (4 - 8 ml/l) |
| SurTec 714 Fe-C Iron Complex (contains also SurTec 714 C) | 9 ml/l | (7-11 ml/l) |
| SurTec 714 C Complexing Agent | 65 ml/l | (55-75 ml/l) |
| SurTec 700 L LCD Booster | as required | (0-1 ml/l) |
| (SurTec 700 S Fume Suppressant | 0.1 ml/l | (0.05-0.2 ml/l)) |

*This value is valid only for KOH 100 %; at lower contents of KOH (see specification of the raw material), the make-up value has to be calculated (Example: KOH 86 % => 170/0.86 = 198 g/l)

| | | | |
|--------------------|---------------------|----------|----------------|
| analytical values: | zinc | 10 g/l | (8-12 g/l) |
| | potassium hydroxide | 170 g/l | (160-210 g/l) |
| | potassium carbonate | 40 g/l | (20-80 g/l) |
| | iron | 180 mg/l | (150-200 mg/l) |
| | SurTec 714 C | 70 ml/l | (60-80 ml/l) |

make-up: Steps for the bath make-up:

1. Fill the calculated amount of SurTec 700 EK Potassium Zincate Electrolyte, 3x Concentrate in the plating tank and fill up with deionised water to almost the final volume.
Alternatively dissolve potassium hydroxide and zinc oxide portion by portion in 20 % deionised water while stirring vigorously (attention: solution becomes hot!). After cooling down, fill up with deionised water to almost the final volume,
or dissolve potassium hydroxide in deionised water while stirring (attention: solution becomes hot!) and dissolve the zinc amount by continuous flow through the zinc generator. This takes several hours, depending on the size of the zinc generator.
2. Admix the additives SurTec 714 I, SurTec 714 C and SurTec 714 Fe-C in this sequence.

temperature: 30°C (25-35°C)
the once chosen temperature has to be kept constant ± 1 °C

current density: 0.75-1.5 A/dm² *barrel*
1.5 - 4 A/dm² *rack*

current efficiency: 65 % (50-85 %)

deposition rate: 0.3 μ m/min at 2 A/dm²

ratio

anode/cathode: 2:1

agitation: rack (cathode) agitation; air agitation cannot be applied

tank material: iron with plastic or rubber coating

filtration: necessary: 2-3 times the bath volume per hour

heating: necessary

cooling: necessary for baths with a high throughput

exhaust: recommended for workers' protection

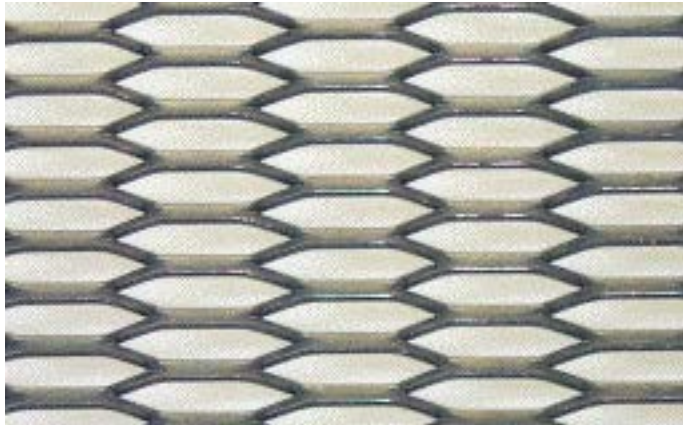
hints: Remove immediately copper containing parts fallen into the bath.

The iron content in the layer should be kept at 0.3-0.7 %.

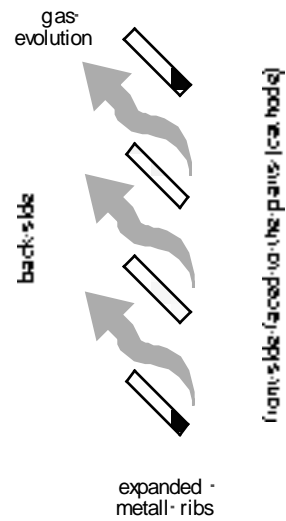
Anodes

Anodes: made of expanded metal (30 mm x 8 mm piccolo mesh, rib width 6 mm, material thickness 2 mm), of mild steel (e.g. ST 37) plated with 15 μ m semi bright nickel. The expanded metal should be installed with the ribs horizontally oriented for driving the gas evolution to the backside.

Before plating the expanded metal with semi bright nickel, it should be vertically stiffened with flat irons leading to the anode hooks. For optimal current distribution, the anodes should be placed at both sides of the cathode along the full width of the plating tank, with an anodic current density up to 20 A/dm².



Flow of the gas evolution when anodes are correctly installed



Zinc generator: with baskets (optimal: 62.5 mm x 62.5 mm x 1000 mm of 1.5 mm perforated mild sheet DD 11 GK according to DIN 10111/10051; perforation Rv 3-5 DIN 24041), plated with SurTec Catalyst. Fill the baskets with zinc clippings (approx. 10 mm Ø, lead content < 0.002 %). Control the zinc concentration in the electrolyte adjusting the exchange rate between plating cell and zinc generator. For an online calculation of the necessary number of catalytic baskets and for determination of the size of the zinc generator, please consult:

<http://calculation.SurTec.com/Zincgenerator.html>

Technical Specification

| (at 20°C) | Appearance | Density (g/ml) | pH-value (conc.) |
|-----------------|------------------------------|-------------------|------------------|
| SurTec 714 I | liquid, colourless-yellowish | 1.017 (1.01-1.03) | 8-10 |
| SurTec 714 Fe-C | liquid, dark red brown | 1.115 (1.08-1.15) | 6-9 |
| SurTec 714 C | liquid, colourless-yellow | 1.035 (1.02-1.05) | 11.2 |
| SurTec 714 Fe | liquid, brown | 1.121 (1.11-1.13) | 0.2-2.2 |
| SurTec 700 L | liquid, yellowish | 1.052 (1.01-1.09) | 12-14 |
| SurTec 700 S | liquid, colourless-opal | 1.001 (1.00-1.01) | 5.5-7.5 |
| SurTec 700 EK | liquid, colourless | 1.485 (1.46-1.51) | > 11 |

Maintenance and Analysis

Analyse and adjust zinc, iron and potassium hydroxide regularly; dose the additives according the table at the topic "consumption" or according to Hull cell tests.

Correct drag-out losses and built-in iron by addition of SurTec 714 Fe-C. An amount of 1 ml SurTec 714 Fe-C equals a dosage of 20 mg iron and 0.5 ml SurTec 714 C.

Regular analysis of the iron content is necessary. Adjust the content of complexing agent, if necessary, by using SurTec 714 C. If there is enough complexing agent inside the bath, but only iron is missing, add SurTec 714 Fe. 1 ml SurTec 714 Fe contains 40 mg iron.

Add SurTec 700 L LCD Booster as required, usually 0-0.2 l per 10 kWh. Avoid strong overdosages because they would reduce the current efficiency and had to be worked out.

Sample Preparation

Take a sample at a homogeneously mixed position and let it cool down to room temperature. If the sample is turbid let the turbidity settle down and decant or filter the solution.

Zinc (Zn) – Analysis by Titration

- reagents: 0.1 mol/l EDTA (Titrplex III)
buffer solution (100 g/l NaOH, 240 ml/l glacial acetic acid)
indicator: xylenol orange tetra sodium salt (1 % in KNO₃)
- procedure: 1. Pipette 5 ml bath sample into a 250 ml Erlenmeyer flask.
2. Dilute with 100 ml deionised (DI-)water.
3. Add 30 ml buffer solution.
4. Add a spatula tip of indicator.
5. Titrate with 0.1 mol/l EDTA from violet to yellow.
- calculation: consumption in ml · 1.3074 = g/l zinc

Potassium Hydroxide (KOH) – Analysis by Titration

- reagents: 1 N sulfuric acid
indicator: Tropaeolin O (0.04 % in 50 % ethanol)
- procedure: 1. Pipette 5 ml bath sample into a 250 ml Erlenmeyer flask.
2. Add 100 ml DI-water.
3. Add 3 drops of indicator.
4. Titrate with 1 N sulfuric acid from red to yellow.
- calculation: consumption in ml · 11.20 = g/l potassium hydroxide
- hint: For the dosage of KOH, the quality (concentration) of the raw material has to be considered (see also „make-up values“)

Iron (Fe) – Analysis by Photometry

- instruments: photometer with adjustable wave length (345 nm)
and a 10 mm rectangular cuvette or
photometer with 340 nm filter and a 11 mm round cuvette
3 ml and 30 ml volumetric pipettes
50 ml volumetric flask
- reagents: conc. hydrochloric acid, p.a. (37 %)
- procedure: 1. Pipette 3 ml filtrated bath sample into a 50 ml volumetric flask.
2. Add exactly 30 ml hydrochloric acid cautiously (volumetric pipette) and mix well. Attention: gas evolution! The solution gets very hot!
3. Cool down to room temperature in a water bath (approx. 15 min).
4. Fill up the flask to its final volume with DI-water and mix well.
5. Pour some ml of this solution into the cuvette and measure the absorbance against DI-water.
The measurement should be done within one hour after the acid addition.
- calculation: absorbance · 294.1 = mg/l iron (345 nm, rectangular cuvette)
or
extinction · 256.4 = mg/l iron (340 nm, 11 mm round cuvette)

Iron (Fe) – Analysis by AAS

| | |
|--------------|--|
| instrument: | atomic absorption spectrometer (AAS) |
| reagents: | hydrochloric acid (conc.) p.a. iron standard solutions |
| procedure: | <ol style="list-style-type: none">1. Pipette 1 ml filtrated bath sample into a 100 ml volumetric flask.2. Acidify with 5 ml conc. hydrochloric acid.3. Fill up to 100 ml with DI-water.4. Measure in comparison to standards. |
| calculation: | measured value in ppm · 100 = mg/l iron |
| correction: | An addition of 1 ml/l SurTec 714 Fe-C increases the Fe content by 20 mg/l. |

SurTec 714 C Complexing Agent – Analysis by Titration

| | |
|--------------|--|
| reagents: | copper chloride solution (50 g/l $\text{CuCl}_2 \cdot 2 \text{H}_2\text{O}$) sodium hydroxide solution (10 %) sulfuric acid (1:1) potassium iodide 0.1 N sodium thiosulfate solution starch solution (1 %, freshly made) |
| procedure: | <ol style="list-style-type: none">1. Pipette 10 ml bath sample into a 100 ml volumetric flask.2. Add 5 ml sodium hydroxide solution.3. Dilute with approx. 50 ml deionised water.4. While rotating, add 10 ml 0.1 N copper chloride solution (solution becomes dark blue with light blue precipitation).5. Fill up the flask to 100 ml with deionised water.6. Mix well for 1 minute.7. Pour the complete solution into a dry 250 ml beaker.8. While stirring, heat up to 50°C.9. Cool down the solution.10. Filter through a blue ribbon filter.11. Pipette 50 ml of the filtrate into a new 250 ml beaker.12. Dilute with deionised water up to approx. 150 ml.13. Add 2 g potassium iodide.14. While stirring, heat up to 50°C.15. Acidify with 5 ml half conc. sulfuric acid (solution colour turns to a turbid light brown).16. Add some ml of starch solution (solution becomes black).17. Titrate with 0.1 N sodium thiosulfate solution to a permanent colour change to colourless-turbid (discolouration must be stable for at least 1 minute). |
| calculation: | consumption of thiosulfate in ml · 12.56 = ml/l SurTec 714 C |

Potassium Carbonate (K_2CO_3) – Analysis by Titration

- reagents: barium nitrate solution (5 %)
 1 N hydrochloric acid
 1 N sodium hydroxide solution
indicator: methyl orange solution (0.04 %)
- procedure: 1. Pipette 10 ml bath sample into a 250 ml Erlenmeyer flask.
 2. Dilute with 50 ml deionised water and boil the solution.
 3. Add 75 ml barium nitrate solution.
 4. After settle down of the precipitate, filtrate with a fine-grained filter paper (blue ribbon) and wash with hot deionised water.
 5. Cautiously put the filter with the precipitate into a 250 ml Erlenmeyer flask.
 6. Add 100 ml deionised water.
 7. Add exact 20 ml 1 N hydrochloric acid.
 8. Boil the solution shortly.
 9. After cooling down, add 3 drops of indicator.
 10. Titrate back the excess of hydrochloric acid with 1 N sodium hydroxide (colour change from red to orange-yellow).
- calculation: $(20 - \text{consumption in ml}) \cdot 6.91 = \text{g/l potassium carbonate}$

Analysis of the Fe Content in the Deposited Layer

- procedure: 1. Plate together with the normal parts one piece of copper tube (approx. 3 cm) in the plating line.
 2. Do not passivate the plated copper piece, but rinse it well and dry it.
 3. Weigh out the plated copper tube at the analytical balance (**A**).
 4. Put the copper tube into a small beaker and add half concentrated hydrochloric acid until the part is completely immersed.
 5. Wait until the zinc/iron layer is completely stripped off the part.
 6. Take out the part and wash it with small amounts of deionised water into the solution. Dry and weigh out the copper tube again (**B**). The difference (**A - B**) should be approx. 0.2-0.4 g.
 7. Transfer the hydrochloric acid completely into a volumetric flask (100 ml).
 8. Complete the solution in the flask with deionised water to the final volume and mix the solution very well.
 9. Without further dilution, analyse the iron content of the solution by AAS (value in ppm = **C**).

calculation: $C / [(A - B) \cdot 100] = \% \text{ Fe}$

hint: For a first indication of the iron content in the deposited layer can be used a freshly made-up bath of Black Chromating SurTec 698 (independent of the passivation used in the plating line). Chromating a freshly Zn/Fe plated test part therein, the Fe content of the layer can be estimated by the help of the Trouble Shooting List (page 10).

Hull Cell Test

Perform all tests in a standard 250 ml Hull cell. For the Hull cell tests it is best to use inert anodes to avoid any depositions on the zinc anode surface. Before plating, prepare well the Hull cell panel (pickling and anodic cleaning), it has to be free of zinc and without oil.

Use the zinc/iron electrolyte at its normal operating temperature (the temperature of the bath in the line). Plate the freshly cleaned panel in the Hull cell at 2 A for 15 min. Rinse the panel with tap water, activate it in 0.5 %vol nitric acid (5-10 s), rinse again and passivate it in an actual sample of the passivation bath which is used in the line (conditions must be the same as in the line). Rinse again and dry it with hot or compressed air.

Examine the panel according to the trouble shooting list. Perform a final Hull cell test with all necessary actions in effect before adding something to the bath.

Because of the high current applied, it is recommended to use fresh electrolyte samples for each variation in the Hull cell test.

Conversion of a Zinc/Iron Electrolyte to SurTec 714

For a complete conversion test, at least 3-4 litres original electrolyte are necessary.

The panels should be plated at 2 A for 15 min, according to the description at the chapter „Hull Cell Test“. But for conversion trials, a freshly made up black passivation has to be used.

First Indication Test:

1. Plate a SurTec 714 panel in a fresh laboratory electrolyte according to the instructions of the chapter "Hull Cell Test".
2. Plate an original panel in the actual electrolyte without any additions. If the original panel is already bright, you can only try the overdosage effect, if it is less bright than panel 1, you can already get an indication on the receptivity of the old system for SurTec 714.
3. Add 1 ml/l SurTec 714 I and 1 ml/l SurTec 714 Fe-C to the 250 ml Hull cell 2 and plate again.
4. Depending on the result of the last panel, add once again SurTec 714 Fe-C, or SurTec 714 C (If panel 3 is black and brightness is enough, add once again 1 ml/l SurTec 714 Fe-C. If panel 3 is not really black but green or brown, add 2 ml/l SurTec 714 Fe-C. If panel 3 is black and matt, add 4 ml/l SurTec 714 C.)

If there is a positive effect (panel 3 and 4 are the same as or better than panel 2), a conversion should be possible without an immediate problem.

Mid Term Compatibility:

1. Fill 1.8 litre original bath into a 2 l beaker, hang in a small Hull cell anode (therefore a zinc anode is necessary, no inert anode!) and a pre-treated jiggle cell panel (or, if not available, a 15 cm long and about 4 cm wide steel sheet) as a cathode, put it on a magnetic stirrer and stir slowly, connect anode and cathode to the rectifier and plate with 1 A for 6 h.
2. Analyse the zinc content and adjust it to the desired value, using zincate solution (e.g. SurTec 700 EK), or deionised water. Fill 250 ml of this adjusted electrolyte into a Hull cell and plate a Hull cell panel according to the instructions of the chapter "Hull Cell Test".
3. Add 2 ml/l SurTec 714 I and 1.5 ml/l SurTec 714 Fe-C to the Hull cell and repeat the test.
4. Repeat the addition of SurTec 714 Fe-C until a good result is obtained.

Long Term Compatibility:

1. Prepare 1 litre of a fresh SurTec 714 electrolyte with the desired values (see first page).
2. Prepare 5 dilutions with a total volume of 250 ml each of the actual (untreated) electrolyte with the fresh electrolyte:
 - a) 225 ml original bath + 25 ml fresh electrolyte
 - b) 175 ml original bath + 75 ml fresh electrolyte
 - c) 125 ml original bath + 125 ml fresh electrolyte
 - d) 75 ml original bath + 175 ml fresh electrolyte
 - e) 25 ml original bath + 225 ml fresh electrolyte

and plate a Hull cell panel in each electrolyte.

If the first indication test (see above) had shown a lack of brightness, or a green layer after passivation, add 0.2 ml SurTec 714 Fe-C for every 25 ml of the actual electrolyte. If the first indication test had shown a matt deposition, add 0.6 ml SurTec 714 C for every 25 ml of the actual electrolyte.

There should not be any negative effect in any dilution. If e.g. the panel plated in bath c) had an unexpected appearance, possible problems must be expected after about 5 weeks of conversion (barrel application) resp. 15-20 weeks (rack application).

If every dilution can be adjusted to a good panel, no problems should be expected by the conversion itself.

Because of the mixture of different complexing agents while a conversion of a zinc/iron bath, carry out a test to prove the flitter tendency. Therefore, at least a last panel of each of the three conversion steps has to be tested as follows:

1. Plate a panel in the last bath conditions at 2 A for 30 min.
2. Rinse, activate and passivate it.
3. After drying, put it for 24 h into an oven at 60°C (not at higher temperature).
4. Check the next day the flitter activity.

The long plating time is necessary to get a higher layer thickness, which will have some tension inside - at too much tension, flitter will appear.

To be sure, that the flitter is a problem of the mixture of different complexing agents, a first test with the original bath should be prepared.

Consumption and Stock Keeping

The additives are consumed by drag-out and electrochemically, e.g. by anodic oxidation and cathodic built-in. The consumptions are consequently depending on the specific volume of drag-out.

The following values per 10,000 Ah can be taken as estimated average consumption:

| | | |
|-----------------|----------------|--------------------------------|
| SurTec 714 I | approx. 0.5 l | |
| SurTec 714 Fe-C | approx. 2 l | (at 0.5 % Fe inside the layer) |
| SurTec 700 L | 0-0.2 l | (as required) |
| (SurTec 700 S | approx. 0.1 l) | |

In order to prevent delays in the production process, per 1,000 l bath, the following amounts should be kept in stock:

| | |
|-----------------|--------|
| SurTec 714 I | 25 kg |
| SurTec 714 Fe-C | 25 kg |
| SurTec 714 C | 25 kg |
| SurTec 714 Fe | 25 kg |
| SurTec 700 L | 25 kg |
| (SurTec 700 S | 25 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 714 I | - | WHC 3 |
| SurTec 714 Fe-C | - | WHC 1 |
| SurTec 714 C | - | WHC 1 |
| SurTec 714 Fe | C - Corrosive | WHC 1 |
| SurTec 700 L | C - Corrosive | WHC 1 |
| SurTec 700 S | - | WHC 1 |
| SurTec 700 EK | C - Corrosive N - Dangerous for the environment | WHC 1 |

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>

Trouble Shooting

| problem | possible cause | remedy |
|---|--|--|
| olive-green appearance of black chromate SurTec 698 | a) iron content in the electrolyte is too low | analyse and adjust the iron content to 180 mg/l with SurTec 714 Fe-C |
| | b) zinc content in the electrolyte too high | adjust the zinc generator |
| | c) temperature of the electrolyte is too low | adjust the temperature to at least 25°C |
| | d) immersion time in the black chromate is too short | increase the immersion time |
| | e) pH-value of the black chromate SurTec 698 is too high | adjust to pH 1 with sulfuric acid |
| | f) SurTec 714 C Complexing Agent is too high | reduce SurTec 714 C by work-out |
| dull-black appearance of the black chromate SurTec 698 | a) iron content in the electrolyte is too high | increase SurTec 714 C Complexing Agent, finally work-out Fe |
| | b) zinc content in the electrolyte is too low | adjust the zinc generator |
| | c) immersion time in the black chromate is too long | decrease the immersion time |
| | d) pH-value of the black chromate SurTec 698 is too low | adjust to pH 1 with NaOH |
| | e) current density is too low | increase current density to at least 1 A/dm ² (barrel) or 2 A/dm ² (rack) |
| dull-black appearance of the black passivation SurTec 695 | a) iron content in the electrolyte is too high | analyse and adjust the iron content to 180 mg/l with SurTec 714 Fe-C |
| | b) zinc content in the electrolyte is too low | adjust the zinc generator |
| | c) temperature of the SurTec 714 electrolyte is too low | warm up the bath to at least 25°C |
| | d) immersion time in the black passivation is too long | decrease the immersion time |
| | e) pH-value of the black passivation SurTec 695 is too low | adjust the pH-value of SurTec 695 to pH 1.9 with Na-carbonate |
| | f) too high concentration of SurTec 714 C Complexing Agent | work out excessive complexing agent |
| bad throwing power, burnings or bad coverage | lack of additive SurTec 714 I Carrier | add SurTec 714 I, check in the Hull cell before |
| spotted chromate layer | partial passivation of the Zn/Fe-deposit in the rinsing water before entering into the black chromate bath | decrease the number of steps and speed up all left steps between Zn/Fe-deposition and black chromate |
| reduced current efficiency | overdosage of SurTec 700 L LCD Booster | work out |