

SurTec® 617

Tri-Cationic Phosphating

Properties

- for spray and immersion application
- suitable for steel and zinc substrates
- produces micro-crystalline layers
- uniform deposition
- excellent corrosion protection
- forms only very small amounts of sludge
- adherent deposition of predominantly phosphophyllite crystals
- excellently prepares the surface for subsequent coatings
- very inert layer, suitable for electrophoretic lacquering

Application

The process SurTec 617 includes the following products:

- SurTec 617 M Make Up Solution is used for make-up
- SurTec 617 R Replenishment Solution is used for replenishment
- SurTec 612 S Accelerator for Phosphatings
- NaOH (99 %), only is used to neutralise too high amounts of Free Acid

| make-up values: | <i>spray</i> | <i>immersion</i> |
|-----------------|--------------|------------------|
| SurTec 617 M | 38 ml/l | 38 ml/l |
| SurTec 612 S | 1.0 ml/l | 1.0 ml/l |

| analytical values: | <i>spray</i> | <i>immersion</i> |
|--------------------|---|------------------|
| Total Acid | 20 - 25 Points | 20 Points |
| Free Acid | 0.8-1.3 Points | 1.3-1.5 Points |
| | to neutralise 1 Point, add 0.4 g/l NaOH | |
| SurTec 612 S | 4.5 Points | (3-6 Points) |
| zinc | 1.2 g/l | (0.9-1.5 g/l) |
| nickel | 0.50 g/l | (0.45-0.60 g/l) |
| manganese | 0.04 g/l | (0.03-0.05 g/l) |
| phosphoric acid | 12 g/l | (11.5-14.7 g/l) |

make-up:

Steps for make-up:

1. Dissolve SurTec 617 M Make Up Solution in deionised water with strong agitation.
2. Pre-dilute SurTec 612 S Accelerator in water and add it to the bath:
e.g. for 1000 l bath: dissolve 1.0 l SurTec 612 S in 5 l water.
3. Analyse the Free Acid Points and adjust them by adding pre-diluted caustic soda very carefully and slowly.

| | | |
|--------------------|--|----------------------|
| temperature: | 48°C | (45-55°C) |
| application time: | <i>immersion process</i> | <i>spray process</i> |
| | 3-5 min | 1-5 min |
| agitation: | In case of immersion, a slight agitation by stirring systems or pumping is recommended. Air agitation is not recommendable since atmospheric oxygen would oxidise SurTec 612 S Accelerator and more sludge is formed and the layer is less uniform. | |
| spraying pressure: | 0.8 bar | (0.6-1.0 bar) |
| tank material: | stainless steel | |
| filtration: | periodically remove sludge: filter sludge and return filtrate to the bath | |
| heating: | necessary: stainless steel heaters (type 1.4571) or steel coated with PTFE | |
| cooling: | not required | |
| exhaust: | required for worker's protection | |
| hints: | The deposited layer weight is very stable. In case of spray application, it varies from 1.5 to 2.0 g/m ² . In case of immersion, it varies from 1.8 to 2.2 g/ m ² . | |

The deposited phosphate layer is excellently suitable as a basis for electrophoretic immersion lacquering. The weight loss of the phosphate layer during the lacquering is only 0.3-0.8 % of the overall weight of the phosphate layer (conventional phosphating processes usually show a weight loss of 5 %). Hence, besides best corrosion resistance of the layer system, also the electrophoretic lacquering system is not contaminated by dissolved phosphating layer.

recommended process sequence:

1. degreasing, e.g. SurTec 168/089 or SurTec 138/089
2. rinsing
3. grain refiner, e.g. SurTec 610 V
4. **phosphating with SurTec 617**
5. rinsing
6. DI-water rinsing
7. drying < 110°C

The rinsing methods have to be adapted to the plating line.

Maintenance and Analysis

Analyse and adjust Free Acid, Total Acid and SurTec 612 S regularly.

In case of high throughput, use an automatic dosing system to avoid varying concentrations.

Replenish evaporation losses continuously by adding DI-water.

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.

Total Acid (TA) – Analysis by Titration

- reagents: 0.1 N sodium hydroxide solution
indicator: phenolphthalein
- procedure: 1. Pipette 10 ml bath sample into a 250 ml Erlenmeyer flask.
2. Dilute with approx. 50 ml deionised water.
3. Add 5 drops of indicator.
4. Titrate with 0.1 N sodium hydroxide solution from colourless to light pink.
- calculation: consumption in ml = TA-Points
- correction: To adjust the Total Acid Points, use SurTec 617 R:
add 1.4 ml/l SurTec 617 R for each missing Total Acid Point

Free Acid (FA) – Analysis by Titration

- reagents: 0.1 N sodium hydroxide solution
indicator: bromophenol blue
- procedure: 1. Pipette 10 ml bath sample into a 250 ml Erlenmeyer flask.
2. Dilute with approx. 50 ml deionised water.
3. Add 5 drops of indicator.
4. Titrate with 0.1 N sodium hydroxide solution from yellow to blue.
- calculation: consumption in ml = FA-Points
- correction: To neutralise 1 Free Acid Point:
add 0.4 g/l NaOH (pre-diluted with water, 10 %)

SurTec 612 S Accelerator – Analysis by Titration

- reagents: 0.1 N potassium permanganate solution
sulfuric acid (50 %)
urea p.a.
- procedure: 1. Pipette 50 ml bath sample into a 250 ml Erlenmeyer flask.
2. Add 1-2 ml sulfuric acid.
3. Titrate with 0.1 N potassium permanganate solution to a stable pink colouration (for at least 15 s).
consumption in ml = **A**
4. Pipette 50 ml bath sample into another 250 ml Erlenmeyer flask.
5. Add 1-2 ml sulfuric acid.
6. Add 4 g urea.
7. Stir the solution until urea has dissolved and wait approx. 5 min.
8. Titrate with 0.1 N potassium permanganate solution to a stable pink colouration (for at least 15 s).
consumption in ml = **B**
- calculation: **A - B = SurTec 612 S - Points**
- correction: For each missing Point:
add 0.27 ml/l SurTec 612 S

Manganese (Mn) – Analysis by Titration

- reagents: 0.1 N silver nitrate solution
0.01 N sodium oxalate solution
sulfuric acid (50 %)
disodium phosphate heptahydrate
sulfuric acid (98 %)
ammonium peroxodisulfate
- procedure: 1. Pipette 5 ml bath sample into a 500 ml Erlenmeyer flask.
2. Dilute with approx. 200 ml DI-water.
3. Add 15 ml sulfuric acid (50 %).
4. Add 3 g disodium phosphate heptahydrate.
5. Heat up until everything has dissolved.
6. Add 10 ml sulfuric acid (98 %).
7. Add 3 g ammonium peroxodisulfate.
8. Heat up until everything has dissolved.
9. Add 5 ml 0.1 N silver nitrate solution and boil 20 min.
10. Titrate with 0.01 N sodium oxalate solution from violet to pink.
- calculation: consumption in ml · 0.042 = g/l Mn

Zinc (Zn) and Nickel (Ni) – Analysis by Titration

- reagents: 0.01 mol/l EDTA solution
0.01 M magnesium sulfate solution
buffer solution pH 10
Eriochrome Black T indicator
buffer solution pH 5.2
dimethylene glyoxim solution (1 %)
tetrachloroethylene
- procedure: 1. Pipette 5 ml bath sample into a 500 ml Erlenmeyer flask.
2. Dilute with approx. 30 ml DI-water.
3. Add exactly 50 ml 0.01 M EDTA solution.
4. Add 20 ml buffer solution pH 10.
5. Add a spatula tip of Eriochrome Black T indicator.
6. Titrate with 0.01 M magnesium sulfate solution to blue.
consumption in ml = **A**
7. Pipette 5 ml bath sample into a 500 ml Erlenmeyer flask with plug.
8. Dilute with approx. 50 ml DI-water.
9. Add 10 ml buffer solution pH 5.2.
10. Add 10 ml dimethylene glyoxim solution.
11. Add 3 ml tetrachloroethylene.
12. Plug the Erlenmeyer flask and shake 15 s.
13. Unplug and rinse the walls with DI-water.
14. Add exactly 50 ml 0.01 M EDTA solution.
15. Add 30 ml buffer solution pH 10.
16. Add a spatula tip of Eriochrome black T indicator.
17. Titrate with 0.01 M magnesium sulfate solution to blue.
consumption in ml = **B**
- calculation: $[(50 - \mathbf{B}) - \text{Mn in g/l} / 0.064] \cdot 0.112 = \text{g/l Zn}$
 $(\mathbf{B} - \mathbf{A}) \cdot 0.061 = \text{g/l Ni}$

Phosphoric Acid (P₂O₅) – Analysis by Titration

- reagents: 0.1 M sodium hydroxide solution
potassium oxalate solution (28 %)
thymolphthaleine indicator
dimethylene yellow indicator
- procedure: 1. Pipette 10 ml bath sample into a 500 ml Erlenmeyer flask.
2. Dilute with approx. 150 ml DI-water.
3. Add 20 ml potassium oxalate solution.
4. Add 15 drops thymolphthaleine indicator.
5. Titrate with 0.1 M sodium hydroxide solution from colourless to blue.
- consumption in ml = **A**
6. Pipette 10 ml bath sample into a 250 ml Erlenmeyer flask.
7. Dilute with approx. 75 ml DI-water.
8. Add 5 drops dimethylene yellow indicator.
9. Titrate with 0.1 M sodium hydroxide solution from red to yellow.
- consumption in ml = **B**
- calculation: $(A - B) \cdot 1.135 = \text{g/l P}_2\text{O}_5$

Layer Weight Determination

- reagents: chromic acid (5 %)
- procedure: 1. Phosphate a test part of known surface area, rinse it with DI-water, dry and weigh it by means of an analytical balance with an accuracy of 0.1 mg (= **A**).
2. Remove the phosphate layer in 5 % chromic acid for 10 min at 75°C. Rinse the part, dry it and weigh again (= **B**).
- calculation: $[A - B] / \text{area in m}^2 = \text{g/ m}^2$

Technical Specification

| (at 20 °C) | Appearance | Density (g/ml) | pH-value (conc.) |
|--------------|--------------------------|-------------------|------------------|
| SurTec 617 M | liquid, green, clear | 1.450 (1.43-1.47) | < 2 |
| SurTec 617 R | liquid, green, clear | 1.465 (1.43-1.48) | < 2 |
| SurTec 612 S | liquid, yellowish, clear | 1.214 (1.19-1.24) | 11.5 (10-12.5) |

Ingredients

SurTec 617 M and SurTec 617 R

- phosphoric acid
- nitric acid
- zinc salts
- nickel salts
- manganese salts

SurTec 612 S

- nitrite salts

Consumption and Stock Keeping

The consumption depends heavily on the drag-out. To determine the exact amounts of drag-out, see [SurTec Technical Letter 11](#).

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

| | |
|--------------|--------|
| SurTec 617 M | 100 kg |
| SurTec 617 R | 100 kg |
| SurTec 612 S | 10 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 617 M | T - Toxic N - Dangerous for the environment | WHC 3 |
| SurTec 617 R | T - Toxic N - Dangerous for the environment | WHC 3 |
| SurTec 612 S | T - Toxic N - Dangerous for the environment | 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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