

# µChem 450

## Nickel/Iron Electrolyte

### Properties

- process especially developed for micro technology
- deposits nickel-iron layers with iron contents up to 45 %
- the electrolyte is already worked in, after addition of the required amount of iron salt and heating up to working temperature, the electrolyte can be used directly

### Application

The process µChem 450 includes the following products:

- µChem 450 Nickel/Iron Electrolyte is an already worked in electrolyte, but without iron (during storage, Fe(II) would be oxidised to Fe(III))
- µChem 450/1 Iron Salt is used to adjust the iron content in the bath
- µChem 450/2 Reduction Agent contains a reduction component in form of an organic acid
- µChem 450/3 Additive contains a compound to increase the structure properties of the precipitated layer
- µChem 450/4 Wetting Agent is necessary for a homogenous electrolyte film on top of the parts

make-up values:

µChem 450 Ni/Fe Electrolyte	undiluted	
µChem 450/1 Iron Salt	5-45 g/l	depending on the desired alloy composition
analytical values:		
nickel	50 g/l	(100-110 g/l)
iron	1-9 g/l	depending on the desired iron alloy (adjust with µChem 450/1)
chloride	18 g/l	(12-20 g/l)
boric acid	45 g/l	(40-45 g/l)

make-up:

Steps for make-up:

1. Fill the already worked in µChem 450 Nickel/Iron Electrolyte into the tank.
2. Pre-dilute the calculated amount of µChem 450/1 Iron Salt in deionised water or in a small amount of the electrolyte and add it to the electrolyte tank while stirring.
3. Heat up to working temperature.
4. Now the electrolyte is ready to use.

temperature: 55°C

pH-value: 3.5  
adjust with sulfuric acid

cathodic  
current density: 3 A/dm<sup>2</sup>

deposition rate:	30 $\mu\text{m/h}$ at 3 $\text{A/dm}^2$
ratio	
anode : cathode:	3 : 1
anodes:	Clippings or bullets out of pure nickel in baskets of titanium with anode bags out of PP; pure iron anodes with PP anode bags. The anode surface ratio nickel : iron should be about 8 : 1.
agitation:	strong circulation is necessary
tank material:	steel with plastic coating or strengthened plastic tanks
filtration:	continuous filtration with 5-6 bath volumes per hour; pore size: 0.2 $\mu\text{m}$
heating:	PTFE coated heating or out of ceramics (no lead, no stainless steel!)
exhaust:	recommended for workers protection
hints:	To prevent an oxidation of Fe(II) to Fe(III), it is necessary to blow some nitrogen into the bath. Additionally, the electrolyte should always be under current (approx. 0.15 $\text{A/dm}^2$ ), also to prevent the oxidation.  At longer working breaks, it is recommended to remove the iron anodes out of the electrolyte.

## Maintenance and Analysis

Compensate evaporation losses with deionised water.

The concentrations of iron and nickel have to be kept constant: they regulate the composition of the nickel alloy. The iron content can be regulated by adjusting the surface of the iron anode, or by adding  $\mu\text{Chem 450/1}$  Iron Salt. To raise the iron content about 1 g/l, an addition of 5 g/l  $\mu\text{Chem 450/1}$  is necessary. The iron salt has to be pre-diluted in deionised water or in a small amount of the electrolyte.

In case of pittings or not plated micro structures,  $\mu\text{Chem 450/4}$  Wetting Agent has to be added (recommendation: 2 ml/l). The wetting agent is also consumed by the throughput (see "Consumption and Stock Keeping").

### Sample Preparation

Take the sample at a homogeneously mixed position and let it cool down to room temperature.

### Nickel – Analysis by Titration

reagents:	0.1 mol/l EDTA (Titrplex III) ammonia solution (conc.) triethanole amine solution (50 %vol) indicator: Murexid (mixed 1:100 with NaCl)
procedure:	<ol style="list-style-type: none"> <li>1. Pipette 2 ml bath sample into a 250 ml Erlenmeyer flask.</li> <li>2. Dilute to approx. 50 ml with deionised water.</li> <li>3. Add 15 ml triethanole amine solution.</li> <li>4. Add 15 ml conc. ammonia solution.</li> <li>5. Add a spatula tip of indicator while stirring.</li> <li>6. Titrate with 0.1 M EDTA to blue-violet.</li> </ol>
calculation:	consumption in ml $\cdot$ 2.934 = g/l nickel

### Iron – Analysis by Titration

- reagents: 0.1 mol/l ceric sulfate solution  
6 mol/l sulfuric acid  
ammonia solution (conc.)  
ferroin indicator solution
- procedure: 1. Pipette 5 ml bath sample into a 250 ml Erlenmeyer flask.  
2. Dilute with approx. 75 ml deionised water.  
3. Add 20 ml sulfuric acid (6 mol/l).  
4. Add 15 ml ammonia solution.  
5. Add 6 drops of ferroin indicator solution while stirring.  
6. Titrate with 0.1 mol/l ceric sulfate solution from light red to blue-green.
- calculation: consumption in ml · 1.118 = g/l iron
- correction: rise by 0.1 g/l = addition of 0.5 g/l µChem 450/1

### Boric Acid – Analysis by Titration

- reagents: potassium hexacyanoferrat(II) solution (200 g/l in water)  
bromocresol purple solution (1 g/l in water)  
mannitol  
0.1 N NaOH
- procedure: 1. Pipette 2 ml bath sample into a 250 ml Erlenmeyer flask.  
2. Dilute to approx. 30 ml with deionised water.  
3. Add 10 ml potassium hexacyanoferrat(II) solution.  
4. Add 6 drops brome cresole red solution. The bath sample should be blue now. If it is still green, titrate with 0.1 N NaOH solution to blue (the consumption has to be ignored!).  
5. Add 5 g mannitol.  
6. Titrate with 0.1 N NaOH solution from blue-green to blue-violet.
- calculation: consumption in ml · 3.09 = g/l boric acid

### Chloride – Analysis by Titration

- reagents: 0.1 N silver nitrate solution  
sodium bicarbonate  
indicator: potassium chromate solution (10 %)
- procedure: 1. Pipette 2 ml bath sample into a 250 ml Erlenmeyer flask.  
2. Dilute with approx. 50 ml deionised water.  
3. Add 20 drops of indicator.  
4. Add 1-2 g sodium bicarbonate.  
5. Titrate with 0.1 N silver nitrate solution from white to yellow-brown.
- calculation: consumption in ml · 1.775 = g/l chloride

## Technical Specification

(at 20 °C)	Appearance	Density (g/ml)	pH-value (conc.)
µChem 450	liquid, green	1.177 (1.15-1.20)	3-4
µChem 450/1	powder, green	600 kg/m <sup>3</sup>	3-4 (at 5 %)
µChem 450/2	powder, white	900 kg/m <sup>3</sup>	< 3 (at 5 %)
µChem 450/3	liquid, colourless - pale yellow	1.086 (1.07-1.10)	3-5
µChem 450/4	liquid, colourless	1.003 (0.97-1.03)	3-5

## Ingredients

µChem 450:

- boric acid

## 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](#).

After plating of about 100 Ah, the following additions are necessary:

µChem 450/2 Reduction Agent	3 g
µChem 450/3 Additive	8 ml
µChem 450/4 Wetting Agent	2 ml

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

µChem 450/1 Iron Salt	2 kg
µChem 450/2 Reduction Agent	2 kg
µChem 450/3 Additive	2 kg
µChem 450/4 Wetting Agent	1 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>
µChem 450	T - Toxic N - Dangerous for the environment	WHC 2
µChem 450/1	Xn - Harmful	WHC 1
µChem 450/2	Xi - Irritant	WHC 1
µChem 450/3	-	-
µChem 450/4	-	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>