

# riag Ni 141

## Bright nickel process for ductile, bright deposits, particularly for technical applications

**riag Ni 141 Additive** is an additive to reduce internal stress in Sulfamate-nickel electrolytes and therefore is particularly used when plating electronic components, including printed circuits and electroforming. Pitting can be monitored by adding wetting agent. The electrolyte can be used in both rack and barrel applications.

### Properties

- Ductile and bright deposits with slight tendency to levelling
- Uniform brightness in nearly all current densities
- Hardness of deposit: ca. 400 HV, lower temperatures and lower pH-values increase the hardness
- Internal stress of nickel deposit: 0 – 35 kg/cm<sup>2</sup> tensile stress  
Comparison with regular „Watts“-nickel electrolyte: ca. 1,000 kg/cm<sup>2</sup> tensile stress

**riag Ni 141** deposits produce an excellent prelayer when coating precious metals, mainly because a hard but ductile and corrosion-resistant primary layer is created and it also substantially improves the abrasion resistance.

### Make up boric acid-free (US Patent No.: 11,396,711)

	Range	Optimum
Nickel sulfamate concentrate (185 g/L Ni)	270 – 450 mL/L	370 mL/L
Nickel chloride (NiCl <sub>2</sub> x 6 H <sub>2</sub> O)	7 – 17 g/L	12 g/L
<b>riag Ni 149 Make up</b>	80 – 120 mL/L	100 mL/L
<b>riag Ni 141 Additive*</b>	0 – 15 mL/L	10 mL/L
<b>riag Ni 138 Tenside M* / riag Ni 139 Tenside L*</b>	1 – 10 mL/L	2 mL/L

\* depending on customer-specific process requirement

## Operating values

	Range	Optimum
Nickel ( $\text{Ni}^{2+}$ )	60 – 90 g/L	75 g/L
Chloride ( $\text{Cl}^-$ )	2 – 5 g/L	3.5 g/L
<b>riag Ni 149 Make up</b>	80 – 120 mL/L	100 mL/L
pH-value	3.0 – 5.2	customer-specific

## Make up

The tank is filled with deionised water to 1/3 of the final volume, the nickel chloride is added and solved in water. Afterwards the nickel sulfamate solution and **riag Ni 149 Make up** are added. The tank is filled with deionised water up to 95 % of its final volume. After adding **riag Ni 141 Additive** and **riag Ni 138 Tenside M / riag Ni 139 Tenside L** the remaining water is added and heated to the set temperature. After adjustment of the desired pH-value the electrolyte is ready for operation.

## Alternative make up containing boric acid

	Range	Optimum
Nickel sulfamate concentrate (185 g/L Ni)	300 – 480 mL/L	400 mL/L
Nickel chloride ( $\text{NiCl}_2 \times 6 \text{ H}_2\text{O}$ )	7 – 17 g/L	12 g/L
Boric acid	30 – 50 g/L	40 g/L
<b>riag Ni 141 Additive*</b>	0 – 15 mL/L	10 mL/L
<b>riag Ni 138 Tenside M* / riag Ni 139 Tenside L*</b>	1 – 10 mL/L	2 mL/L

\*depending on customer-specific process requirements

## Operating values

	Richtwerte	Optimum
Nickel ( $\text{Ni}^{2+}$ )	60 – 90 g/L	75 g/L
Chloride ( $\text{Cl}^-$ )	2 – 5 g/L	3.5 g/L
Boric acid ( $\text{H}_3\text{BO}_3$ )	30 – 50 g/L	40 g/L
pH-value	3.0 – 4.5	3.6

## Make up

The tank is filled with deionised water to 1/3 of the final volume, the nickel chloride is added and solved in water. Afterwards the nickel sulfamate solution and the boric acid are added. The tank is filled with deionised water up to 95 % of its final volume. To completely solve the boric acid the electrolyte has to be heated-up and stirred. After adding **riag Ni 141 Additive** and **riag Ni 138 Tenside M / riag Ni 139 Tenside L** we recommend a filtration of the electrolyte during 2 – 4 hours.

## Operating parameters

Temperature:	55 °C (50 – 65 °C)	
pH-value:	Boric acid free:	3.0 – 5.2 (customer-specific)
	Containing boric acid:	3.0 – 4.5 (customer-specific)
	The pH-value of the boric acid-free version should always be measured at operating temperature.	
	Increased pH-values influence the anode solubility. The use of (depolarised) anodes and halogen salts (chloride or bromide) can compensate for this.	
Cathodic current density:	Rack :	4 A/dm <sup>2</sup> (1 – 10 A/dm <sup>2</sup> )
	Barrel:	0.75 A/dm <sup>2</sup> (0.5 – 1.0 A/dm <sup>2</sup> )
Current efficiency:	100 %	
Deposition rate:	Rack:	at 3 A/dm <sup>2</sup> ca. 0.6 µm/min.
	Barrel:	at 0.75 A/dm <sup>2</sup> ca. 0.15 µm/min.
	At 4 A/dm <sup>2</sup> , 50 µm per hour are deposited.	
Agitation:	Electrolyte agitation 2 – 3 m/min. or air agitation with oil-free compressed air	
Anodes:	„S“ Nickel with polypropylene bags. Titanium baskets may be used.	
Tanks:	Semi hard PVC or polypropylene	
Filtration:	Continuous filtration is essential using 3 – 5 µm polypropylene filter cartridges	
Heating:	Porcelain immersion heaters or titanium heating coils with thermostatic control	
pH-value adjustment:	Adding <b>riag Ni 149 Buffer</b> lowers the pH-value. To lower the pH-value, add <b>riag Ni 149 Buffer</b> first. Only afterwards, if necessary, use sulfamic acid. To increase the pH only use nickel carbonate, never ammonia or ammonium compounds.	
Metallic contamination:	Metallic contamination can be removed by frequent selective plating-out at 0.1 – 0.3 A/dm <sup>2</sup> . The filter pump should be on with the filter outlet directed at the panels. This will ensure thorough electrolyte circulation and essential agitation at the same time.	
Rectifier:	Barrel up to approx. 8 volt, rack up to ca. 4 volt An ampere-hour meter simplifies the support	
Maintenance:	Nickel sulfamate, nickel chloride and <b>riag Ni 149 Make up</b> respectively boric acid should be analysed and corrected regularly.	
Additive consumption:	The additives are consumed during electrolytic reactions as well a drag-out losses. The use is therefore process-related.	
	<b>riag Ni 141 Additive</b>	1.0 – 1.5 L per 10 kWh
	<b>riag Ni Tenside M / L</b>	0.1 – 0.3 L per 10 kWh

**The use of boric acid results in an additional consumption of 20 - 30 % of brightener.**

## **Function of electrolyte components**

### **riag Ni 149 Make up**

Contains the buffer system neutralised with nickel carbonate and purified with activated carbon. Due to the addition during preparation, as well as during maintenance, the pH value in the electrolyte changes only slightly. The buffering effect is present over a very wide range and exceeds that of boric acid.

### **riag Ni 149 Buffer**

Is used to compensate for deficiencies in the buffer system. **riag Ni 149 Buffer** contains the same amount of active ingredient as **riag Ni 149 Make up**, but is not pH-neutral and reacts strongly acidic. It is added in aliquot quantities for drag-out, ideally when the pH should be adjusted with acid, instead of acid. By adding 5 mL/L the pH value of the nickel electrolyte drops by approx. 0.2 units. It is therefore recommended to add the buffer in small steps (empirical values). If there is a very large shortage in the buffer substance, we recommend switching to **riag Ni 149 Make up** (pH neutralised).

### **riag Ni 141 Additive**

We recommend frequent additions of **riag Ni 141 Additive** to produce uniform, bright coatings.

### **riag Ni Tenside M / L**

The **riag Ni Tensides** reduce the surface tension and prevent pitting. This lowers the adsorption of hydrogen molecules and dirt particles on the cathode surface, this pitting in the nickel coating is prevented.

## **Activated Carbon**

Continuous filtration over activated carbon is recommended. This can be done via a by-pass whereby the carbon will remove organic contaminants such as oils and breakdown products.

We recommend our **riag Carb SF** dust-free product which has an active surface area of 1500 m<sup>2</sup> /g. A significant contamination of the electrolyte can be removed by filtration in the bypass (filter pump with a filled sack of **riag Carb GR**). **riag Carb GR** is ideally suited for this purpose, as such treatment may be performed during the plating process. **riag** can provide such filter system.

### **riag Ni 147 Oxidant**

Iron contamination (pitting) can be removed effectively by additions of **riag Ni 147 Oxidant**. The maximum concentration of 0.5 g/L should not be exceeded. The salt is first dissolved in hot water and the iron is removed via the filter.

## General information

1. If required the internal stress of the deposit may be checked periodically with a spiral contractometer (Brenner) or a similar instrument.
2. In order to maintain the high deposit quality, which can be negatively influenced by foreign organic matter, we recommend treating the electrolyte periodically in the bypass or permanently by means of a filter pump (depending on the degree of contamination) with activated carbon. For this purpose, the product **riag Carb SF** has proven to be very effective.
3. Deposits with compressed stress are particularly well suited for the finishing of high-tensile steel and its components.

## Environmental considerations and product safety

All concentrates, rinse waters and waste solution must be treated and discharged in accordance with local effluent control regulations. Information can be gleaned from the material safety data sheets. Chemicals shall not be stored below 10 °C.

## Liability

This instruction manual was compiled with reference to the state of the art and all current standards, and is based on the long-term knowledge and experience of riag. However, riag cannot monitor compliance with this instruction manual and the methods described herein at the customer/end-user's premises. Work carried out with riag products must be adapted accordingly to meet local conditions. In particular, riag cannot accept liability for damage, loss or cost incurred due to a failure to adhere to this instruction manual, improper application of the methods, unauthorised technical modifications, insufficient maintenance or the absence of maintenance in respect of the requisite technical hardware or equipment, or in the event of use by unqualified personnel. riag is not liable for damage or loss caused by riag or its employees except where intention or gross negligence can be proved. riag furthermore reserves the right to make changes in relation to products, methods and the instruction manual without prior notice.

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This transaction is governed by material Swiss law (Law of Obligations), excluding private international law (conflict of laws) and intergovernmental treaties, specifically the CISG.

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## Analysis (Analytical Methods)

Sample preparation:

The sample must be taken from a well-mixed point and allowed to cool down to 25°C.

### riag Ni 149 Make up / riag Ni 149 Buffer

Reagents:		bromide – bromate solution 0,05 mol/L Br <sub>2</sub> (0,1 N) sodium thiosulfate solution 0.1 mol/L (0,1 N) potassium iodide solution 100 g/L hydrochloric acid (1:1) starch solution 1% in deion water
Procedure:	2 mL	electrolyte are transferred via pipette into a Erlenmeyer flask, pipette
	25 mL	bromide – bromate solution, add
	25 mL	hydrochloric acid and immediately close the flask with the stopper mix well and place the flask in a dark place for 10 minutes, add
	10 mL	potassium iodide solution, rinse the wall with deion. water and titrate with sodium thiosulfate solution to a light brown, add
	5 mL	starch solution and continue to titrate until the dark blue colour disappears

Calculation:  $(25 - \text{consumption in mL}) \times 13.2 = \text{mL/L riag Ni 149 Make up / riag Ni 149 Buffer}$

Replenishment of **riag Ni 149 Buffer** =  $(100 - \text{value of riag Ni 149 Buffer})$  in mL/L

\*Explanations for the supplement are included in the product description **riag Ni 149 Buffer**

### Nickel chloride hexahydrate

Reagents:		Silver nitrate solution 0.1 mol/L Potassium dichromate solution 5 %
Procedure:	5 mL 250 mL 50 mL 10 drops	electrolyte are transferred into a glass beaker and diluted with deionised water. of Potassium dichromate solution are added. Titration with 0.1 mol/L Silver nitrate solution from white yellow to a light brown end point.
Calculation:		Consumption of mL AgNO <sub>3</sub> x 2.380 = Nickel chloride hexahydrate (g/L) = <b>B</b> Consumption of mL AgNO <sub>3</sub> x 0.709 = Chloride (g/L)

**Nickel sulfamate  $\text{Ni}(\text{NH}_2\text{SO}_3)_2 \times 4 \text{H}_2\text{O}$** 

Reagents:	Buffer solution pH 10 Na <sub>2</sub> EDTA 0.1 mol/L Murexide (Sodium chloride 1: 100)	
Procedure:	10 mL 100 mL 10 mL 15 mL 100 mL 1 spatula tip	electrolyte are transferred via pipette into a measuring flask and filled- up to the mark with deionised water and mixed well of this mixture is given into a 250 mL glass beaker by pipette followed by Buffer solution addition deionised water and of Murexide are added The sample colour should then be deep yellow Titrate immediately with the Na <sub>2</sub> EDTA 0.1 mol/L to a blue-end- point
Calculation:	Consumption of mL Na <sub>2</sub> EDTA 0.1 mol/L x 5.869 = Nickel (g/L) = <b>A</b>  [A – (B x 0.247)] x 5.41 = mL/L Nickel sulfamate solution  A = Nickel concentration in g/L  B = Nickel chloride conc. in g/L	

**riag Ni 138 Tenside M**

Reagents:	Glycerine Butyl phosphate solution: Mix 5 mL Tri-n-Butyl phosphate 500 mL Methanol 500 mL water DI	
Procedure:	25 mL 3 drops 0.5 mL	electrolyte are transferred via pipette into a 300 mL Erlenmeyer flask, add glycerine, shake well, to form a foam cover. Add in steps of butyl phosphate solution, shake well after each addition, until the foam cover disappears within 10 seconds
Calculation:	consumption in mL = mL/L <b>riag Ni 138 Tenside M</b>	

**Boric acid**

Reagents:	Sodium hydroxide solution 0.1 mol/L Bromcresol purple (1 % in Ethanol) Mannitol														
Procedure:	<table><tr><td>10 mL</td><td>electrolyte are transferred via pipette into a 100 mL measuring flask and filled up to the mark with deionised water followed by mixing well.</td></tr><tr><td>10 mL</td><td>of this mixture is given into a</td></tr><tr><td>250 mL</td><td>beaker via pipette followed by</td></tr><tr><td>100 mL</td><td>deionised water addition.</td></tr><tr><td>2 – 3 g</td><td>Mannitol is added followed by addition of 10 drops Bromcresol purple.</td></tr><tr><td></td><td>Titration with</td></tr><tr><td>0.1 mol/L</td><td>Sodium hydroxide from yellow to green, to dark green and finally to blue-violet.</td></tr></table>	10 mL	electrolyte are transferred via pipette into a 100 mL measuring flask and filled up to the mark with deionised water followed by mixing well.	10 mL	of this mixture is given into a	250 mL	beaker via pipette followed by	100 mL	deionised water addition.	2 – 3 g	Mannitol is added followed by addition of 10 drops Bromcresol purple.		Titration with	0.1 mol/L	Sodium hydroxide from yellow to green, to dark green and finally to blue-violet.
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	Titration with														
0.1 mol/L	Sodium hydroxide from yellow to green, to dark green and finally to blue-violet.														
Calculation:	Consumption of mL NaOH x 6.18 = Boric acid (g/L)														