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riag Ni 122

Bright Nickel barrel plating process

The **riag Ni 122** barrel Nickel electroplating process deposits nickel layers with the following advantages:

Properties

- excellent brightness
- light coloured white deposits
- excellent metal deposit thickness distribution
- good ductility
- excellent brightness throwing power

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

	Range	Optimum
Nickel sulphate ($NiSO_4$ x 6 H_2O)	150 – 220 g/L	170 g/L
Nickel chloride ($NiCl_2$ x 6 H_2O)	50 – 100 g/L	85 g/L
riag Ni 149 Make up	100 mL/L	100 mL/L
riag Ni 132 Make up	10 – 15 mL/L	12 mL/L
riag Ni 135 Carrier	2 – 5 mL/L	3 mL/L
riag Ni 122 Brightener	0.2 – 0.4 mL/L	0.2 mL/L
riag Ni 138 Tenside M * or **riag Ni 139 Tenside L ***	* 1 – 3 mL/L	*2 mL/L
pH-value	3.8 – 5,2	4.9

* depending on customer-specific process requirements

Operating values

	Range	Optimum
Nickel (Ni^{2+})	50 – 75 g/L	60 g/L
Chloride (Cl^-)	15 – 30 g/L	25 g/L
riag Ni 149 Make up*	80 – 120 mL/L	100 mL/L

*corresponds to the **riag Ni 149 Buffer**

Make up

A separate tank is filled with deionised water to 3/4 of the final volume.
The water is then heated to at least 60 °C after which the chemicals are added and the tank is filled to final volume with deionised water. To remove contaminants 0.5 mL/L hydrogen peroxide is added and the solution is stirred for at least one hour. This is followed by addition of 3 g/L **riag Carb SF** activated carbon and mixing for another 30 minutes. After settling, preferably overnight, the electrolyte is transferred to the working tank by filtration. Finally, the necessary quantities of **riag Ni 149 Make up** and all other required additives are added. The electrolyte is ready for use.
It is possible to obtain ready-to-use electrolytes, e.g. **riag Ni 149 electrolyte** as a rack version.

Alternative make up containing boric acid

	Range	Optimum
Nickel sulphate ($NiSO_4 \times 6\ H_2O$)	180 – 250 g/L	200 g/L
Nicke chloride ($NiCl_2 \times 6\ H_2O$)	50 – 100 g/L	85 g/L
Boric acid (H_3BO_3)	40 – 45 g/L	42 g/L
riag Ni 132 Make up	10 – 15 mL/L	12 mL/L
riag Ni 135 Carrier	3 – 6 mL/L	5 mL/L
riag Ni 122 Brightener	0.2 – 0.4 mL/L	0.2 mL/L
riag Ni 138 Tenside M * oder **riag Ni 139 Tenside L ***	* 1 – 3 mL/L	*2 mL/L
pH-value	3.8 – 4.5	4.2

* depending on customer-specific process requirements

Operating Values

Nickel (Ni^{2+})	50 – 75 g/L	60 g/L
Chloride (Cl^-)	15 – 30 g/L	25 g/L
Boric acid	40 – 50 g/L	42 g/L

Make up

A separate tank is filled with deionised water to 3/4 of the final volume.
The water is then heated to at least 60 °C after which the chemicals are added and the tank is filled to final volume with deionised water. To remove contaminants 0.5 mL/L hydrogen peroxide is added and the solution is stirred for at least one hour. This is followed by addition of 3 – 5 g/L **riag Carb SF** activated carbon and mixing for another 30 minutes. After settling, preferably overnight, the electrolyte is transferred to the working tank by filtration. Finally, the correct undiluted volumes of
riag Ni 132 Make up, **riag Ni 135 Carrier**, **riag Ni 122 Brightener** and **riag Ni 138 Tenside M / riag Ni 139 Tenside L** are added whilst stirring.

Operating Parameters

Temperature:	55 °C (50 – 60 °C)
pH-value:	Boric acid free: 4.9 (3.8 – 5.2) Containing boric acid: 4.2 (3.8 – 4.5) The pH-value of the boric acid-free version should always be measured at operating temperature.
Cathodic current density:	0.1 – 2.0 A/dm^2
Anodic current density:	< 3.0 A/dm^2
Current efficiency:	< 100 %
Deposition rate:	at 1 A/dm^2 ca. 0.2 µm/min
Anodes :	Minimum purity 99.7 % Ni. We recommend polypropylene anode bags
Agitation:	Essential: Barrel rotation, filter pump
Tanks:	Plastic or lined steel
Filtration:	It is important to use continuous filtration and we recommend including activated carbon filtration as well. The filtration rate should be two to three times electrolyte volume per hour.
Heating:	Immersion heaters, but thermostatic control is essential
Cooling:	not required
Fume extraction:	recommended
Maintenance:	Nickel sulphate, nickel chloride and **riag Ni 149 Make up** respectively boric acid should be analysed and corrected regularly. Additions of **riag Ni 122 Brightener**, **riag Ni 133 Carrier** and **riag Ni 135 Carrier** are made via ampere-hour consumption. As an alternative to **riag Ni 133 Carrier**, **riag Ni 132 Make up** can also be added.
Metallic contamination:	Metallic contamination can be removed by frequent selective plating-out at 0.1 – 0.3 A/dm^2. 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.
pH-value adjustment:	Adding **riag Ni 149 Buffer** lowers the pH-value. To lower the pH-value, add **riag Ni 149 Buffer** first, if necessary. Only afterwards, if necessary, use chemically pure sulphuric acid (10 %). In exceptional cases (usually for a limited time), pure hydrochloric acid is used to increase the chloride content of the electrolyte. To increase the pH-value, only nickel carbonate must be used, never ammonia or ammonium compounds.

Additive consumption:	The additives are consumed during electrolytic reactions as well a drag-out losses and the use per 10 kAh can therefore vary.

riag Ni 122 Brightener	0.8 – 1.6 L/10 kAh
riag Ni 135 Carrier	0.5 – 1.2 L/10 kAh
riag Ni 133 Carrier	0.3 – 0.7 L/10 kAh
riag Ni Tenside M / L	0.1 – 0.3 L/10 kAh

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

Operating downtimes/ Storage:	In case of longer production interruptions (e.g.company holidays), we recommend to reduce the pH-value of the electrolyte to below 4.5.

Function of electrolyte components

riag Ni 149 Make up

Contains the buffer system neutralised with nickel carbonate and purified with activated cabon. 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 122 Brightener

For uniform bright coatings it is essential to add the **riag Ni 122 Brightener** in small doses and according to the operating instructions. Smaller more frequent additions are important for optimal brightness and deposit quality. Additions of small but regular amounts increase the deposit quality and reduce the consumption of **riag Ni 122 Brightener**. Regular monitoring and correction reduce the **riag Ni 122 Brightener** consumption as well.

riag Ni 135 Carrier

The **riag Ni 135 Carrier** is responsible for uniform bright coatings over a wide current density range (especially for low current density) and can be determined analytically.

riag Ni 132 Make up / riag Ni 133 Carrier

The **riag Ni 132 Make up / riag Ni 133 Carrier** content can be determined analytically. To get ductile deposits, we recommend not to fall below the operating value. A lack of **riag Ni 132 Make up / riag Ni 133 Carrier** is indicated by shadows at medium to high current densities. In this case, 3 – 8 mL/L **riag Ni 132 Make up** have to be added.

riag Ni Tenside M / L

The consumption of **riag Ni 138 Tenside M / riag Ni 139 Tenside L** may vary due to drag out. It reduces the surface tension and prevents pitting.

A minimum amount of **riag Ni 138 Tenside M / riag Ni 139 Tenside L** is recommended in barrel applications for example to avoid the formation of perforation spots on flat parts that keep "sticking" to the walls.

riag Ni 143 Purifier

Zinc-die casting processing in rack or barrel mode often leads to zinc and copper electrolyte contamination. This can be treated by additions of 0.03 – 0.1 mL/L **riag Ni 143 Purifier**. The volume **riag Ni 143 Purifier** to be added depends on contamination levels but overdosing results in loss of deposit brightness as well as levelling and must be avoided.

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.
For this we recommend our **riag Carb SF** dust-free product which has an active surface area of 1500 m^2 /g. The additional consumption of **riag Ni 122 Brightener** should not exceed 5 %.
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.
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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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Analysis (Analytical Methods)

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

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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 Br2 (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 – consumption in mL) x 13.2 = mL/L **riag Ni 149 Make up / riag Ni 149 Buffer**

Replenishment of **riag Ni 149 Buffer** = (100 – 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:	Nickel chloride hexahydrate (g/L) = consumption of mL $AgNO_3$ x 2.380 Chloride (g/L) = consumption of mL $AgNO_3$ x 0.709	

Nickel

Reagents:	Buffer solution pH 10 Na_2EDTA 0.1 mol/L Murexide (Sodium chloride 1: 100)	
Procedure:	10 mL	electrolyte are transferred via pipette into a
	100 mL	measuring flask and filled- up to the mark with deionised water and mixed well
	10 mL	of this mixture is given into a 250 mL glass beaker by pipette followed by
	15 mL	Buffer solution addition
	100 mL	deionised water and
	1 spatula tip	of Murexide are added The sample colour should then be deep yellow Titrate immediately with Na_2EDTA 0.1 mol/L to a blue- end- point
Calculation:	Nickel (g/L) = consumption of mL Na_2EDTA 0.1 mol/L x 5.869 Nickel sulphate hexahydrate (g/L) = [A – (B x 0.247)] x 4.48 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	electrolyte are transferred via pipette into a 300 mL Erlenmeyer flask, add
	3 drops	glycerine, shake well, to form a foam cover. Add in steps of
	0.5 mL	butyl phosphate solution, shake well after each addition, until the foam cover disappears within 10 seconds
Calculation:	consumption in mL = mL/L **riag Ni 138 Tenside M**	

Boric acid

Reagents:	Sodium hydroxide solution 0.1 mol/L Bromcresol purple (1 % in Ethanol) Mannitol	
Procedure:	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.
Calculation:	Boric acid (g/L) = consumption of mL NaOH x 6.18	