

## DURNI-COAT<sup>®</sup> DNC 471

### Lead-free electroless plating nickel electrolyte for high wear and corrosion resistant applications

**DNC 471** is a process for the electroless plating of mirror finish nickel-phosphorus alloys, particularly those intended for functional applications. The process deposits high-phosphorus layers with a phosphorus-alloy-content of 10 – 14 % (incl. alloying-elements), and displays a high level of operating tolerance. The coatings are totally lead-free.

#### Mechanical characteristics of coating

Hardness:	In state of deposition, 570 HV 0.05 ± 50 The use of a heat-treatment is not recommended
Dilatation:	1.5 – 2.0 %, measured on sections of foil using the dome method
Modulus of elasticity:	170 to 200 kN/mm <sup>2</sup>
Wear resistance:	Taber-abrasion CS 10: approx. 30 – 40 mg/1000 revolutions
Internal stress:	Low levels of compressive stress, low internal tensile stress at end of electrolyte lifetime (< 30 N/mm <sup>2</sup> )

#### Corrosion resistance

The corrosion resistance of these coatings fulfils class 4 of DIN EN ISO 4527 and MIL-C-26074B (very high corrosion resistance):

- according to DIN EN ISO 6988 (Kesternich test SFW 0.2) > 7 cycles
- according to DIN EN ISO 9227 - AASS (acetic acid salt-spray test): > 480 hours

These coatings meet nitric acid testing standards (0.5 min exposition time to HNO<sub>3</sub> 65 %, 20 °C without discolouration) and are highly resistant to the action of chemicals.

## Physical characteristics of the coating

Density (at 10 to 14 % P):	7.9 to 8.2 kg/dm <sup>3</sup>
Melting point:	1140 to 1170 K
Specific el. resistance:	approx. 49 μΩcm
Heat conductance:	0.04 W/(cm x °C)
Linear heat-expansion coefficient:	12 to 13 x 10 <sup>-6</sup> 1/°C
Phosphorus content (incl. alloying elements): (ICP-OES)	10 to 14 %

All technical values are subject to the mentioned test conditions. We therefore expressly point out that due to varying conditions of use and application only the user's own practical test and proof on-site will determine the true level of performance of the coating and/or coating system.

**DNC 471** is suitable for the coating of all metallic materials. The **DNC 471** process can be applied to both rack and barrel items. The deposition rate (assuming that the permitted operating tolerances are observed) is around 8 – 12 μm/h.

**DNC 471** is supplied in four liquid concentrates:

**DNC 471 Make up A**

**DNC 471 Make up B**

**DNC 471 Replenisher 1**

**DNC 471 Replenisher 2**

A make up requires:

**DNC 471 Make up A**

**DNC 471 Make up B**

**DNC Stabiliser 10 (optional)**

For running the electrolyte:

**DNC 471 Replenisher 1 & 2**

and diluted ammonia

**DNC Stabiliser 10** can be added to the electrolyte solution to improve the pH-stabilisation of the electrolyte.

## Tank and equipment

**DNC 471** can be used in existing plants designed for electroless nickel plating, provided heat-resistant plastics (95 °C) or stainless steel tanks with anodic protection are used.

Heating should be carried out using a PTFE or stainless steel steam coil, or an electric immersion heater (casing: stainless steel with anodic protection, glass or PTFE).

An exhaust ventilation system must be provided for the extraction of spray-mist and steam. A cover should be placed over the electrolyte during breaks in production to stop evaporation loss at working or near working temperatures. It will also prevent the entry of dirt or other impurities from the surrounding air.

## Filtration and tank agitation

Continuous filtration of the **DNC 471** electrolyte during the operation helps to ensure optimum deposition. The materials used to make the parts of the filtering system that come into contact with the **DNC 471** electrolyte should be resistant to both heat and chemicals. The filtering system should consist of an immersed centrifugal pump with downstream filter housings (the pump being used to provide tank agitation). A tank circulation rate of at least 10 – 14 tank volumes per hour is recommended for ensuring that continuous operation is accompanied by optimum mixing of the electrolyte and inflowing replenishers. The system should be fitted with 3 µm polypropylene filters (cartridge- or bag type) for continuous operation, or 1 µm for non-continuous operation.

## Operating conditions

### Solution make up:

distilled or deionised water                    55 vol.-% (electrical conductivity < 5 µS/cm)

**DNC 471 Make up A**                            20 vol.-%

**DNC 471 Make up B**                            20 vol.-%

Improved pH-stabilisation can be achieved by adding the following to the electrolyte solution:

**DNC Stabiliser 10**                            10 vol.-%  
(but add no more than 50 vol.-% dist. water)

This adjusts the pH to the target value at approx. 20 °C.

Otherwise, the pH-value is to be adjusted, after the solution make up, at room temperature, using conc. ammonia (chem. pure).

Replenishment:	<b>DNC 471 Replenisher 1</b>	120 g/L nickel
	<b>DNC 471 Replenisher 2</b>	648 g/L hypophosphite
	15 % ammonia	600 mL/L 25 % ammonia

Dosing ratio:                    1 : 1 : 0.44                    **Repl. 1 : Repl. 2 : ammonia**

Operating temp.:                90 – 94 °C

pH value:                        4.4 – 4.8 (measured at 20 °C, electrometric)

Nickel content:                 5.0 ± 0.6 g/L

Reducing agent:                40 ± 5 g/L

Litre charge:                    0.2 – 1.0 dm<sup>2</sup>/L

Deposition rate:                8 – 12 µm/h (depending on pH value & temperature)

Agitation:                        Partial agitation is useful, but not absolutely vital

## Equipment preparation

Before making up a new **DNC 471** electrolyte, treat with concentrated nitric acid all those system components that are likely to come into contact with the **DNC 471** electrolyte solution. After thorough flushing of all these items with normal and then distilled water, check the quality of the water flowing through the filter.

The volume of distilled water (electrical conductivity < 5 µS/cm) required for the electrolyte solution is filled into the receiving vessel. Activate the filter circuit and add the **DNC 471** make up chemicals. Wait for the system to warm up to operating temperature and then take another pH-reading.

## Working instructions

After careful pre-treatment the items to be electroless nickel-plated are simply placed in the **DNC 471** solution and kept immersed until the coating is of the desired thickness. If you do not intend to work any further with the **DNC 471**, it is advisable to let it cool down ( $t < 40$  °C). This is in order to ensure the maximum lifetime life (8 metal turnover) and stability of the solution.

If you intend to treat only aluminium-based materials in the **DNC 471**, the lifetime of the electrolyte depends exclusively on the accumulation of the decomposition product orthophosphite, and on the contamination with zinc. Wrought alloys can be coated up to a maximum of 6 MTO. Pre-treatment using the zincate process is required in order to ensure good adhesion of the deposited electroless nickel layers.

This results in a carry-over of zinc ions into the **DNC 471**, which must not exceed a maximum concentration limit of 50 mg/L in the **DNC 471** electrolyte.

## Base materials

**DNC 471** can be used on all ferrous alloys (steel, stainless steel, etc.), nickel-iron alloys, copper alloys, copper-nickel alloys, aluminium alloys and their derivatives.

riag-Oberflächentechnik will be pleased to supply pre-treatment instructions designed for specific applications.

## Operating temperature

The normal operating temperature is between 90 and 94 °C; the optimum start-up temperature is 90 °C. Lower temperatures reduce the rate of deposition. The **DNC 471** solution should be agitated during the warm-up and cooling phases to prevent the formation of localised hot-spots.

## Electrolyte maintenance

The safeguarding of optimum deposition rates requires that the specified electrolyte parameters described under "Operating conditions" are maintained. Under normal operating conditions, one litre of **DNC 471 Replenisher 1** can cover approx. 67 dm<sup>2</sup> to a thickness of 25 µm. For a volume unit of **DNC 471 Replenisher 1**, add 1.0 part by volume of **DNC 471 Replenisher 2**, plus 0.44 parts by volume of diluted ammonia solution.

Ensure when doing so that the solution does not fluctuate by more than 12 % from the demanded metal-content (see "Operating conditions"). Additions should be made slowly, at regular intervals and in small quantities, or – in the case of large electrolyte volumes – by means of an automatic pH-value and (particularly) nickel-content control system.

**Attention:** If there should be deviations in the hypophosphite content of more than 2 g/L (eg 37 g/L), **DNC 471 Replenisher 2** should be added gradually, this means, the concentration of hypophosphite must never be increased by more than 2 g/L at one time! If larger amounts of hypophosphite have to be added, addition has to be done in steps of 2 g/L with a waiting time of at least 30 minutes between two steps.

We recommend twice a day (morning and evening) analysis of the amounts of nickel and reducing agent present. A metal turnover (MTO) cycle is achieved when 5.0 g/L nickel has been deposited from the solution. An MTO cycle is likewise achieved after consumption of 42 mL/L of **DNC 471 Replenisher 1**.

### **Stabiliser concentration**

It may be necessary to increase the concentration of the stabiliser due to various working methods, be it the parts to be coated (e.g., rack or barrel), equipment (large or small areas) or customer demand (low or high layer thickness).

DNC XXX Replenisher 2 (70)

Example: Concentration stabiliser: 70% of the common version.  
We are happy to advise should a change be necessary.

### **pH value**

The working pH range lies between 4.4 and 4.8. The initial pH value of a new electrolyte solution is 4,4 – 4,5. Monitoring of the electrolyte solution is carried out electrometrically (measured at  $t = 20\text{ °C}$ ).

### **Correcting the pH value**

The pH is lowered by adding approx. 10 % sulphuric acid (60 mL/L concentrated sulphuric acid p.a.).  
pH is increased by adding approx. 15 % ammonia (600 mL concentrated ammonia/L).

All additions must be made slowly and with thorough stirring. Observe the applicable accident-prevention regulations for alkaline and acid substances when handling ammonia and sulphuric acid.

### **Waste water treatment**

**DNC 471** and its rinsing water must be decontaminated and neutralised before disposal in the drain outlet to the sewer system. riag can supply details of these waste water treatment methods on request.

## Possible hazards and safety precautions

These details can be found in the material safety data sheets for **DNC 471 Make up A & B, Replenisher 1 & 2** and **DNC Stabiliser 10**. The relevant material safety data sheets for the handling of ammonia, caustic soda and sodium carbonate solutions should be requested from their respective suppliers.

The **DNC 471 Make up A & B, Replenisher 1 & 2** and **DNC Stabiliser 10**, along with the ammonia solutions, should all be stored between 5 and 25 °C.  
If excessive cooling should cause partial crystallisation of the solution, warm it up to > 20 °C (stirring is recommended).

Prevent skin or eye contact with **DNC 471 Make up A & B, Replenisher 1 & 2, DNC Stabiliser 10** or ammonia solution. In case of skin contact, rinse the affected area with copious quantities of cold running water. Seek medical attention IMMEDIATELY if eye injuries are involved.

## Liability

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## Analysis – Analytic methods

### Nickel

Target value: 5.0 g Ni/L

Required reagents: Na<sub>2</sub>EDTA 0.1 mol/L  
NH<sub>4</sub>OH solution, concentrated (approx. 25 %)  
Murexide powder (1 g murexide and 99 g NaCl)  
Distilled water

Apparatus required: Erlenmeyer flask, 300 mL  
Pipette, 5 mL  
microburette (Bang), 10 mL

Method: Pipette to add 5 mL of electrolyte (20 °C) to a 300 mL Erlenmeyer flask. After adding 10 mL of NH<sub>4</sub>OH and a spatula-tip of murexide powder, top up to about 150 mL with distilled water. Titration now takes place with Na<sub>2</sub>EDTA 0.1 mol/L until there is an abrupt colour-change from yellow to violet.

Calculation: nickel (g/L) = 1.174 x consumed mL Na<sub>2</sub>EDTA 0.1 mol/L

This analysis procedure should be carried out at least twice daily. It is also used for checking the function of the flow-rate photometer. Ensure also that each batch of newly made-up electrolyte is checked in this way.

### Reducing agent

Target value: 40 g/L sodium hypophosphite

Required reagents: Starch solution 1 %  
6 mol/L HCl (600 mL/L 32 % HCl)  
0.05 mol/L KJO<sub>3</sub>/KJ (iodate-iodide)  
0.1 mol/L Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> (sodium thiosulphate)

Apparatus required Pipette, 2 mL  
2 burettes, 50 mL -1/20 division- with fitting-stopper glass taps or Teflon tap cocks  
automatic tipping device, 20 mL  
Erlenmeyer flask with tight-fitting glass stopper (iodine-count flask)

Method: Pipette 2 mL electrolyte (20 °C) in an Erlenmeyer flask, add 25 mL 0.05 mol/L potassium iodide-iodate and acidify with 20 mL 6 mol/L HCl.

Tightly seal Erlenmeyer flask with stopper and allow sample to react for half an hour in total darkness.

Then titrate with 0.1 mol/L sodium thiosulphate solution until a pale yellowish coloration becomes apparent.

Add two drops of 1 % starch solution to mark the transition point exactly. Now continue to titrate until there is a transition from bluish-violet to colourless.

Calculation: reducing agent (g/L) = (mL 0.05 mol/L KJO<sub>3</sub>/KJ – mL 0.1 mol/L Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>) x 2.65