

PALLUNA® 463

Operating Instructions

Edition: 14 May 2009

Ammoniacal palladium-nickel electrolyte

- white, bright and low-pore coatings
- ductile and low-stress coatings
- corrosion- and wear-resistant coatings with high hardness
- stable electrolyte with simple bath maintenance
- for decorative and technical applications
- for rack and barrel operation

Bath Characteristics

PALLUNA® 463 is a weakly alkaline electrolyte for technical and decorative applications for the deposition of palladium-nickel coatings with 80 wt.% Palladium.

The electrolyte is stable, simple to maintain and suit-able for rack and barrel operation.

Bath type:	weakly ammoniacal
Palladium content:	10 g/l Pd
Nickel content:	10 g/l Ni
pH-value:	8.2
Temperature:	room temperature (25°C)
Current density:	1.5 A/dm ²
Deposition speed:	approx. 0.33 µm/min

Coating Characteristics

The white, low-pore coatings are perfectly bright at layer thicknesses of up to 10 µm. The ductile, low-stress coatings have high hardness and a very good wear resistance as well as a good corrosion and tarnish resistance.

Coating:	palladium-nickel
Alloy composition:	approx. 80 wt.% Pd approx. 20 wt.% Ni
Colour:	white
Hardness:	approx. 550 HV 0.025
Density of the coating:	approx. 11 g/cm ³

Form of Supply

Bath makeup:	a) PALLUNA® 463 Initial Concentrate 400 ml for 1 litre of bath Storage stability: min. 3 years
	b) PALLUNA® Palladium Solution 460 100 ml for 1 litre of bath (10 g/l Pd) Storage stability: min. 3 years

- c) PALLUNA® 463 Brightener 1
30 ml for 1 litre of bath
Storage stability: min. 2 years
- d) PALLUNA® 463 Brightener 2
1 ml for 1 litre of bath
Storage stability: min. 2 years
- e) PALLUNA® Wetting Agent 11
10 ml for 1 litre of bath
Storage stability: min. 2 years

Bath replenish f₁) PALLUNA® Palladium Solution 460
ment: 100 g/l Pd
1000 ml for the deposition
of 100 g Palladium
Storage stability: min. 3 years

as an alternative:

- f₂) Umicore Palladium Salt 210
41-43 % Pd
238 g salt for the deposition
of 100 g palladium
Storage stability: unlimited

One unit for the deposition of 100 g palladium (=125 g alloy with 80 % Pd) contains:

- g) PALLUNA® 463 Nickel Complex Solution
100 g/l Ni
250 ml for 100 g palladium
Storage stability: min. 3 years
- h) PALLUNA® 463 Brightener 1 (as item c)
50 ml for 100 g palladium
Storage stability: min. 2 years
- i) PALLUNA® 463 Brightener 2 (as item d)
50 ml for 100 g palladium
Storage stability: min. 2 years

- Corrections:
- j) For the adjustment of the pH-value, ammonia solution 25 % and diluted hydrochloric acid (both chem. pure) should be available.
 - k) PALLUNA® Density Correction Salt 5 Storage stability: unlimited
 - l) PALLUNA® Wetting Agent 11 Storage stability: min. 2 years

PALLUNA® 463

Bath Makeup

Preparation: All materials coming into contact with the bath (bath tanks, filter cartridges, pumps, pipes, hoses, barrels, rack insulation, etc.) must be leached in sulphuric acid prior to first use and subsequently in caustic soda solution. Before filling the bath thoroughly rinse with deionized water to make it alkali-free.

Makeup sequence: To make up 1 litre of bath, stir 400 ml Initial Concentrate into 400 ml deionized water. Afterwards add one after another 100 ml PALLUNA® Palladium Solution 460, 30 ml Brightener 1, 1 ml Brightener 2, and 10 ml PALLUNA® Wetting Agent 11 and mix thoroughly. Then fill up to 1 litre with deionized water. Adjust the pH-value at 25 °C with ammonia or hydrochloric acid to pH 8.2.

Attention:

When making up the bath the personnel must take the necessary preventive measures, such as wearing goggles, protective gloves, protective clothing.

Example: To make up 100 l of bath:
40 l PALLUNA® 463 Initial Concentrate
10 l PALLUNA® Palladium Solution 460
3 l PALLUNA® 463 Brightener 1
0.1 l PALLUNA® 463 Brightener 2
1 l PALLUNA® Wetting Agent 11
45.9 l Deionized Water

Operating Conditions

Palladium content:	10 g/l	(9 - 11 g/l)
Nickel content:	10 g/l	(9 - 11 g/l)
Operating temperature:	25 °C	(20 - 35 °C)
pH-value:	8.2 (25 °C)	(7.7 - 8.7)
Bath density:	1.05 g/cm ³	(1.04 - 1.09 g/cm ³)
Product agitation:	Required. 1-3 m/min, freely variable	
Bath agitation:	Required. Via filter pump. Recirculation approx. 2-3 times the bath volume/hour.	
Filtration:	Continuous filtration through polypropylene filter cartridges, pore width ≤5 µm, is recommended.	
Current density:	1.5 A/dm ²	(0.5 - 2.5 A/dm ²)

Deposition rate: approx. 24 mg/Amin (1.5 A/dm²)
Deposition speed: approx. 0.33 µm/min (1.5 A/dm²)

Exposition time: approx. 3 min. for 1 µm

Loading per litre: up to approx. 1 A/l

Calculation of Coating Thickness and Plating Time

Coating weight in mg = surface in cm² x 1.1 x coating thickness in µm

Plating time in minutes =

$\frac{\text{required coating weight in mg}}{\text{deposition rate in mg/Amin} \times \text{current in amperes}}$

Bath Replenishment

Replenish at the latest if 10 % of the palladium content has been removed. 100 g Pd corresponds to 125 g alloy with 80 % Pd and a current quantity of approx. 5200 Amin (86.7 Ah). To install a Ah-counter is recommended in order to exactly keep the replenishment intervals. For the consumption of 100 g palladium (125 g alloy) the following approximate values can be valid:

1000 ml PALLUNA® Palladium Solution 460
alternatively:
238 g Umicore Palladium Salt 210

as well as
250 ml PALLUNA® 463 Nickel Complex Solution,
50 ml PALLUNA® 463 Brightener 1 and
50 ml PALLUNA® 463 Brightener 2

For continuous replenishment the operating conditions which are different from user to user, such as current density applied, temperature, agitation, filter performance, drag out, etc., must be observed and the approximate values mentioned above must be changed accordingly, if necessary.

Bath Monitoring and Correction

The analytical control is made according to the analytical control methods for PALLUNA® 463 which can be obtained on request.

Metals:

Check the metal contents regularly depending on throughput (e.g. weekly).
Make corrections with:

PALLUNA® Palladium Solution 460
Concentration 100 g/l Pd (10 ml for 1 g Pd)

and

PALLUNA® 463 Nickel Complex Solution
Concentration 100 g/l Ni (10 ml for 1 g Ni).

PALLUNA® 463

pH-value:

During operation of the bath and idle times the pH-value drops. Therefore it must be measured every day and adjusted with ammonia solution 25 % or 1:1 diluted hydrochloric acid (chem. pure).

The pH-value can be increased by blowing in of gaseous ammonia by means of an appropriate dosage unit. We expressly recommend this method!

Bath density:

Depending on the throughput the bath density should be regularly checked (e.g. weekly). To increase the bath density by 0.01 g/cm³ add 40 g/l PALLUNA® Density Correction Salt 5. It is necessary to add in portions and to check the pH-value.

Additives:

Brightener 1 causes the deposition of brilliant layers over the entire current density area. A lack shows in a haze deposition. A slight excessive dose is not critical.

Brightener 2 is necessary for highest brilliance. An excessive dose reduces the deposition speed, a lack causes heavy internal stress, especially when using high current density.

PALLUNA® Wetting Agent 11 is only weakly foaming. In case of wetting problems add 1ml/l to the bath.

If brilliance is missing there might be a lack of brightener or contaminations in the bath. The brighteners can be checked by making a Hull cell test (see below), or by an analysis at the supplier.

Contaminations:

Metallic and organic contaminants interfere and should be avoided by appropriate measures (rinsing steps, etc.).

Active carbon purification:

In case of lack in brilliance caused by organic contaminations an active carbon purification is necessary. For this, stir at least 2 g/l Active Carbon 1 for 2 hours into the bath and filter it subsequently. By the active carbon treatment the Brightener 1 is practically not removed. Brightener 2 is only slightly (<0,5 ml/l) removed. PALLUNA® Wetting Agent 11 is removed for the most part. After an active carbon purification 2 ml/l are usually added. Palladium and nickel are hardly removed.

Alloy composition:

The composition of the deposited alloy is influenced by following parameters:

The palladium content in the alloy increases in case of:

- increasing ratio Pd:Ni in the electrolyte
- increasing bath density
- low temperature
- increasing pH-value
- stronger agitation

Hull cell test:

The Hull cell test is a quick method to check the bath.

The operating conditions are:

- 250 ml Hull cell with anode
- 1 A cell current
- 5 min plating time
- Magnetic stirrer (500 U/min, 25 mm stirring rod)
- Normal working conditions of the bath

The Hull cell should be shiny, with only one little burnt area (approx. 1 cm, area of high current density). For pores in high current density area 1 ml/l PALLUNA® Wetting Agent 11 is added.

Special Process Hints

Layer thickness: If only very thin layers are deposited, it can be worked with reduced metal contents in the electrolyte. The opposite happens for very thick layers (>10 µm), thus an increase of metal contents is necessary.

Pre- and post treatment: Place the parts in the electrolyte free from oxides and grease. Adequate degreasing and acid dip should always be guaranteed in addition to sufficient rinsing steps. We recommend as last step prior to the treatment to dip into a 2 % ammonia solution.

PALLUNA® coatings can be subsequently adhesively plated with PdNi.

An electrolytical degreasing of the palladium-nickel coatings has to be in any case anodic. Cathodic degreasing causes cracks.

Economy rinse: Turbidity in the economy rinse is avoided by adjusting the pH to 10 with ammonia solution.

Equipment

Bath tanks: PPH, PVC, glass, Teflon
Preferably polypropylene natural. The bath must be sealed tightly with a cover when the bath is not operated.

Attention:

Stainless steel, rubber or coloured plastic must not be used as tank material!

Heating: Immersion heaters out of porcelain, quartz glass, or Teflon, with temperature control.

PALLUNA® 463

Filtration and bath agitation: Continuous filtration through polypropylene filter cartridges, pore width $\leq 5 \mu\text{m}$, is recommended. Recirculation via the filter pump approx. 2-3 times the bath volume / hour. The pump used should be a magnetic coupled centrifugal pump out of alkali-resistant plastic. As material for the pumps and hoses, PP, PE or PTFE is recommended.

using our Anodes: We recommend PLATINODE® 167 as anode material. As an alternative the use of graphite anodes is possible, e.g. from the companies Conradt / Nürnberg or SGL Carbon AG / Meitingen.

Important:
Stainless steel or platinized titanium must not be used under any circumstances. The use of such materials causes permanently considerable problems and result in new make up of the bath.

The anode current density should be max. 1 A/dm^2 .

Material: Materials out of copper, brass or stainless steel must not be exposed to the bath vapours and must not come into contact with the bath itself. These metals are attacked by the bath and the ammonia vapours. We recommend titanium, or thickly nickel plated material.

Rectifier: Infinitely variable, with current display and Ah-meter. Residual ripple $< 5 \%$.

Exhaust system: Effective exhaust system required

Note

Our information relating to the storage stability refers to storage in closed original storage containers under the conditions stated on the label.

Precautionary Measures/Safety Hints

For information on safety, please see the corresponding Material Safety Data Sheets! The valid accident prevention regulations and safety information must be observed.

Reference to

Analytical control: available on request

The information and statements contained herein are provided free of charge. They are believed to be accurate at the time of publication, but we make no warranty with respect thereto, including but not limited to any results to be obtained or the infringement of any proprietary rights.

Use or application of such information or statements is at the user's sole discretion, without any liability on our part. Nothing herein shall be construed as a license of or recommendation for use which infringes upon any proprietary rights. All sales are subject to our General Conditions of Sale and Delivery.

Umicore Galvanotechnik GmbH

P.O. Box 12 40 • D-73502 Schwaebisch Gmuend

Delivery address:

Klarenbergstraße 53-79 • D-73525 Schwaebisch Gmuend GERMANY

Telephone +49 (0) 71 71 / 6 07 - 01

Fax +49 (0) 71 71 / 6 07 - 3 16

e-mail: galvano@eu.umicore.com

www.umicore-galvano.com