

PROCIRC 964

ELECTROLESS NICKEL

INTRODUCTION

Procirc 964 Electroless Nickel has been specially developed as an integral part of the Procirc SMD process sequence for electroless nickel and immersion gold plating of printed circuit boards for surface mount applications.

This data sheet incorporates the products:-

Procirc 964 Electroless Nickel MU -		this is simply diluted with water to prepare the working solution.
Procirc 964 RA	-	contains the nickel replenisher, complexants and brightener.
Procirc 964 RB	-	contains sodium hypophosphite, complexants and stabilisers.

BENEFITS

Bright corrosion resistant deposits (6 - 8% phosphorus).

Consistent performance over 0 - 6 metal turnovers.

High bath stability - user friendly.

Built in pH adjustment chemistry.

Flat uniform finish for SMD.

Very receptive to immersion gold deposition.

SOLUTION MAKE-UP

Procirc 964 Electroless Nickel MU	30% v/v.
Deionised water	70% v/v.

OPERATING DATA

	<u>Range</u>	<u>Optimum</u>
Nickel	5.8-6.2 g/l	6 g/l
Sodium hypophosphite	26-32 g/l	30 g/l
Temperature	84-86 deg C	85 degC \pm 1 deg
pH	4.5-4.9	4.7
Loading	0.5-1.5sq.dm/l	1 sq.dm/l
Agitation	Gentle work and solution movement.	
Filtration	Continuous, 5-10 microns.	
Plating Rate	5 microns in 16-21 minutes.	

EQUIPMENT

Tanks	High temperature polypropylene or stainless steel.
Heating	PTFE, stainless steel or immersion heaters with thermostatic control.
Filtration	All plastic, nitric acid resistant.
Extraction	Recommended.

See NOTES for more detailed information on equipment.

INSTALLATION

It is essential that the tanks to be used for Procirc 964 Electroless Nickel are thoroughly cleaned and leached before any chemistry is introduced.

Contact PMD (UK) Limited Technical Department for appropriate procedure.

1. Half fill the clean empty tank with DI water.
2. Add the required volume of Procirc 964 MU.
3. Make up to the operating volume with DI water.
4. Mix thoroughly.
5. Heat solution to operating temperature.

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1. Procirc SP264 Acid Cleaner.
2. Rinse.
3. Rinse.
4. Procirc 921 Microetch.
5. Rinse.
6. Rinse.
7. 10% v/v sulphuric acid.
8. Rinse.
9. Rinse.
10. Procirc 963 Activator.
11. Rinse.
12. Rinse.
13. Procirc 964 Electroless Nickel.
14. Rinse.
15. Rinse.
16. Cirgold 965 Immersion Gold.
17. Drag out.
18. Rinse.
19. Dry.

MAINTENANCE AND CONTROL

The solution should be analysed regularly and maintained at the optimum concentrations detailed under OPERATING DATA.

pH should be maintained at 4.7, and the replenishment chemistry is formulated to provide consistent pH. However, the occasional adjustment with a 50% solution of ammonia may be necessary.

The evaporation rate from the tank is high because of the operating temperature, therefore the solution level should be checked at least every hour and volume maintained by the addition of hot DI water.

Maintenance additions of 964 RA and 964 RB should be based on a calculation of surface area/deposit thickness processed, and supplemented by regular chemical analysis for nickel and sodium hypophosphite. Ideally, the solution should not be allowed to drop below 95% of the optimum nickel concentration.

e.g. for a 100 litre bath

Total nickel content = 600 g

Solution should be replenished after 30 g nickel has been removed,
i.e. 75 sq.dm. plated to 5 microns thickness.

MAINTENANCE AND CONTROL (Cont.)

Replenishment required = 0.3 litres Procirc 964 RA

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0.38 litres Procirc 964 RB

Allowance must also be made for drag out losses which will vary widely depending on the type of board being processed.

Depending on the work throughput this replenishment should be supplemented by twice-daily or daily analysis.

Auto control is preferred for optimum process performance.

Procedure

- (a) Make up bath to volume with water and mix.
- (b) Analyse for nickel and hypophosphite.
- (c) Raise nickel concentration to optimum by addition of Procirc 964 RA and mix.
- (d) Raise the hypophosphite concentration to optimum by addition of Procirc 964RB and mix.
- (e) Record additions in the log.
- (f) Check the pH and adjust.

Replenishment

10 ml/l Procirc 964 RA will increase nickel by 1 g/l.

3 ml/l Procirc 964 RB will increase hypophosphite by 1 g/l.

Additions based upon the analysis should be made frequently to prevent large variations in concentration which will affect the thickness and quality of the deposit. The solution should never be allowed to drop below 90% of the optimum given in the table in OPERATING DATA. If this occurs replenish the nickel deficiency using Procirc 964 MU, i.e. for every 1 g/l nickel low, add 50 ml/l Procirc 964 MU. Re-analyse for nickel and hypophosphite, and adjust with Procirc 964 A and B.

Where regular production is carried out, it is preferable to add the replenishments using drip-feed or metering pumps. Never add the replenishments directly over work in the tank but pour them in at one side while vigorously agitating.

When drip feed replenishment is not being used, at the end of a shift for a day's use the bath should be left at a low concentration and additions only made prior to the commencement of the new work period. Bulk replenishment can be made providing the temperature of the solution is below 50 deg C.

ANALYSIS METHOD

Nickel Concentration

Reagents

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0.1M EDTA (standard volumetric solution).
50% ammonia solution.
Murexide indicator.

Method

- (a) Take a cooled sample of solution and pipette 5.0 ml into a 250 ml conical flask.
- (b) Add 10 ml of DI water.
- (c) Add 7 ml of 50% ammonia solution.
- (d) Add a small quantity (tip of spatula) of Murexide indicator. The solution will turn yellow.
- (e) Titrate to a purple end point with 0.1M EDTA solution.
- (f) Record titre = t ml.

Calculation

$t \times 1.174 = \text{g/l nickel.}$

Hypophosphite Concentration

Reagents

0.1N iodine (standard volumetric solution).
Concentrated hydrochloric acid.
0.1N sodium thiosulphate (standard volumetric solution).
Iodine indicator.

Method

- (a) Take a cooled sample of solution and pipette 5.0 ml into an iodine flask.
- (b) Add with shaking, 12 ml of hydrochloric acid.
- (c) Cool the sample.
- (d) Add 50.0 ml of standardised 0.1N iodine solution and stopper the flask.
- (e) Leave in a cool dark place for 30 minutes.
- (f) Titrate with 0.1N sodium thiosulphate to a clear end point.
- (g) Record titre = t ml.

Calculation

$(50 - t) \times 1.08 = \text{g/l sodium hypophosphite.}$

NOTES

TANKS

Tanks can be constructed of a suitable high temperature plastic or 316 grade stainless steel. Stainless steel tanks must be protected by passivation with nitric acid and should have anodic protection equipment. It is normal to use

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two tanks so that one can be used while the other is being cleaned. (The passivation of stainless steel is achieved automatically during the nitric acid cleaning process). The size of the tank is determined by the surface area to be plated and a good rule of thumb is to allow 1 litre of tank capacity for each 1 dm²/l (100 cm²) of load surface area to be plated.

Tanks should be fitted with lids when not in use to prevent ingress of contaminating materials.

HEATING

Direct heating with special stainless steel sheathed heaters with a 1W/cm or less output is recommended. Alternatively PTFE electric immersion heaters can be used. For large volumes PTFE steam coils or jacketed tanks can be used.

FILTRATION

The pump casing and impellor, filter bowl and filter should be of all plastic construction capable of continuous operation above 86 deg C and of resisting the nitric acid used for tank cleaning. Primary seals should be of Teflon, glass filled, with a water washed bearing.

Procirc 964 should be continuously filtered through a 5 to 10 micron filter in order to achieve the highest deposit quality and reduce maintenance down-time to a minimum. Filter bags are recommended rather than wound cartridges. The pump should have the capacity and the filter bag the surface area, to allow a minimum of 10 tank turnovers per hour.

JIGS AND RACKS

Teflon coated stainless steel jigs or racks are recommended as they are easily stripped in nitric acid without attack. Racks should allow 2-3cm between printed circuit boards.

FUME EXTRACTION

It is recommended that fume extraction should be fitted in order to remove the steam produced by the high temperature operating of the solution. A further advantage is that it will prevent fine droplets of solution, which can be irritating due to the well known dermatitic effect of nickel, from escaping into the atmosphere.

EQUIPMENT MAINTENANCE

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When preparing brand new equipment fill the tank with fresh 30% nitric acid and pump round the system for a minimum of 8 hours to passivate stainless steel and leach out soluble organics from plastic equipment. This should be followed by thorough washing and neutralisation with sodium carbonate as indicated below in order to remove any possibility of nitrate contamination.

Used tanks should be cleaned on a regular basis as follows:-

1. Discharge the solution into a cleaned spare tank or second operating tank and remove and clean filter bags.
2. Rinse and pump the water round the equipment.
3. Fill the tank with 30% nitric acid from the storage tank.
4. Ensure the acid contacts all surfaces including the air pipes, filter pump and housing hoses. Avoid air locks.
5. Leave overnight to strip nickel nodules which may have built up.
6. Pump the acid back into storage.
7. Wash out the tank and all the other equipment thoroughly, including the air pipes and any dead spaces. Do not scrub or scrape at the wall of the tank to avoid activation of the surface.
8. Neutralise by pumping 10% sodium carbonate solution round the system.
9. Rinse by pumping round DI water. All nitrate must be removed. Check rinse water with nitrate papers to ensure good rinsing.
10. Repair any leaks at service joints and valves. Clean bars and hoists, if used, to ensure no contamination can drop in from above.
11. Fit a new filter.
12. Pump the Procirc 964 back into the cleaned tank and make up to volume.
13. Analyse and make any adjustments to concentration and pH.
14. Heat to operating temperature.

"Good Housekeeping" in electroless nickel is essential and a set time each day should be set aside to carry out cleaning and maintenance procedures. All electroless nickel processes are sensitive to contamination such as lead, chromium, copper, iron, tin, aluminium, magnesium, bismuth, zinc, sulphur compounds and nitrates, and all particulate materials such as dust. Great care should be taken to ensure that these materials do not come into contact with the solution.

It is good practice to pump the solution out through the filter each day and

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wash the tank out, and once a week (or more often if required) carry out the full stripping procedure above. Do not go too long without treating the tank as it is more difficult to remove heavy deposits of nickel.

- Treatment in nitric acid
- (a) de-activates surfaces
 - (b) decreases plant down-time
 - (c) prevents spontaneous plate out
 - (d) reduces chemical consumption

When the solution is not in use either in the main tank or in storage, cover with a lid to reduce evaporation and ingress of contaminants such as dust etc.

WATER

Procirc 964 requires DI water or distilled water for make up and maintenance. Distilled water is best but DI water is suitable.

TEMPERATURE

The plating rate of Procirc 964 will vary with the temperature; the higher the temperature the faster the plating rate, however, this is accompanied by a decrease in solution stability. For all practical purposes deposition ceases at temperatures below 50 deg C. For consistent deposits, it is recommended that temperature is kept at 85 deg C \pm 1.0 deg C by means of accurate thermostatic control.

Do not keep the tank at working temperature with no load for long periods of time as this will cause a shortening of the solution life and will increase maintenance time. It is better to lower the temperature to 60 deg C or below and then heat up again. Continuous filtration is still required. Some solution movement is recommended to avoid temperature layering and this can be carried out by several different methods.

AGITATION

Agitation of the solution is recommended to avoid temperature layering so that the whole tank is at an even temperature and also to ensure that chemically depleted solution near the surface of the board being processed is quickly removed and replaced by fresh solution. This agitation can be achieved in three ways or a mixture of all three.

(a) The outflow from the filter pump is the most effective way of moving the solution, especially if it is directed through correctly positioned sparge tubes.

(b) Low pressure air from a blower can be used but should be well filtered to remove any particles. The air pipes should direct the air downwards, not over the components, therefore causing a rolling motion of the solution within the tank. Air agitation will reduce overall chemical efficiency by

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approximately 5%.

(c) The printed circuit boards themselves can be moved in the solution or the solution stirred mechanically.

Boards should be suspended securely so that agitation does not cause them to touch each other or the tank walls.

PLATING RATE

The average plating rate of Procirc 964 at 85 deg C and pH 4.7 is 16 microns per hour over the life of the solution. A new solution will plate faster initially; up to 17 microns per hour, but after this will quickly settle to about 14 microns per hour until after about 6 metal turn-overs the plating rate will drop to 10 microns per hour. At this point the bath should be discarded.

The plating rate will increase as temperature and pH are raised but this will cause stability to decrease. Variations in the nickel and hypophosphite concentration will also cause a variation in plating rate, therefore all these factors should be kept constant for reproducible, high quality work and the most economical operation.

SOLUTION LIFE

The optimum life of the Procirc 964 solution is six complete replenishments of metal content.

6 x 100% metal changes = 6 x 6.0 g/l nickel which is equivalent to the addition of 360 ml/l Procirc 964 RA.

After this volume of replenishment the rate of deposition decreases progressively.

LOADING

The ideal loading for Procirc 964 is 1 dm²/l.

Whilst it is not always possible to achieve this in practice it should be noted that too high (1.8 dm²/l) or too low (0.3 dm²/l) an area may cause a drop in plating rate of 15% to 20%. Between 0.5 and 1.5 dm²/l the plating rate is virtually unaffected.

DISPOSAL

Dispose of in accordance with local authority requirements.

PRODUCT FAMILIES

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The following products or product families are referred to in this data sheet.

<u>PRODUCT</u>	<u>CODE NUMBER</u>
Procirc 264 Acid Cleaner	907004
Procirc 921 Microetch	923001
Procirc 963 Activator	967001
Procirc 964 MU	967002
Procirc 964 RA	967003
Procirc 964 RB	965002
Cirgold 965 Immersion Gold	965001

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