

**N-1018 – Rev 0610**

# TECHNI NICKEL S

The **Techni Nickel S** process is designed to produce a low stress, semi-bright, ductile nickel deposit for electronics applications in rack/barrel and SBE plating equipment. The bath can be used with soluble or insoluble anodes. This process meets the requirements of Fed. Spec. QQ-N-290A, Class 1 and 2 (AMS 2403).

## **FEATURES AND BENEFITS**

1. Soft to hard deposits with good ductility.
2. Ductile deposits with controllable or tensile stress.
3. Ability to alter physical properties of the deposit for various uses.
4. Good corrosion resistance.
5. Good tolerance to impurities.
6. Ease of solution purification. Continuous filtration through activated carbon and continuous electrolytic purification are possible.
7. Stability over long periods of operation.
8. Controllable electrical resistivity of deposit

**DEPOSIT CHARACTERISTICS**

Composition	100% Nickel
Hardness:	175 – 210 VHN
Elongation:	15 – 25%
Tensile Strength:	90,000 psi Max.
Ductility:	10 – 15 180° bends @ 100µ-in (2.5 µm) thickness

**EQUIPMENT/MATERIALS OF CONSTRUCTION**

All equipment specified can be purchased from and installed by Technic Equipment Division, 485 Prospect Street, Pawtucket, RI 02860. (401)728-7081, Fax (401)722-1720.

- Tanks: Temperature stabilized translucent white Polypropylene, Polyethylene, rigid PVC, Koroseal lined steel with Plastisol coated exterior or other materials that will not contaminate the plating solution. Approved, rubber-lined, steel tanks should be used for the Technic Sulfamate Nickel Plating Process. A list of acceptable rubber linings can be obtained from the Technic Laboratory. If there is any doubt as to whether a tank lining has been approved, consult the Technic Laboratory or send a sample of the lining to the laboratory for approval.  
DO NOT USE STAINLESS STEEL.  
New or used tanks must be leached out overnight.
- Anodes: Technic or Inco S nickel rounds or rolled depolarized nickel anodes are recommended for this bath.  
Monel or titanium hooks must not be allowed to touch the solution.  
DO NOT USE STAINLESS STEEL HOOKS.  
Bagging is strongly recommended.
- Anode Bags: Anodes should be contained in un-napped Terylene<sup>®</sup>, Polypropylene, Dynel<sup>®</sup> or equivalent anode bags to prevent the anode film particulates from contaminating the solution.
- Heating coils: Teflon, Quartz or Titanium Immersion Heaters with thermostatic control should be used. Steam heating coils is an option as well. Coils should be made of titanium. Inquire through Technic, Inc.
- Cooling coils: Teflon, Quartz or Titanium Immersion coils with thermostatic control should be used. Coils should be made of titanium. Inquire through Technic, Inc.
- Filtration: Continuous, using leached 1 – 5 micron (1 micron preferable) woven polypropylene filter cartridges with a flow rate of three (3) – five (5) times the tank volume/hour.

**EQUIPMENT PREPARATION****Tanks:**

Prior to makeup, the process tank and ancillary equipment should be thoroughly cleaned and then leached with a sulfuric acid solution.

This procedure is particularly important for new equipment or equipment previously used for other processes.

<b><u>Cleaning Solution</u></b>	<b><u>Metric</u></b>		<b><u>U.S.</u></b>	
	<b><u>Range</u></b>	<b><u>Recommended</u></b>	<b><u>Range</u></b>	<b><u>Recommended</u></b>
Tri-Sodium Phosphate	22.5-67.5 g/l	45 g/l	3-9 oz/gal	6 oz/gal
and/or Potassium Hydroxide	22.5-67.5 g/l	45 g/l	3-9 oz/gal	6 oz/gal
<b><u>Leaching Solution</u></b>				
Sulfuric Acid	100 ml/l	100 ml/l	10% v/v	10% v/v

**Cleaning/Leaching Procedure:**

1. Thoroughly wash down the tank and ancillary equipment with clean water. Recirculate the water through the complete system to remove water soluble materials.
2. Discard the water.
3. Add **cleaning solution** to the tank, heat to 38 – 50° C (100 – 120° F) and recirculate through the complete system for 4 – 8 hours.
4. Discard the cleaning solution.
5. Recirculate water through the complete system.
6. Discard the water.
7. Add the **leaching solution** and recirculate through the complete system. Leave the leaching solution in the tank for a minimum of eight (8) hours.
8. Recirculate the leaching solution through the complete system.
9. Discard the leaching solution.
10. Recirculate water through the complete system.
11. Discard the water.

**Anode Bags & Polypropylene Filter Cartridges:**

1. Wash thoroughly in hot (140° F/60° C) deionized water.
2. Leach with 10% v/v (100 ml/l) sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) solution for eight (8) hours.
3. Rinse thoroughly with deionized water.

**SOLUTION MAKE-UP**

Chemicals Required	1 liter	1 gallon
Nickel Sulfamate Conc. (24 oz/gal) (180 g/l Ni metal) OR Nickel Sulfate Conc. (134 g/l Ni metal)	440 ml	44% v/v
Nickel Bromide Conc. (210-235 g/l Nickel Bromide)	40 ml	4.0% v/v
Boric Acid	30 g	4.0 oz
Wetting Agent: HN-5 (air agitation) or HN-6 (mechanical agitation)	10 ml	1.0% v/v
Nickel Sulfamate Semi-Bright Additive (optional)	7.5 ml	0.75% v/v
Nickel Sulfate Stress Reducer (optional)	25 ml	2.5% v/v
DI water	Balance	

**SOLUTION MAKE-UP/ORDER OF ADDITION**

1. The solution is prepared in either a storage tank or the plating tank
2. Fill the tank 20% full with D.I. Water. Water quality should be at least 1 meg-ohm quality or better. Note: The boric acid should be added last so that it can be dissolved properly.
3. Add **Nickel Sulfamate Concentrate (24 oz/gal) OR Nickel Sulfate Concentrate** and mix thoroughly.
4. Add **Nickel Bromide Concentrate** and mix thoroughly.
5. Heat to 120-130°F (50-55°C).
6. Electrolyze the bath at low current density, about 5 ASF (0.5 ASD), using corrugated dummy cathode plates. Continue electrolysis for approximately 12 hours or until the deposit in the recesses of the corrugated cathodes is light in color.
7. Add **Boric Acid** and mix thoroughly.
8. Add **HN-5** or **HN-6** Wetting Agent and mix thoroughly.
9. Add **Nickel Sulfamate Semi-bright Additive** (optional).
10. Add **Nickel Sulfate Stress Reducer** (optional).
11. Bring the tank to final volume with DI water and mix well for a minimum of 1 hour.
12. Replace anodes/anode baskets in plating cells and filters in filter chambers.

**OPERATING PARAMETERS****Metric**

Parameters	Range	Optimum
Nickel metal as Nickel Sulfamate Conc. OR Nickel Sulfate Conc.	60 – 90 g/l	79 g/l
Nickel Bromide Conc.	30 – 50 ml/l	40 ml/l
Boric Acid	22.5 – 37.5 g/l	30 g/l
HN-5/HN-6	5 – 15 ml/l	10 ml/l
Nickel Sulfamate Semi-Bright Additive (optional)	5 – 10 ml/l	7.5 ml
Nickel Sulfate Stress Reducer (optional)	20 – 30 ml/l	25 ml/l
pH	3 – 4.5	3.8
Temperature	49 – 60°C	54°C
Cathode Current Density:	0.3 – 5 ASD	Dependent upon application
Anode Current Density:	< 15 ASD	< 15 ASD
Anode to Cathode Ratio:	2:1 minimum	
Agitation:	Vigorous solution movement	
Efficiency:	≥ 95%	
Deposition rate:	0.3 µm/min @ 1.5 ASD	

US

Parameters	Range	Optimum
Nickel metal as Nickel Sulfamate Conc. OR Nickel Sulfate Conc.	8 – 12 oz/gal	10 .7 oz/gal
Nickel Bromide Conc.	3.0 – 5.0% v/v	4.0% v/v
Boric Acid	3.0 – 5.0 oz/gal	4.0 oz/gal
HN-5/HN-6	0.5 – 1.5% v/v	1.0% v/v
Nickel Sulfamate Semi-Bright Additive (optional)	0.5 – 1.0% v/v	0.75% v/v
Nickel Sulfate Stress Reducer (optional)	2.0 – 3.0% v/v	2.5% v/v
pH	3 – 4.5	3.8
Temperature	120 – 140°F	130°F
Cathode Current Density:	3 – 50 ASF	Dependent upon application
Anode Current Density:	< 150 ASF	< 150 ASF
Anode to Cathode Ratio:	2:1 minimum	
Agitation:	Vigorous solution movement	
Efficiency:	≥ 95%	
Deposition rate:	12 μ-in/min @ 15 ASF	

**SOLUTION MAINTENANCE & CONTROL**

**Nickel Metal Concentration** - Nickel metal is maintained by the addition of Nickel Sulfamate Concentrate (24 oz/gallon; 180 g/l) or Nickel Sulfate Concentrate (134 g/l Ni metal). An addition of 4.2 gallons of Nickel Sulfamate Concentrate (24 oz/gal) per 100 gallons of solution (42 ml/l) or an addition of 5.6 gallons of Nickel Sulfate Concentrate per 100 gallons of solution (56 ml/l) will increase the nickel metal concentration 1 oz per gallon (7.5 g/l). Analytical procedures are located in the next section.

**Nickel Bromide** - Nickel Bromide is maintained by the addition of Nickel Bromide Conc. Nickel Bromide promotes favorable anode corrosion as it does in the case of other nickel solutions and in so doing not only maintains the nickel content and pH at a satisfactory level, but also minimizes oxidizing effects at the anodes. Low concentrations will result in passivation of anodes. To assure proper corrosion of anodes make additions on a regular basis.

**Boric Acid** - This constituent exerts its usual buffering action at the cathode film. When making additions slurry the Boric Acid using hot Nickel Sulfamate solution and slowly add to the tank. Make additions after all plating for the day is done. Salts may also be placed in an anode bag and hung in the back of the tank to dissolve as needed.

**pH** - Lower the pH with Sulfamic Acid. An addition of 30 grams will lower the pH 0.1 units per 100 gallons (380 liters) of solution. Nickel Carbonate is used to raise the pH.

**Nickel Sulfamate Semi-Bright Additive**, when added, will increase the tensile strength of the deposit where harder deposits are required. It will increase brightness of deposit and is added based on appearance. If it is required to analyze for Nickel Sulfamate Stress Reducer in the bath (See Appendix for Analytical Procedure), Nickel Sulfamate Semi-Bright Additive should **not** be used, as it will interfere with the Analytical Procedure.

**Nickel Sulfamate Stress Reducer** should be added at a rate of one liter per 8,000 amp hours. This stress reducer, when added, produces a profound effect upon the properties of the deposited nickel. The stress reducer may be added when the tensile stress of the deposit is high or when a hard deposit is required. It will also increase electrical resistivity and give brightness to the deposit. It may also be used when compressive stress is desired.

**HN-5** is a surfactant which should be used when air agitation systems are employed. It is the sole wetting agent used to prevent pitting and is replenished based on need.

**HN-6** is a surfactant which should be used when mechanical agitation systems are employed. It is the sole wetting agent used to prevent pitting and is replenished based on need.

## **PURIFICATION**

### **1. Carbon Treatment:**

Purification of a bath that has been in operation for quite some time and which has become contaminated with either organic or metallic impurities is accomplished by carbon treatment and/or electrolytic purification. Many types of organic contamination may be encountered in nickel plating solutions. When the contamination is not severe, the solution can be continuously filtered through small amounts of carbon. This filtration will remove a small proportion (1 to 5%) of semi-bright additive and stress reducer.

Severe organic contamination may be removed by continuous filtration or batch purification. When using continuous filtration through large amounts of carbon, the concentration of the additives should be determined and adjusted to operating limit. This is true also for batch purification.

Batch treatments are accomplished by pumping the solution into a suitable treatment tank supplied with heating coils and mechanical mixing. After pumping the plating solution to the storage tank, heat the bath to 140° F (60° C). Do not adjust the pH in the bath. However, in case of certain types of organic contamination the pH should be lowered to 1.0 to 1.5 electrometric. If iron or chromium are to be removed, you will need to raise the pH to 5.0 to 5.3 electrometric. Add a slurry of carbon, usually 2 to 5 lb/100 gal (2.5 to 6 g/l), and agitate for approximately six hours while maintaining temperature. After this treatment, the solution is filtered back into the plating tank. Adjustments of pH and concentration of additives and HN-5/HN-6 should then be made.

### **2. Electrolytic Purification:**

The most common metallic impurities found in nickel solutions are lead, chromium, copper, zinc, and iron. Except for chromium, they can all be removed efficiently by co-deposition at low current densities. This is accomplished by using corrugated dummy cathodes and an over all current density of 5 ASF (0.5 ASD). Chromium may be best removed by chemical reduction followed by precipitation at high pH.

If electrolytic purification is to be carried out in the plating tank itself, insert dummy cathodes during off production hours and plate at 5 ASF (0.5 ASD). Agitation will increase the rate of removal of the above metals. The operating pH and temperature should be maintained during the low current density electrolysis. The time of electrolysis is dependent upon the concentration of the metallic impurities. Normally, when the deposit in the recesses of dummies is white or light gray, elimination of the contaminants is indicated.

Certain installations will require a separate purification tank. Normally 0.05 ampere-hours of electrolysis per gallon (0.0132 amp-hrs/l) of solution per hour of operation will control most cases of metallic contamination. For 1000 gallons (3785 liters) of nickel solution 50 ampere should be employed in the purification tank. At 5 ASF (0.5 ASD), 10 square feet (0.93 sq.m) of corrugated cathodes should be used. The tank size should be designed to take care of the necessary cathode area. The voltage required should be about 1 or 2 volts. It is also advisable to flow the solution from the plating tank to the purification tank, then to the filter and back into the plating tank.

The wise choice of well-engineered equipment will simplify these purification treatments and produce consistent results.

### **HANDLING PRECAUTIONS**

**BEFORE HANDLING ANY CHEMICAL PRODUCTS, IT IS IMPORTANT TO READ THE APPROPRIATE MATERIAL SAFETY DATA SHEET.**

When handling chemicals, always wear the prescribed protective clothing as detailed in the appropriate Material Safety Data Sheet.

In case of skin contact, flush affected area with copious amounts of cold, clean water for at least 10 minutes. In case of serious exposure, particularly for eyes, obtain medical attention.

### **STORAGE**

Store all chemicals in upright, original containers away from direct sunlight and in a dry area at 50 – 90° F (10 – 32° C). Keep container closed when not in use.

### **WASTE TREATMENT**

It is the user's responsibility to verify that treatment procedures comply with federal, state, and local regulations. Working solutions should be diluted, neutralized and disposed of in accordance with local and federal regulations. Consult your local agencies for recommendations for your area. Consult your Technic Inc representative for further information.



**ORDERING INFORMATION**

Contact your local Technic company to order products and/or Material Safety Data Sheets:

Product Code	Product Name	Solid	Liquid	Application
H-1009	HN-5		X	For Makeup and Replenishment
H-1129	HN-6		X	For Makeup and Replenishment
N-1013	Nickel Bromide Concentrate		X	For Makeup and Replenishment
450972	Boric Acid	X		For Makeup and Replenishment
N-1018	Nickel Sulfamate Conc. (24 oz/gallon)		X	For Makeup and Replenishment
130501SB	Nickel Sulfamate Semi Bright Additive		X	For Makeup and Replenishment
132469	Nickel Sulfate Stress Reducer		X	For Makeup and Replenishment
130459	Nickel Sulfate Concentrate		X	For Makeup and Replenishment

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Determination of Nickel Metal

**Equipment:** 1 2 ml pipette  
1 250 ml Erlenmeyer flask  
1 50 ml burette

**Reagents:** 0.1M EDTA  
Concentrated Ammonium Hydroxide  
Murexide Tablets

**Procedure:**

1. Pipette a 2 ml sample into a 250 ml Erlenmeyer flask.
2. Add approximately 50 ml of DI water.
3. Add ammonia drop-wise to blue color.
4. Add 2 Murexide tablets to brown color. (If the solution turns green add more ammonia hydroxide to brown color.)
5. Titrate with 0.1M EDTA to a clear purple end-point.
6. If end-point fades, repeat test with a third Murexide tablet.

**Calculation:**

$$\text{mls } 0.1\text{M EDTA} \times 0.39 = \text{av. oz./gal Nickel Metal}$$

$$\text{mls } 0.1\text{M EDTA} \times 2.93 = \text{g/l Nickel Metal}$$

Determination of Boric Acid

**Equipment:** 1 2 ml pipette  
1 150 ml beaker  
1 50 ml burette

**Reagents:** 0.1N Sodium Hydroxide (NaOH)  
Bromocresol Purple Indicator (BCP)  
0.1N Hydrochloric Acid (HCl)  
Mannitol powder

**Procedure:**

1. Pipette a 2 ml sample into a 150 ml beaker.
2. Add 10-20 drops BCP.
3. Add 0.1N NaOH to purple.
4. Add 0.1N HCl to yellow.
5. Add 0.1N NaOH dropwise to red.
6. Add Mannitol until thick yellow paste is formed.
7. Titrate with 0.10 NaOH to purple.

**Calculation:**

$$\text{ml } 0.1\text{N NaOH} \times 0.413 = \text{oz/gal Boric Acid}$$

$$\text{ml } 0.1\text{N NaOH} \times 3.09 = \text{g/l Boric Acid}$$

Determination of Nickel Bromide

**Equipment:** 1 10 ml pipette  
 1 250 ml Erlenmeyer flask  
 1 50 ml burette

**Reagents:** 0.1N Silver Nitrate (AgNO<sub>3</sub>)  
 40% Ferric Ammonium Sulfate  
 0.1N Thiocyanate (SCN)  
 Distilled Water  
 Concentrated Nitric Acid

**Procedure:**

- 1) Pipette a 10 ml sample into a 250 ml Erlenmeyer flask.
- 2) Add approximately 100 mls of distilled water.
- 3) Add about 5 mls of concentrated Nitric Acid and exactly 25 mls of 0.1N Silver Nitrate.
- 4) Add 2 mls of 40% Ferric Ammonium Sulfate Indicator
- 5) Titrate with 0.1 SCN to the first permanent reddish brown endpoint.

**Calculation:**

$$(25.0 \text{ mls AgNO}_3 \times N \text{ Ag NO}_3) - (\text{ml SCN} \times N \text{ SCN}) \times 1.46 = \text{av. oz/gal Nickel Bromide}$$

$$(25.0 \text{ mls AgNO}_3 \times N \text{ Ag NO}_3) - (\text{ml SCN} \times N \text{ SCN}) \times 10.95 = \text{g/l Nickel Bromide}$$

Example: If 0.1N AgNO<sub>3</sub> and 0.1N SCN

$$\text{Then: } (2.5 - x \text{ mls SCN} \times 0.1) \times 1.46 = \text{av. oz/gal Nickel Bromide}$$

$$(2.5 - x \text{ mls SCN} \times 0.1) \times 10.95 = \text{g/l Nickel Bromide}$$

*To determine the addition of Nickel Bromide Concentrate needed  
 av. oz/gal Nickel Bromide x 4.59 = fl oz/gal Nickel Bromide Concentrate*

OR

$$\text{g/l Nickel Bromide} \times 4.59 = \text{ml/l Nickel Bromide Concentrate}$$

**Analytical Procedure for Nickel Sulfate Stress Reducer**

<b>Equipment:</b>	1	5 ml pipette
	1	25 ml pipette
	1	1000 ml volumetric flask
	2	100 ml volumetric flask
	1	UV/VIS Spectrophotometer
	1	Quartz cuvette

**Preparation of Standard:**

1. Pipette 25 ml of Nickel Sulfate Stress Reducer into a 1000 ml volumetric flask.
2. Dilute to 1 liter with deionized or distilled water.
3. Pipette 5 ml of the above solution into a 100 ml volumetric flask and dilute to volume with deionized or distilled water.

**Procedure:**

1. Add 5 ml of the plating solution sample into a 100 ml volumetric flask.
2. Dilute to volume with deionized or distilled water.
3. Set the UV/VIS Spectrophotometer to 279 nm.
4. Using a quartz cuvette with deionized or distilled water, set absorbance to zero (0).
5. Read the standard as prepared above and record the absorbance as A.
6. Read the diluted sample and record the absorbance as B.

**Calculations:**

$$\text{ml/l Nickel Sulfate Stress Reducer} = \frac{B}{A} \times 25$$

$$\% \text{ v/v Nickel Sulfate Stress Reducer} = \frac{B}{A} \times 2.5$$