

Nickel Sulfamate

Recommended Purification Procedures

The physical properties of electrolytic nickel will be affected by the accumulation of inorganic metallic and organic contaminants. Therefore, when installing new baths and to maintain the desirable properties of production solutions the following purification procedures are recommended. Depending on the concentrations and variety of contaminants, one or a combination of the following procedures may be required for effective treatment. The most common procedures are high pH, electrolytic dummy purification and carbon batch treatment or carbon polishing.

Contaminant	Maximum Conc. (ppm)	Purification Treatment
Aluminum	60	High pH
Chromium	10	High pH
Copper	30	High pH and Electrolysis
Iron	50	High pH and Electrolysis
Lead	2	Electrolysis
Zinc	20	High pH and Electrolysis
Calcium	pH dependant	Will precipitate at saturation
Organic	Variable	Batch Carbon or Carbon Polish

Note: Limits may be different when multiple contaminants are present simultaneously.

High pH Treatment (Precipitation)

The high pH treatment process is based on the precipitation of metallic impurities, primarily iron and chromium. It is best to carry out this procedure in a dedicated off line treatment tank.

Begin the procedure by increasing the temperature to 150 °F.

Sufficient nickel carbonate is added to the tank to raise the pH to 5.2 – 5.8. Depending on the initial pH as much as 5 pounds (dry weight) of nickel carbonate per 100 gallons, added as a slurry, will be required to increase the pH to the optimum treatment range.

Add approximately 0.5 to 1.0 milliliters per liter of 30% hydrogen peroxide to the tank.

Agitate the solution at temperature for at least 2 hours and allow sufficient time to settle.

Readjust pH to the specified operating range and filter back to the production tank.

Electrolyze the solution with the low current dummy cycle to ensure that the hydrogen peroxide has been destroyed and verify the treatment effectiveness and quality of deposit with the Hull cell (2 amps, 5 minutes) prior to resuming production.

Electrolytic Purification (Dummy Plating)

The most common metallic impurities found in nickel solutions are lead, chromium, copper, zinc, and iron. Except chromium, they can be removed efficiently by preferential co-deposition at low current densities. This is commonly known as “dummying the bath” either as a start up procedure on new chemistry installation or for purification and maintenance of production solutions. This is accomplished by a lengthy low current density electrolysis using dedicated corrugated stainless steel dummy cathodes.

The efficiency of the dummy cycle can be improved by reducing the solution pH to 3.0 prior to the start of electrolysis. The cathode area should be large and this is easily accomplished by creasing and folding the cathode with recesses of one to two inches peak to valley. Continuous mild agitation is recommended during the dummy plating cycle. Due to the differing potentials of the various metallic contaminants, multiple current ranges are recommended. Low current densities of 1 to 2 asf will preferentially remove lead and copper. Higher applied current densities of 4 to 6 asf will be more effective for the removal of zinc and iron contaminants.

Dummy cycle:

- 2 Hours @ 2ASF
- 2 Hours @ 5ASF
- 1 Hour @ 15-20ASF *

* Note: Due to the larger than normal cathode surface area, make sure the corrugated dummies strap hangers do not over heat during the high current final period. If the straps tend to heat up, reduce the applied current of the final portion of the dummy cycle to an acceptable level.

Normally, when the appearance of the nickel deposit in the recesses (LCD) of the corrugated dummies is matte light gray, elimination of the metallic contaminants is complete and the solution has been sufficiently electrolyzed. Verify the treatment effectiveness with a 2-amp, 5 minute Hull cell test prior to resuming production. The correct appearance of the panel should be a uniform matte light gray deposit across the entire panel from edge to edge.

Peroxide/Carbon Treatment (Batch)

Due to the manufacturing process used to create activated carbon extremely small particles, “fines” are normally to be expected. These fines are difficult to remove from treated solutions except with rigorous sub-micron filtration. Therefore, batch treatment in the process tank should be avoided unless sufficient settling time and efficient filtration are available. Final filtration prior to resuming production should be four solution turnovers per hour through “unrated” one-micron filter cartridges. If the level of organic contamination is reasonably low, carbon polishing with cartridges may be adequate (see carbon polishing section).

Maximum effectiveness of the batch process depends on achieving sufficient carbon-solution contact time for the adsorption of the organic contaminants. The carbon filtration system should be capable of at least four solution turnovers per hour based on the volume of the process tank. Activated carbon is made from a variety of organic sources each with variable residual trace elements that may be undesirable. Therefore, follow the recommendation for carbon selection for maximum effectiveness.

1. Remove the existing filter cartridges from filter chamber (a conversion kit may have been supplied or is available from the equipment manufacturer).
2. Remove anode bars or anode baskets. (Store in 2% Sulfuric solution)
3. Check that anode bags drain freely and replace any that are partially plugged.
4. Heat nickel solution to 120°F minimum.
5. Add 1 liter per 100 gallons of solution of 30% Hydrogen Peroxide.
6. Turn off air, if used and circulate solution with filter pump only for 1 hour after the addition of the peroxide.
7. Install granular carbon in filter bag (Specify Calgon CPG-LF, Norit SX 4, or Norit RO 0.8 carbon type) and circulate for 2 hours. Replace carbon until bath has been exposed to 4 lbs. per 100 gals of solution.
8. Load clean stainless corrugated dummies into tank for electrolytic purification.
Note: Plating will not initiate until peroxide has been destroyed.
9. Remove the carbon pack from the filtration unit, replace with unrated one- micron cartridges of the appropriate length, and circulate 2 hours prior to resuming production.
10. Add grain refiner and wetter per technical data sheet and verify treatment effectiveness with a 2 amp, 5 minute Hull cell.

Carbon Polish Procedure

1. Carbon polishing of the nickel solution is a beneficial preventative maintenance procedure and typically does not remove large quantities of the grain refiner and wetter.
2. Once a week, carbon polish the solution for 2 hours using a 10" carbon tube. Frequency and/or size of carbon tube may be increased based on the size of the tank and loading.