

Thermo. Titr. Application Note No. H-052

Title: Determination of nickel in electroless plating solutions

Scope: Thermometric titration of nickel in electroless plating solution with disodium dimethylglyoximate.

Principle: Titration of Ni(II) with standard sodium dimethylglyoximate (DMG) solution in buffered ammonia solution to an exothermic endpoint. Two moles of DMG react with one mole of Ni(II). Acidic Ni solutions should be complexed with citrate prior to basification.

Reagents: Titrant: 0.5 mol/L disodium dimethylglyoximate. Dissolve 153.6 g disodium dimethylglyoximate (99% pure, FW = 304.21) in deionized water and make to 1000mL in a volumetric flask.
NH₃/NH₄Cl buffer: Dissolve 17.5 g A.R. NH₄Cl in 172 mL A.R. conc. NH₃ soln. Make to 250 mL with deionized water.

Method: Basic Experimental Parameters:

Titration delivery rate (mL/min.)	2
No. of exothermic endpoints	1
Data smoothing factor	55
Stirrer speed	10-12*

Procedure: Pipette aliquots of Ni solution ranging from approximately 0.3 – 1 mmole Ni(II) into titration vessel. Add 2mL NH₃/NH₄Cl buffer solution, and sufficient DI water to give a starting volume ranging between 35mL for 0.3 mmole Ni, and 50mL for 1 mmole Ni. For low amounts of Ni, employ a stirring speed of 10, and for higher amounts a speed of 12 (the suspension becomes quite viscous with higher amounts of Ni, hence the higher stirring speeds* and larger than usual volumes of water). Titrate to a single exothermic endpoint with 0.5 mol/L disodium dimethylglyoximate.

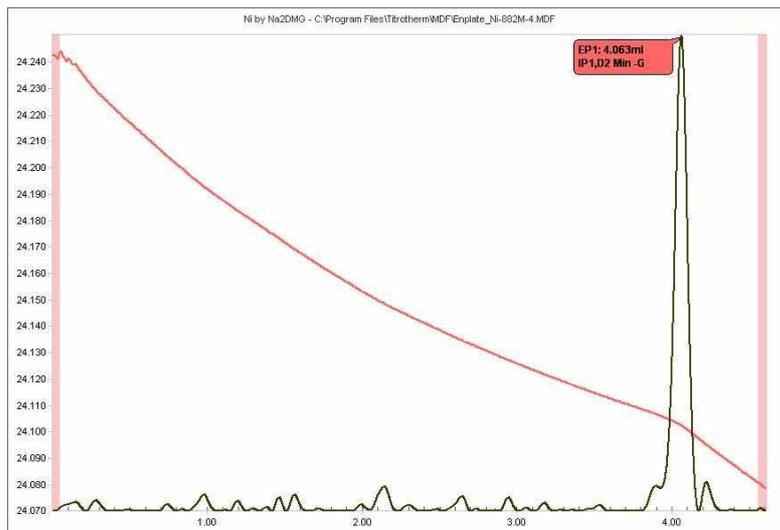
Equipment stained by the Ni dimethylglyoximate precipitate may be cleaned by soaking in a solution of a strong mineral acid; eg, 1 mol/L HCl.

The exposed thermistor may be cleaned periodically with a soft toothbrush, although it is also rapidly cleaned by soaking in 1 mol/L HCl .

Results: Titration of aliquots of electroless plating solution containing nickel and sodium hypophosphite

Ni = 29.51 ± 0.03 g/L (%RSD = 0.11, n=6)

Thermometric Titration Plot:



Legend:

Red = solution temperature curve

Black = second derivative curve