

## Thermo. Titr. Application Note No. H-090

**Title:** Nickel in electroless nickel solutions by thermometric EDTA titration

**Scope:** Automated thermometric titration of the nickel content of electroless nickel plating solutions. The determination is suitable for fully automated titration employing a 814 Sample Processor.

**Principle:** An aliquot of electroless nickel plating solution is reacted with an excess of standard tetrasodium EDTA solution. The excess EDTA is back-titrated to a single thermometric endpoint with standard copper sulfate solution. Nickel reacts too slowly with EDTA to permit a direct titration.

**Reagents:**

*Titration 1:* 1mol/L Na<sub>4</sub>EDTA

*Titration 2:* 1mol/L CuSO<sub>4</sub>

*NH<sub>3</sub>/NH<sub>4</sub>Cl buffer.* Dissolve 87.5g NH<sub>4</sub>Cl in 568mL 28% w/v NH<sub>3</sub> solution and dilute to 1000mL with DI water

**Method:**

*Basic Experimental Parameters:*

Titration delivery rate (mL/min.)	4
No. of exothermic endpoints	1
Data smoothing factor	62
Stirring speed (802 stirrer)	8
Delay before start (secs.)	5

*Sample Preparation*

An aliquot containing approximately 2.5mmol Ni is diluted to 30mL with DI water in a titration vessel

*Titration Program*

A titration program is set up to pre-dose in sequence 5mL of standard Na<sub>4</sub>EDTA solution followed by 5mL buffer solution

*CuSO<sub>4</sub> standardization.* The CuSO<sub>4</sub> solution is standardized against the standard Na<sub>4</sub>EDTA solution by titrating aliquots of 1, 2, 3, 4 and 5mL Na<sub>4</sub>EDTA solution. From a plot of mmol Na<sub>4</sub>EDTA (x-axis) against mL CuSO<sub>4</sub> (y-axis), the gradient of the linear regression is computed. The molarity of the CuSO<sub>4</sub> solution is equal to the reciprocal of the gradient.

**Examples:** *Electroless nickel plating solutions containing nickel and boric acid, submitted by a customer*

Solution # 1: Ni = 5.40±0.024% w/v (n=8)

Solution #2: Ni = 2.76±0.025% w/v (n=8)

**Calculations:**

$$\%w / v \text{ Ni} = \frac{((V_1 - (\frac{V_2 \times M_2}{M_1}) \times M_1 \times AM \text{ Ni} \times 100))}{(V_s \times 1000)}$$

**Legend:**

V <sub>1</sub>	Volume of Na <sub>4</sub> EDTA solution pre-dosed, mL
V <sub>2</sub>	Volume of CuSO <sub>4</sub> solution titrated, mL
V <sub>s</sub>	Volume of sample solution, mL
M <sub>1</sub>	Molarity of Na <sub>4</sub> EDTA solution, mol/L
M <sub>2</sub>	Molarity of CuSO <sub>4</sub> solution, mol/L
AM	Atomic mass of Ni, 58.6934

**Thermometric Titration Plot:**

**Legend:**  
 Red = solution temperature curve  
 Black = second derivative curve (for endpoints)

