

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:

Titrant 1: 1mol/L Na₄EDTA

Titrant 2: 1mol/L CuSO₄

NH₃/NH₄Cl buffer. Dissolve 87.5g NH₄Cl in 568mL 28% w/v NH₃ solution and dilute to 1000mL with DI water

Method:

Basic Experimental Parameters:

Titrant 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 Na₄EDTA solution followed by 5mL buffer solution

CuSO₄ standardization. The CuSO₄ solution is standardized against the standard Na₄EDTA solution by titrating aliquots of 1, 2, 3, 4 and 5mL Na₄EDTA solution. From a plot of mmol Na₄EDTA (x-axis) against mL CuSO₄ (y-axis), the gradient of the linear regression is computed. The molarity of the CuSO₄ 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 ₁	Volume of Na ₄ EDTA solution pre-dosed, mL
V ₂	Volume of CuSO ₄ solution titrated, mL
V _s	Volume of sample solution, mL
M ₁	Molarity of Na ₄ EDTA solution, mol/L
M ₂	Molarity of CuSO ₄ solution, mol/L
AM	Atomic mass of Ni, 58.6934

Thermometric Titration Plot:

Legend:

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

