Thermo. Titr. Application Note No. H-047

Title:	**Determination of Nickel by EDTA Back-Titration**

***Scope*:**	Determination of nickel in refinery and plating solutions. When other metals capable of being complexed by EDTA are present, these will interfere and enhance the result for nickel.

***Principles*:**	Tetrasodium EDTA (Na_4EDTA) is the preferred reagent for the thermometric complexometric titration of metals, due to its much higher solubility than the normally used dibasic salt Na_2H_2EDTA. Nickel reacts too slowly with EDTA to be considered for a direct thermometric titration. In this determination, an excess of EDTA is added, sufficient time allowed to complete the reaction, and the excess EDTA back-titrated with standard Cu solution. The thermometric titration of nickel (and associated metals) with EDTA is carried out in an ammonia/ammonium chloride buffer (~pH 10) environment. The endpoint is marked by a slight upswing in temperature, caused by the formation of the Cu ammine complex, whose reaction is slightly more exothermic than that of the Cu-EDTA complex

Reagents:	Titrant 1. 1 mol/L tetrasodium EDTA (standardized) – Dosino #1 Titrant 2. 1 mol/L Cu^{2+} solution, prepared from A.R. $CuSO_4.5H_2O$ and standardized against standard Na_4EDTA solution – Dosino #2 Buffer: NH_3/NH_4Cl solution, pH 10. Dissolve 70g NH_4Cl in 688mL conc. NH_3 soln. and make to 1000mL with D.I. water – Dosino #3

Method:	Basic Experimental Parameters:	
	Titrant delivery rate (mL/min.)	2
	No. of exothermic endpoints	1
	Data smoothing factor	60
	Stirring speed (802 stirrer)	6
	Delay before start (secs.)	60
	Buffer pre-dose (mL)	5
	Na_4EDTA pre-dose (mL)	5
	For this exercise, a 0.15 mol/L Ni^{2+} solution was prepared from A.R. $NiSO_4.6H_20$. A 20mL aliquot is pipetted into a titration vessel, and the titration program commenced. Titrate to a change in gradient of the temperature curve.	

Calculation:	
	$\%Ni = \frac{(((EDTA\ mL - (Cu^{2+}mL \times MCu^{2+} / MEDTA)) \times MEDTA \times FW\,Ni \times 100)}{(Sample\,mass, g \times 1000)}$

Results (example):	
Sample: A.R. $NiSO_4.6H_20$	%Ni = 99.99±0.14, n=11

Thermometric Titration Plot:

Legend:

Red = solution temperature curve

Black =second derivative curve