

## Thermo. Titr. Application Note No. H-046

**Title:** Standardization of copper back-titrant by EDTA

**Scope:** Standardization of copper back-titrant using standard tetrasodium EDTA titrant in the determination of metals.

**Principles:** Tetrasodium EDTA ( $\text{Na}_4\text{EDTA}$ ) is the preferred reagent for the thermometric complexometric titration of metals, due to its much higher solubility than the normally used dibasic salt  $\text{Na}_2\text{H}_2\text{EDTA}$ .

Some metals such as nickel and cobalt react too slowly with EDTA to be suitable candidates for determination by direct thermometric titration. In these cases, a known excess of standard EDTA is added to an aliquot of the sample, sufficient time allowed to permit complete complexation, and the excess EDTA back-titrated with a standard solution of a metal (such as copper) with fast complexation kinetics.

The thermometric titration of copper 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, which is slightly more exothermic than that of the Cu-EDTA complex.

**Reagents:** Titrant. 1 mol/L tetrasodium EDTA – Dosino #1  
Buffer:  $\text{NH}_3/\text{NH}_4\text{Cl}$  solution, pH 10. Dissolve 70g  $\text{NH}_4\text{Cl}$  in 688mL conc.  $\text{NH}_3$  soln. and make to 1000mL with D.I. water – Dosino #2  
Copper back-titrant. Prepare an approximately 1 mol/L solution from A.R.  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$  – Dosino #3

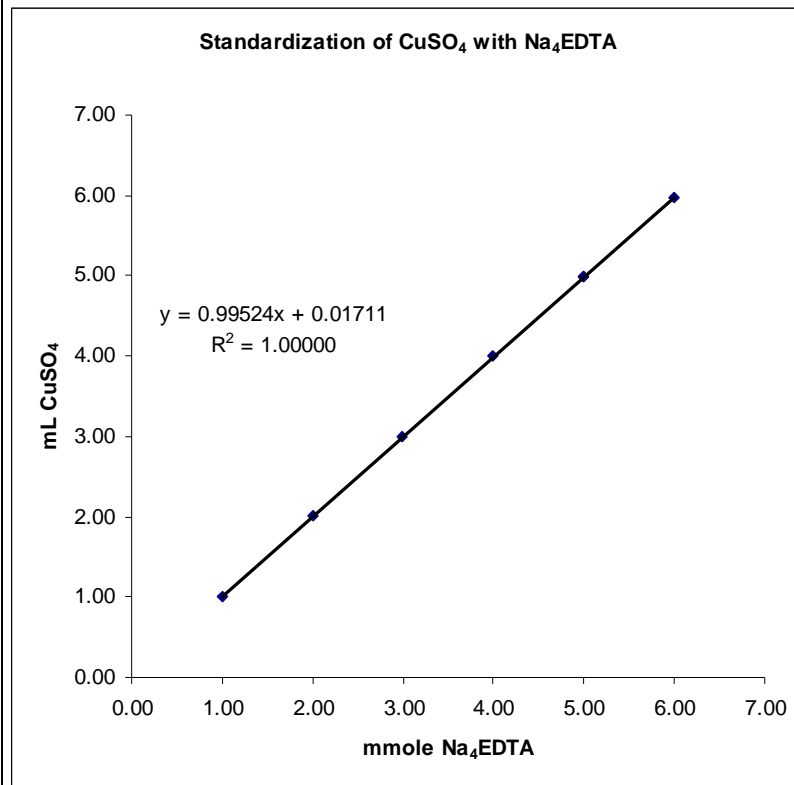
<b>Method:</b>	Basic Experimental Parameters:	
	Titrant delivery rate (mL/min.)	2
	No. of endpoints	1
	Data smoothing factor	50
	Stirring speed (802 stirrer)	6
	Delay before start (secs.)	15
	Buffer pre-dose (from Dosino #2), mL	5
	Copper solution pre-doses (from Dosino #3), mL	1, 2, 3, 4, 5, 6
	Measure 20mL D.I. water into titration vessels and use the titration program to titrate aliquots of CuSO <sub>4</sub> solution ranging from 1 – 6 mL.	

<b>Results (example):</b>		
	Cu <sup>2+</sup> aliquot, mL	Titre Na <sub>4</sub> EDTA, mL
Titrant: 0.9990 mol/L Na <sub>4</sub> EDTA	1	5.977
	2	4.991, 4.989
	3	4.000
	4	2.996
	5	2.005
	6	1.011

**Determination of titrant strength and method blank:**

Molarity = 1/gradient  
= 1/0.99524  
= 1.0048 mol/L

Method blank  
= y-intercept = 0.0171 mL



**Thermometric Titration Plot:**

**Legend:**

*Red = solution  
temperature curve*

*Black =second  
derivative curve*

