

# Thermo. Titr. Application Note No. H-044

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

**Scope:** Standardization of tetrasodium EDTA titrant for use 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  $\text{Na}_2\text{H}_2\text{EDTA}$ .

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

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.

Standard copper solution. Degrease and dry sufficient high purity copper foil to make 500mL of a 0.2 mol/L Cu(II) solution. Place the weighed amount into a 250mL wide mouth erlenmeyer flask, together with a PTFE coated magnetic spin bar. Transfer to a fume hood. Add 30mL concentrated A.R. nitric acid through a funnel which is intended to prevent loss of Cu. After the initial effervescence has subsided, ensure that all copper has dissolved before washing down the sides of the beaker with DI water. Make the volume to approximately 150mL with DI water, and place on a hot plate magnetic stirrer. Boil the solution while stirring vigorously for approximately 30 minutes, to ensure that nitrogen oxides have been expelled. Finally, cautiously add ~1g sulfamic acid in small portions to eliminate the last traces of nitrogen oxides. Cool, and make to volume with DI water in a 500mL volumetric flask.

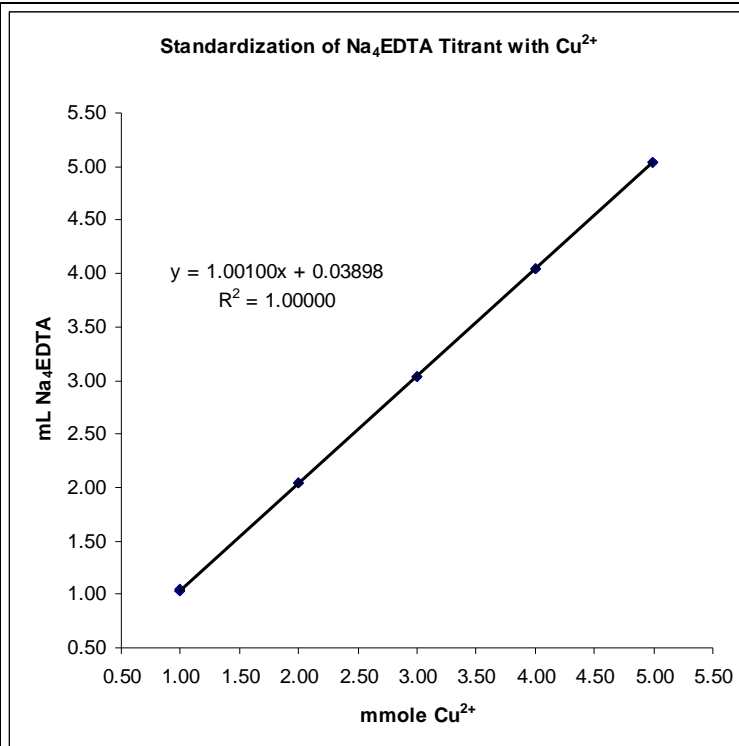
<b>Method:</b>	Basic Experimental Parameters:	
	Titrant delivery rate (mL/min.)	2
	No. of endpoints	1
	Data smoothing factor	50
	Stirring speed (802 stirrer)	6
	Delay before start (secs.)	15
	Buffer pre-dose (from Dosino), mL	5
Pipette aliquots of Cu standard solution into a titration vessel. Allowing for the addition of 5mL of buffer solution, make up the difference in the volume to 30mL with DI water. Titrate to change in gradient in the temperature curve.		

<b>Results (example):</b>			
	<b>Aliquot, mL</b>	<b>mmole Cu<sup>2+</sup></b>	<b>Titre, mL</b>
6.3588 Cu (certified min. 99.9%) dissolved and made to 500mL	25	4.9983	5.043, 5.042
	20	3.9986	4.044, 4.038
	15	2.9990	3.041, 3.041
	10	1.9993	2.040, 2.044
	5	0.9997	1.041, 1.037, 1.038

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

Molarity =  $1/\text{gradient}$   
=  $1/1.00100$   
= 0.9990 mol/L

Method blank  
= y-intercept = 0.0390 mL



**Thermometric Titration Plot:**

**Legend:**

*Red = solution temperature curve*

*Black = second derivative curve*

