

Thermo. Titr. Application Note No. H-045

Title: Standardization of EDTA titrant by magnesium

Scope: Standardization of tetrasodium EDTA titrant for use in the determination of magnesium.

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 $\text{Na}_2\text{H}_2\text{EDTA}$.

The thermometric titration of magnesium with EDTA is carried out in an ammonia/ammonium chloride buffer (~pH 10) environment. The endpoint is marked by a slight upswing in temperature, as the reaction between EDTA and magnesium is endothermic.

Reagents: Titrant. 1 mol/L tetrasodium EDTA

Buffer: $\text{NH}_3/\text{NH}_4\text{Cl}$ solution, pH 10. Dissolve 70g NH_4Cl in 688mL conc. NH_3 soln. and make to 1000mL with D.I. water.

Standard magnesium solution. Sufficient A.R. magnesium metal (ribbon) to make 500mL of a 0.2 mol/L solution is scraped clean of an oxide to a uniform shiny surface and degreased. After weighing, it is transferred to a wide mouth erlenmeyer flask, and covered with 100mL D.I. water. 25mL conc. HCl is added slowly through a funnel in the mouth of the flask, maintaining a steady effervescence until all the metal is dissolved. The solution is cooled, and quantitatively transferred to a 500mL volumetric flask, making to volume with D.I. water.

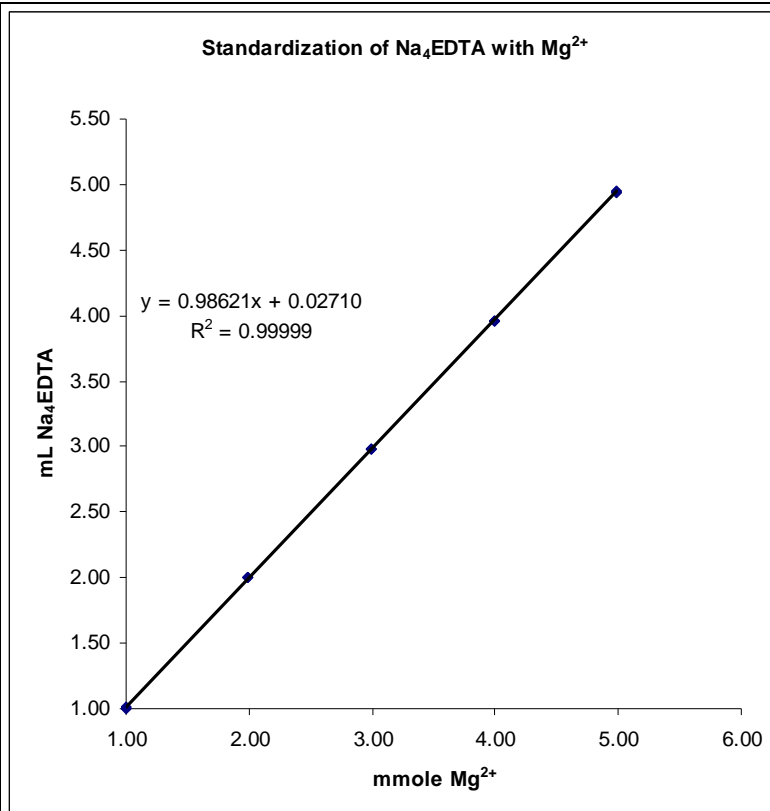
Method:	Basic Experimental Parameters:	
	Titrant delivery rate (mL/min.)	2
	No. of endpoints	1
	Data smoothing factor	50
	Stirring speed (802 stirrer)	6
	Delay before start (secs.)	15
	Buffer pre-dose (from Dosino), mL	5
Pipette aliquots of Mg standard solution into a titration vessel. Allowing for the 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.		

Results (example):			
For this exercise, ~99% Mg (Lab. Reagent grade) was used.	Aliquot, mL	mmole Mg ²⁺	Titre, mL
	25	4.9893	4.941, 4.947
	20	3.9914	3.965, 3.965
	15	2.9936	2.982, 2.981
	10	1.9957	2.001, 2.001
	5	0.9979	1.004, 1.007

Determination of titrant strength and method blank:

Molarity = 1/gradient
= 1/0.98621
= 1.0140 mol/L

Method blank
= y-intercept = 0.0271 mL



Thermometric Titration Plot:

Legend:

*Red = solution
temperature curve*

*Black =second derivative
curve*

