

Thermo. Titr. Application Note No. H-075

Title: Standardization of tetrasodium EDTA solutions

Scope: Standardization of ~1mol/L tetrasodium EDTA solutions for thermometric complexometric analysis.

Principle: $Mg^{2+} + Y^{4-} \leftrightarrow MgY^{2-}$ (Y = EDTA)
Thermodynamic Constant:
Heat of chelation, Mg^{2+} with EDTA: $\Delta H_r \approx +20.1$ KJ/mol
The reaction with Mg^{2+} is thus endothermic.

Reagents:

3.1. 1mol/L Na_4EDTA . Prepare from A.R. tetrasodium EDTA. Alternatively, weigh 372.24g A.R. Na_2H_2EDTA and quantitatively transfer to a 1000mL volumetric flask. 80g A.R. NaOH is carefully dissolved in 500mL D.I. water, cooled, and added to the same flask. When all solids have dissolved (with the addition of more water), make to volume and mix well.

3.2. NH_3/NH_4Cl buffer. Dissolve 17.5 g A.R. NH_4Cl in 172 mL A.R. conc. (28%) NH_3 soln. and make to 250 mL with deionised water.

3.3. 0.2 mol/L Mg^{2+} solution. Prepare from A.R. Mg metal (ribbon). Remove oxide film with emery paper until the surface is uniformly bright and smooth. Wash in 2-propanol and dry. Weigh out an amount calculated to give a 0.2 mol/L solution in the desired volume. Transfer the metal quantitatively to the appropriate volumetric flask. Add some DI water, then add slowly an amount of concentrated HCl calculated to give a slight excess over the required stoichiometric quantity. Tilt the volumetric flask slightly to prevent losses up the neck of the flask during the effervescent dissolution of metal. After all metal has dissolved, make to volume with DI water.

Method:
Basic Experimental Parameters:

Titrant delivery rate (mL/min.)	4
No. of endothermic endpoints	1
Data smoothing factor (DSF)	70
Stirring speed (802 stirrer)	15
Delay before start of titration (secs.)	10

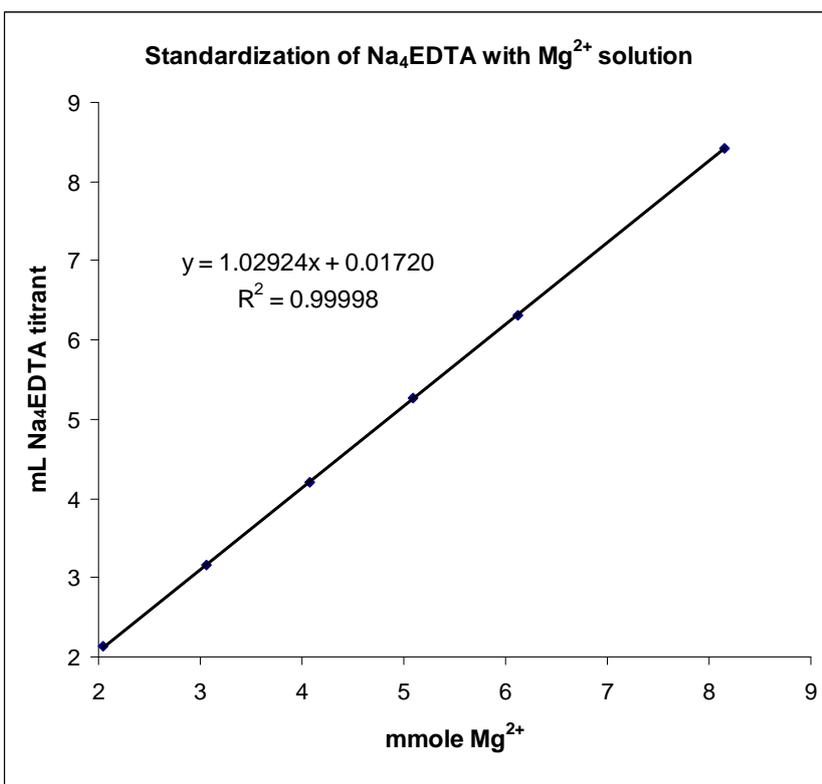
Pipette volumes of 10, 15, 20, 25 and 30mL of standard 0.2mol/L Mg^{2+} solution into a titration vessel. Add 5mL buffer solution to each solution and titrate to an endothermic endpoint.

Plot mmole Mg (x-axis) against mL of Na_4EDTA to the endpoint (y-axis). Calculate molarity of the Na_4EDTA titrant from the reciprocal of the gradient.

Example of standardization plot:

From plot, gradient = 1.02924

Molarity of Na_4EDTA solution = $1/1.02924$
 $= 0.9716 \text{ mol/L}$



Thermometric Titration Plot:

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

