

C 241 (Experiment No. _____)

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VOLUMETRIC ESTIMATION OF METAL IONS Ca(II), Mg(II) AND Cu(II) BY EDTA (COMPLEXOMETRIC TITRATION)

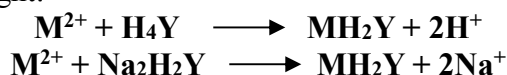
AIM

To estimate the amount of Ca(II), Mg(II) and Cu(II) present in the given unknown solutions volumetrically (complexometric titration) using a chelating agent EDTA.

GENERAL THEORY

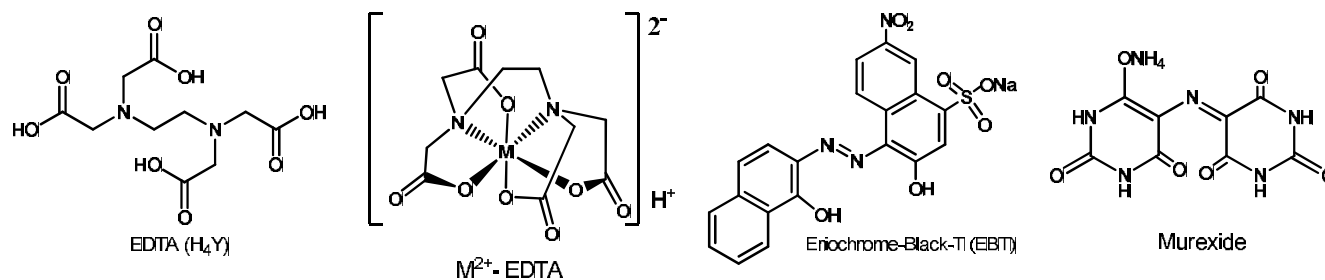
Complexometric titrations are one of the analytical applications of complexation reaction between metal ion and a chelating agent *viz.* ethylenediaminetetraacetic acid, EDTA. Because of involvement of chelating agent it is known as chelometric or chelatometric titration or chelometry. EDTA has four carboxyl groups and two amine groups that can act as electron pair donors or Lewis bases. The ability of EDTA to potentially donate six lone pairs of electrons for the formation of coordinate covalent bonds to metal cations makes EDTA a hexadentate ligand.

However, in practice EDTA is usually only partially ionized, and thus forms fewer than six coordinate covalent bonds with metal cations. Disodium EDTA is commonly used to standardize the aqueous solutions of transition metal cations. Disodium EDTA (often written as $\text{Na}_2\text{H}_2\text{Y}$; $\text{Y} = \text{EDTA}$) a multidentate ligand only forms four coordinate covalent bonds to metal cations at pH values ≤ 12 . In this pH range, the amine groups remain protonated and are thus unable to donate electrons to the formation of coordinate covalent bonds. The shorthand form $\text{Na}_{4-x}\text{H}_x\text{Y}$ can be used to represent any species of EDTA, with 'x' designating the number of acidic protons bonded to the EDTA molecule. EDTA forms an octahedral complex with most 2+ metal cations, M^{2+} , in aqueous solution. The main reason that EDTA is used so extensively in the standardization of metal cation solutions is that the formation constant for most metal cation-EDTA complexes is very high, meaning that the equilibrium for the reaction lies far to the right:



Carrying out the reaction in a basic medium, the buffer solution removes H^+ as it is formed, which also favors the formation of the EDTA-metal cation complex reaction product. For most purposes, it can be considered that the formation of the metal cation-EDTA complex goes to completion and this is chiefly why EDTA is used in titrations/standardization of this type. To carry out metal cation titrations using EDTA, it is almost always necessary to use a complexometric indicator to determine when the end point has been reached. Common indicators are organic dyes such as Eriochrome Black-T, Eriochrome Red B or Murexide. These dyes bind to the metal cations in solutions to form colored complexes.

However, since EDTA binds to metal cations much more strongly than does the dye used as an indicator, the EDTA will displace the dye from the metal cations as it is added to the solution of analyte. A color change in the solution being titrated indicates that all of the dye has been from the metal cations in solution, and that the end point has been reached.



MATERIALS REQUIRED

EDTA, $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$ (std), $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$, $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$, $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$, Conc. NH_3 , NH_4Cl , KNO_3 ,

Erichrome Black-T, Mureoxide P, methanol, ethanol, NaOH, H₂SO₄, Burette, Pipette, Watch glass, Measuring cylinder, conical flask (250 mL), volumetric flask, distilled water, hot plate etc.

PROCEDURE:

1. Preparation of Standard ZnSO₄ solution:

Prepare 100 mL of 0.01M ZnSO₄ solution as primary standard.

2. Standardization of EDTA

Standardize unknown EDTA solution as secondary standard by primary standard ZnSO₄ solution.

1. Pipette out 10 mL of ZnSO₄ solution in to a 250 mL conical flask.
2. Add 5 mL of pH 10 buffer and dilute to 100 mL with deionized water.
3. Warm to about 40°C; add 30 mg of solid EBT+KNO₃ indicator mixture (1% ethanolic or 0.4% methanolic solution of the indicator mixture can also be used].
4. Swirl it well and titrate against EDTA solution until the color of the solution changes from wine red to blue.
5. Repeat the titration to get three concurrent results.
6. Find out the strength of EDTA solution from the table.

[Note: An unknown EDTA solution, EBT+KNO₃ indicator mixture and buffer will be provided]

S.No.	Volume of ZnSO ₄ taken (mL)	Burette Readings (mL)		Volume of EDTA consumed (mL)
		Initial	Final	
1				
2				
3				

3. Estimation of Calcium:

Ca²⁺ ions readily react with EDTA to form stable Ca-EDTA complex and it also reacts with EBT to form a less stable wine-red colored Ca-indicator complex in the pH range 7-11. EDTA at first reacts with free Ca²⁺ ions to form Ca-EDTA complex, the solution changes from wine red to blue at the end point.



1. Pipette out 10 mL of unknown Ca-salt solution into a 250 mL conical flask.
2. Add 5 mL of pH 10 buffer and dilute to 100 mL with deionized water.
3. Warm to about 40°C; add 30 mg of solid EBT+KNO₃ indicator mixture.
4. Swirl it well and titrate against EDTA solution until the color of the solution changes from wine red to blue.
5. Repeat the titration to get concurrent results.
6. Find out the strength of Ca ion in Ca-salt solution.

S.No.	Volume of Ca-salt taken (mL)	Burette Readings (mL)		Volume of EDTA consumed (mL)
		Initial	Final	

4. Estimation of Magnesium:

Mg²⁺ ions readily react with EDTA to form stable Mg-EDTA complex and it also reacts EBT to form a less stable wine-red colored Mg-indicator complex in the pH range 7-11. EDTA at first reacts with free Mg²⁺ ions to form Mg-EDTA complex and then reacts with wine-red colored Mg-indicator complex to set free blue indicator ion and so the color of the solution changes from wine red to blue at the end point.



1. Pipette out 10 mL of unknown Mg-salt solution into a 250 mL conical flask.
2. Add 5 mL of pH 10 buffer and dilute to 100 mL with deionized water.
3. Warm to about 40°C; add 30 mg of solid EBT+KNO₃ indicator mixture.
4. Swirl it well and titrate against EDTA solution until the color of the solution changes from wine red to blue.
5. Repeat the titration to get concurrent results.
6. Find out the strength of Mg ion in Mg-salt solution.

S.No.	Volume of Mg-salt taken (mL)	Burette Readings (mL)		Volume of EDTA consumed (mL)
		Initial	Final	

5. Estimation of Copper:

You have estimated Ni & Cu gravimetrically and iodometrically respectively in previous experiments. You can estimate these by complexometric titrations also using standard EDTA. Here you have to estimate Cu by complexometric titration. Apply murexide indicator for this titration (Eriochrome Black-T can also be used). During the titration, the EDTA²⁻ forms a more stable complex with copper and frees the indicator; which changes yellow to green near to the endpoint and immediately to slight purple-blue at the exact endpoint.

1. Weigh accurately ~0.1 gm of the given cupric salt and note down the exact weight taken.
2. Dissolve it in 50 mL of deionized water in a conical flask.
3. Add 5-6 drops of murexide indicator to it. The mixture in the flask turns to yellow color.
4. Titrate it against standard EDTA until the color in the flask changes to green near the endpoint and then immediately to slight purplish blue at the exact endpoint.
5. Note down the reading and repeat the procedure for concurrent readings.
6. You can also repeat the procedure with different weight of cupric salt to get concurrent Cu%.

S.No.	Amount of Cu(II) taken (gm)	Burette Readings(mL)		Volume of EDTA consumed (mL)
		Initial	Final	

Calculation and results

Strength of EDTA = _____

Strength of Ca-salt= _____

Strength of Mg-salt= _____

Strength of Cu-salt= _____

Amount of Ca(II) and Mg(II) in the unknown solutions = _____

% Cu content in the Cupric salt = _____ %.