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Complexometric Titration

UNIT-III

- **Precipitation titrations:** Mohr's method, Volhard's, Modified Volhard's, Fajans method, estimation of sodium chloride.
- **Complexometric titration:** Classification, metal ion indicators, masking and demasking reagents, estimation of Magnesium sulphate, and calcium gluconate.
- **Gravimetry:** Principle and steps involved in gravimetric analysis. Purity of the precipitate: co-precipitation and post precipitation, Estimation of barium sulphate.
- Basic Principles, methods and application of diazotisation titration.

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Complexometric Titration

Classification

1. Metal ion indicators
2. Masking and Demasking reagents
3. Estimation of Magnesium Sulphate
4. Estimation of Calcium gluconate

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Complexometric Titration

- It is a type of titration in which the formation of a colored complex is used to indicate the end point.
- It is particularly used for the determination of mixture of different metal ions in a solution.

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Principle

- In this titrations, the metal ions are titrated with a complexing agents (ligands) or a chelating agents or EDTA.
- It involves the transformation of simple metal ions into complex ion and metal ion indicator used for the determination of end point by changing the color.
- It is also known as chilometric titration, chelatometric, and EDTA titrations.



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Ligands

These are complexing or chelating agents which have ability to donate electrons and bind with metal ions to produce a complex ion.

They are of following:-

Monodentate/ Unidentate:- These ligands are bound to metal ions only at one place or only have one donor atom.

Eg :- NH_3 , Halide ions etc.



(Monodentate ligand)

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Ligands

Bidentate :- These ligands are bound to metal ions at two place or have two donor atom

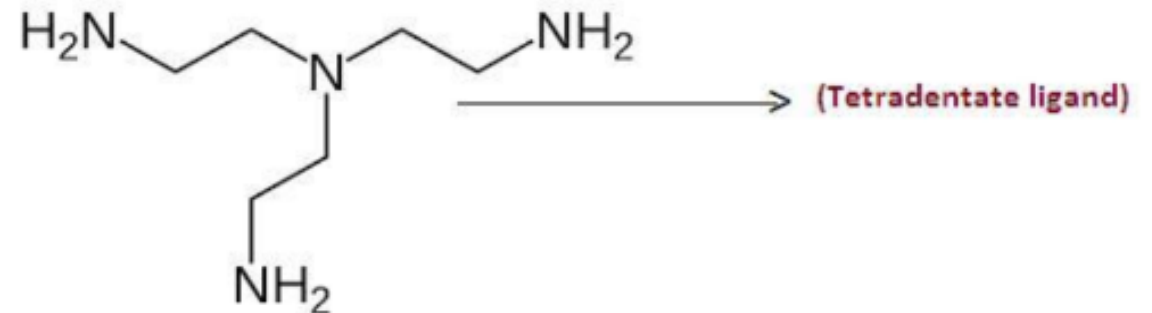


Eg :- Ethylene diamine

Multidentate Ligands:- These ligands are bound to metal ions at more than one or two place or have more than one donor atom.

Eg :- EDTA [$C_{10}H_{16}N_2O_8$]

(Ethylene Diamine Tetra Acetic Acid)



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Classification of Complexometric Titration

It is of four types-

Direct Titration :- It is similar to acid-base titrations which involves the titration of metal ion solution against chelating agents till end point.

Indirect Titration :- It is similar as Volhard Methods, in which the excess amount of EDTA (ligand) solution is used for the titration of metal ion solution, then this excess EDTA is titrated with standard solution of a second metal ion.

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Replacement Titration :- In this, the metal ion to be analyzed is displace the metal from the metal ligand complex.

It is used when direct or indirect titration do not give sharp end point.



Alkalimetric Titration :- It is used for the determination of anions which do not react with EDTA chelate.

Protons (H^+) from disodium EDTA are displaced by a heavy metal and titration with sodium alkali.

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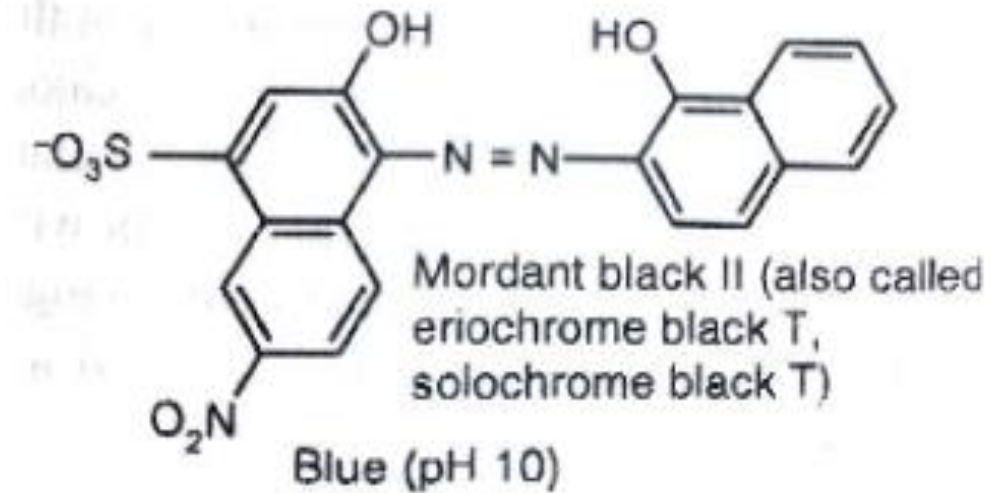
Metal Ion Indicators

- ❑ These are those indicators which are used in complexometric titration.
- ❑ It is used for the determination of end point of metal ions, that's why it is known as metal ion indicator.
- ❑ These indicators show different of two colors in different conditions i.e. different in its free state and different color in its complex state.
- ❑ Also known as pH indicators.

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Metal ion indicators (Complexometric titration indicators)

1. Eriochromeblack-T



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2. Xylenol orange



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3. Murexide



Murexide

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Characteristics

1. It should be chemically stable.
2. The Dye-metal complex formed should be of equal ratio.
3. The colour of the indicator should differ from colour of the metal ion.
4. It should be selective.
5. It should not complete with the EDTA.

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Applications of complexometric Titration

Used in determination of

- Aluminium hydroxide gel
- Aluminium sulphate
- Calcium chloride
- Calcium gluconate
- Magnesium sulphate

Also used for determination of permanent and temporary hardness of water separately.

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Masking Agent & Demasking Agent

Masking Agent:- The agent which prevent interfering ion from reaction without physical separation.

This reagent form complexes with interfering ion which are most stable then complex form with indicator & EDTA.

Eg:-

- a) KCN which is used for Ag, Cu, Co, Cd ion.
- b) Tri ethanol amine which is used for FeAl etc.
- c) Ammonium fluoride which is used for ferric ion & Al ions

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Demasking Agents

Agent which replaces the mask ion & this ion now can take part in reaction.

Eg:- Masking of CN ion can be removed by mixture HCHO & chloral hydrate.

Various other methods of demasking are:-

1. Decomposition of masking agent.
2. By changing pH.
3. By changing the oxide state of ion
4. Or Volatisation of one of the component of masked system.

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Estimation of Magnesium Sulphate

It can be determined by complexometric titrations, EDTA is used as titrant.

Molecular Formula:- $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$

Molecular Weight:- 246.4g/mol

Principle:-

EDTA reacts with magnesium sulphate and forms complex and for end point use mordant black-2



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Estimation of Magnesium Sulphate

Procedure :-

1. About 0.3gm of magnesium sulphate heptahydrate is dissolved in 50ml of distilled water.
2. Add 10ml of strong ammonia-ammonium chloride solution.
3. Now, this mixture is titrated with 0.05M disodium EDTA, by using 0.1gm of mordant black-2 mixture as indicator.
4. Titration is continued till the blue color is obtained.
5. Each ml of 0.05M disodium EDTA = 0.00602gm of MgSO_4

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Estimation of Calcium Gluconate

It is also determined/ estimated by complexometric titration by using disodium EDTA as titrant and mordant black-2 as an indicator.

Calcium gluconate :- $C_{12}H_{22}CaO_{14}$

Molecular weight :- 430.373g/mol

Principle:-

EDTA reacts with calcium gluconate and forms complex and for end point used mordant black-2 as an indicator.



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Estimation of Calcium Gluconate

Procedure:-

1. About 0.4gm of calcium gluconate taken in conical flask and dissolve in 50ml of warm water.
2. Add 5ml of 0.05M disodium EDTA.
3. Now, add 10ml of strong ammonium-ammonium chloride solution.
4. Add 2-3 drops of mordant black-2 as indicator.
5. Titration is continued till color changes from wine red to pure blue (indicates the end point).
6. Each ml of 0.05M EDTA = 0.02242gm of Calcium gluconate.