ست آمنة عدنان

ملزمة اللاعضوية العملي (الكورس الثاني)
المرحلة الثالثة (علوم كيمياء)

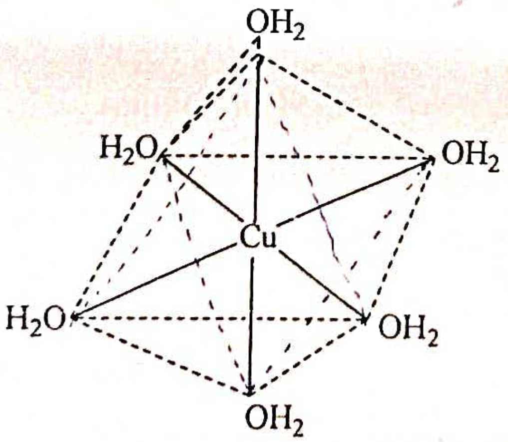
Experiment No. 7:

Preparation and spectral study of some copper (II) complexes

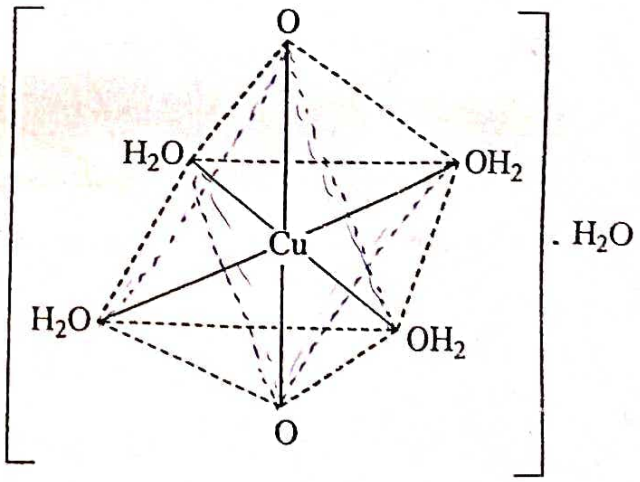
Theory:

Oxidation state (+2) is the most important oxidation states of copper. It is the most stable oxidation state with electronic configuration [Ar]3d^9. Throughout copper (II) chemistry, Jahn – Teller distortions are observed although the degree of distortion varies considerably.

Aqueous solutions of Cu^{+2} contain $[Cu(H_2O)_6]^{+2}$ ion and this has been isolated in several salts including $Cu(ClO_4)_2.6H_2O$. The solid state structures of this salt reveal distortions of $[Cu(H_2O)_6]^{+2}$ such that there are three pairs of Cu-O distances, Cu-O bond lengths are 209, 216 and 228 pm crystals of the blue hydrated sulfate $CuSO_4.5H_2O$ contain square planer $[Cu(H_2O)_4]^{+2}$ units with two sulfate O atoms completing the remaining sites in an elongated octahedral coordination sphere. The solid state structure incorporates a non-coordinated H_2O molecule.



$[Cu(H_2O)_6](ClO_4)_2$

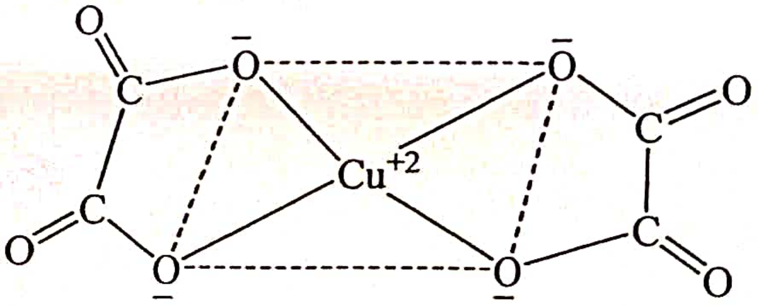


$[Cu(H_2O)_4(SO_4)].H_2O$

Complexes containing N-and O-donor ligands are very common, and coordination number of 4, 5, and 6 predominate.

When NH_3 is added to aqueous Cu^{+2}, only four aqua (H_2O) ligands in $[Cu(H_2O)_6]^{+2}$ are replaced and the complex $[Cu(NH_3)_4]^{+2}$ formed. The complex $[Cu(NH_3)_6]^{+2}$ can be made in liquid NH_3. The complex $[Cu(en)_3]^{+2}$ is formed in very concentrated aqueous solutions of ethylenediamine. Deep blue aqueous $[Cu(NH_3)_4](OH)_2$ formed when $Cu(OH)_2$ is dissolved in aqueous NH_3.

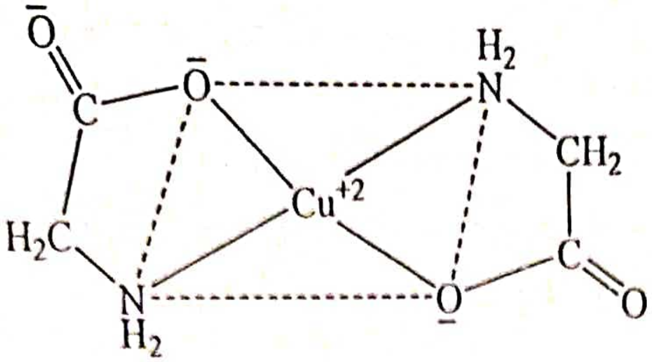
Further examples of complexes with N- and O-donor ligands are the square planer bis(oxalate) Cu(II) complex $[Cu(ox)_2]^{-2}$ in which the bidentate oxalate [ox = $C_2O_4^{2-}$] ligands coordinated to Cu(II) ions through two oxygen atoms. The cis- and trans- bis(glycenato) Cu(II) complexes in which the glycine ligand act as mononegative bidentate coordinating through amino nitrogen and carboxylate oxygen atoms. The complex $[Cu(en)_2]^{+2}$ containing two bidentate ethylenediamine ligand coordinated through the amino nitrogen atoms, as shown below.



$[Cu(ox)_2]^{2-}$

Oxalato-complex

ox = $C_2O_4^{2-}$, dinegative bidentate ligand



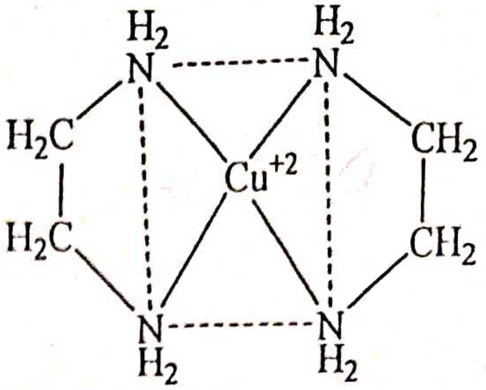
$[Cu(gly)_2]$

Glycinato - complex

gly = $NHCH_2COO^-$

uninegative

bidentate ligand



$[Cu(en)_2]^{+2}$

ethylenediamine complex

en = $NH_2CH_2CH_2NH_2$

neutral bidentate ligand

Procedure:

A . Preparation of the complex:

Bis(glycinato) copper(II) dihydrate $[Cu(gly)_2].2H_2O$

1. Dissolve (1.05 g) of hydrated copper sulphate ($CuSO4.5H_2O$) in (10 ml) of distilled water in 150 ml capacity beaker, then heat the solution to boiling temperature for (10 min.), keeping the volume of the solution of (10 ml) by adding distilled water during heating.
2. Weigh (0.85 g) glycine and dissolve it in (4.5 ml) of 2M sodium hydroxide (NaOH) in a beaker and heat at boiling point for (5 min.).
3. Add the glycine solution to the copper sulphate solution prepared in step 1 above with continuous stirring .
4. Filter quickly under reduced pressure (Buchner) while it is hot and neglect the precipitate. [illegible] [illegible]

5. Cool the filtrate to room temperature, then in ice bath to complete precipitation.
6. Filter under reduced pressure (Buchner) then wash the precipitate with (6 ml) ice-cold water and (4 ml) ethanol. [illegible]
7. Dry the precipitate in an oven at 60 °C for one hour.
8. Weigh the precipitate and calculate the % yield.

B . Preparation of the complex:

Bis(ethylenediamine) copper(II) sulphate dihydrate

$[Cu(en)_2]SO_4.2H_2O$

1. Weigh (0.55 g) of copper sulphate hydrate ($CuSO_4.5H_2O$) and dissolve it in (4 ml) distilled water in a 100 ml beaker.
2. Prepare solution of (4.15 ml) 95% ethanol, (4.15 ml) distilled water, and (4.15 ml) of 100% ethylenediamine in another 100 ml beaker.
3. Add the solution of copper (II) sulphate prepared in step 1, to the ethylenediamine solution prepared in step 2 with shaking.
4. Add (10 ml) 95% ethanol from a burette with continuous stirring during (20 min.).
5. Filter the precipitate and wash it with ethanol (5 ml) then with ether (5 ml).
6. Dry the precipitate in an oven at 60 °C for one hour.
7. Weigh the precipitate and calculate the % yield.

C. Preparation of the complex:

Potassium dioxalato cuprate (II) dihydrate $K_2[Cu(C_2O_4)_2].2H_2O$

1. Weigh (1.05 g) copper (II) sulphate hydrate ($CuSO_4.5H_2O$) and dissolve it in (2 ml) distilled water in a 100 ml beaker, then heat the solution to 90 °C for (5 min.) (add distilled water to keep the volume of solution 2 ml).
2. Weigh (3.05 g) potassium oxalate hydrate ($K_2C_2O_4.H_2O$) and dissolve it in (8.5 ml) distilled water in a 100 ml beaker then heat at 90 °C for (5 min.) [keep the volume of solution (2 ml) as above].
3. Add copper (II) sulphate solution (prepared in step 1) to potassium oxalate solution quickly with continuous stirring.
4. Cool the solution to about 10 °C in an ice-bath..
5. Filter the precipitate and wash it with cold water (4 ml) then with ethanol (5 ml) and finally with ether (5 ml).
6. Dry the precipitate in an oven at 60 °C for at least one hour.
7. Weigh the dry precipitate and calculate the % yield.

D. Study the visible spectra of the complexes.

1. Prepare (50 ml) of 0.01M solution (using volumetric flasks) of each of the prepared complexes (the glycinato-complex, A; the ethylenediamine-complex, B; and the oxalato-complex, C) in distilled water, use the following equation to calculate the required weight.

$$\text{M (molarity)} = \frac{\text{W (weight)} \times 1000}{\text{M.W (molecular weight)} \times \text{V(required volume)}}$$

2. Prepare three diluted solution for each complex by pipating 5, 10 and 15 ml of the stock solution of each complex (prepared in step 1) and transfer it to three 50 ml volumetric flasks and complete the volume to the mark by distilled water.
3. Record the visible spectrum for each complex at the range (450 – 850 nm) by measuring the absorbance of the stock solution of each complex at various water lengths to find the maximum absorbance wavelength λmax for each complex (using spectrophotometer).
4. Measure the absorbance of the three diluted solution of each complex (prepared in step 2) at λmax of each complex.
5. Plot the relation between absorbance versus wavelength for each complex to find λmax for each complex graphically.
6. Plot the absorbance versus the concentration (mol / l) for the three diluted solutions of each of the prepared complexes at λmax (note that the straight line passing through the origin), find the slope and apply beer's-Lambert law:

$A = \varepsilon C L$

Where A = absorbance , ε = molar extinction coefficient.

C = concentration (mol / liter);

L = cell width usually 1 cm.

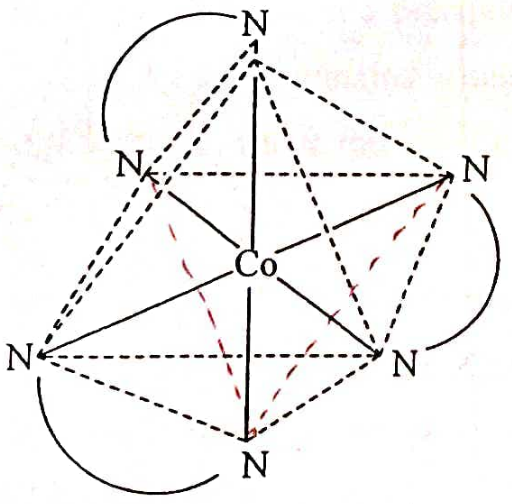
Experiment No. 8:

Optical isomers

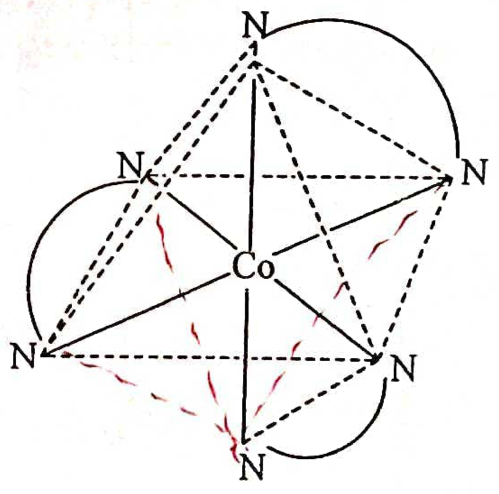
Theory:

Optical isomers formed by a chiral molecules. A molecule is chairal if it is non-superposable on its mirror image. Another commonly used criterion for identifying a chiral species is the lack of an inversion مركز انقلاب centre, I, and plane of symmetry, σ.

6-coordinated complexes such as $[Co(en)_3]^{+3}$. In which there are three bidentate chelating ligands possess non-superposable mirror images (Fig.1). Chiral molecules can rotate the plane of polarized light. This property is known as optical activity and the two mirror images are known as optical isomers or enantiomers. Enantiomers rotate the light to equal extents, but in opposite directions, the dextrorotatory (d) enantiomer to the right and the laevorotatory (ℓ) enantiomer to the left. If the two enatiomers interconvert rapidly to give an equilibrium mixture containing equal amounts of the two forms, no overall rotation occurs. A mixture of equal amount of two enantiomers is called a "racemic mixture".

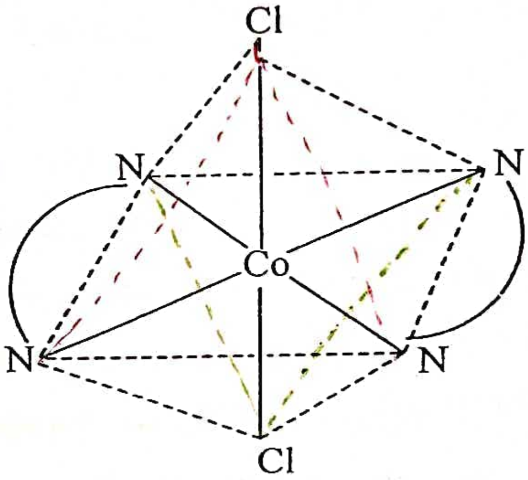


d-$[Co(en)_3]^{+3}$

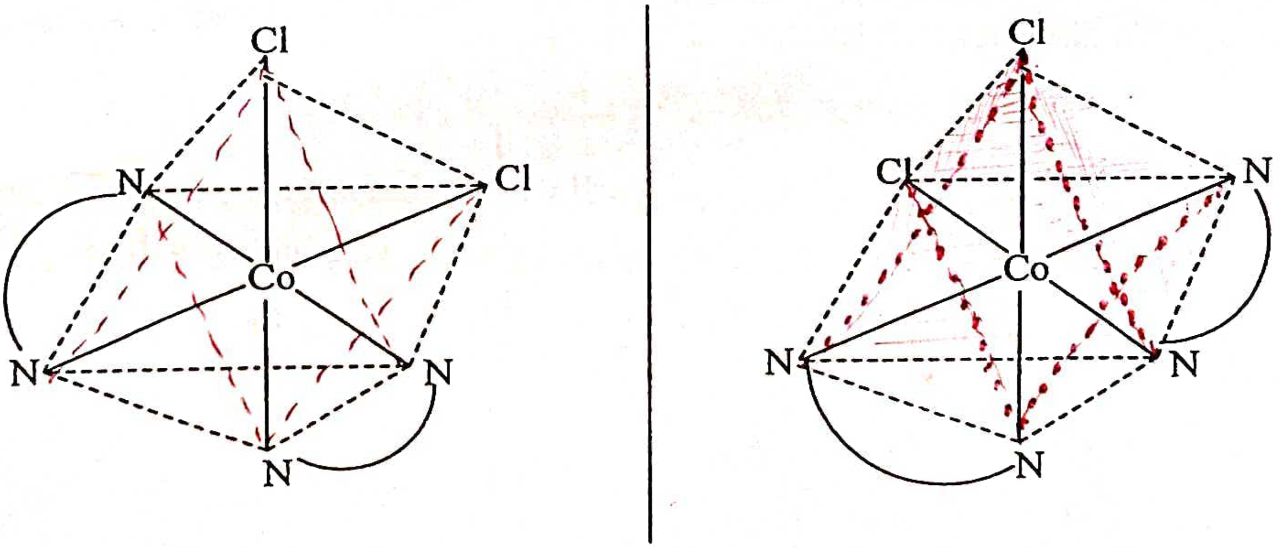


l-$[Co(en)_3]^{+3}$

Enantiomers of a coordination compound most often occur when chelating ligands are involved as shown by the octahedral tris-chelate complexes (Fig.1). For octahedral bis-chelate complexes such as $[Co(en)_2Cl_2]^+$. In this case, only cis-isomer possesses enantiomers (optical isomers); the trans-isomer is optically in-active (have a plane of symmetry) (Fig.2).



trans-isomer
opticaly inactive



d-$[Co(en)_2Cl_2]^+$

l-$[Co(en)_2Cl_2]^+$

Cis-isomer

Laboratory preparation of compounds that can exhibits optical activity almost invaraiably yield 50-50 (racemic) mixtures of the two optical isomers (enantiomers) and hence an optically inactive material. Therefore, the basic step in the laboratory preparation of an optically active coordination compound is separation from its optical isomer.

Racemic $[Co(en)_3]^{+3}$ is readily prepared by the air oxidation of a cobalt (II) salt in a medium containing excess ethylenediamine and a catalytic amount of activated charcoal. Since optical isomers are so very much a like, special separation techniques are required.

The most common techniques involve the principle that each of a pair of optical isomers will interact differently with a third optically active material. There is a subtle structural difference between optical isomers, and this will lead one isomer to be more strongly attracted to a third asymmetric molecule. For example, the salt d-$[Co(en)_3]$ (d-tartrate)$Cl.5H_2O$ is less soluble than l-$[Co(en)_3]$ (d-tartrate)$Cl.5H_2O$. This indicate that d-$[Co(en)_3]^{+3}$ forms a more stable crystalline lattice with d-tartarate than does l-$[Co(en)_3]^{+3}$. Therefore, the addition of a solution containing d-tartarate anion to a concentrated solution of racemic $[Co(en)_3]^{+3}$ causes the precipitation of d-$[Co(en)_3]$ (d-tartrate)$Cl.5H_2O$. The l-$[Co(en)_3]^{+3}$ remains in solution and can be collected by the addition of I^- solution, which forms l-$[Co(en)_3]^{+3}I_3$.

The rotation of plane of polarized light, α, may be measured in an instrument called a polarimeter. In practice, the amount of rotation depends upon the wavelength of the light, temperature and the concentration of compound present in solution. The specific rotation [α] is given by equation :

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الدوران النوعي $$[\alpha]_{\lambda}^{t} = \frac{\overset{\theta}{\alpha}}{C \times L}$$ طول الانبوبة

Where ; α = observed rotation.

L = path length of solution in polarimeter (in dm).

c = concentration (in g / cm^3).

λ = wavelength of the light.

تشبع معقد

Procedure:

A . Preparation of cobalt (II) d-tartarate:

1. Dissolve (2.8 g) cobalt (II) sulphate heptahydrate ($CoSO_4.7H_2O$) in (7.5 ml) distilled water at 60 °C, and (2.3 g) sodium d-tartarate in (7.5 ml) distilled water at 65 °C.
2. Mix the two solutions and keep the temperature of the mixture at 65 °C with constant stirring for (15 min.).
3. Cool the mixture in an ice-bath for (20 min.). After complete precipitation, filter (using Buchner) then ~~wash~~ the precipitate with (15 ml) of ice-cold water followed by acetone (5 ml) and finally dry with air of water suction. بدون غسل

B .Preparation of d-[Co(en)3] (d-tartrate)Cl.5H2O:

1. Put (10 ml) of aquous alcoholic solution of ethylenediamine in a 250 ml conical flask, and add (1 ml) concentrated hydrochloric acid dropwise, keeping the mixture temperature at 4-8 °C.
2. Add cobalt (II) d-tartarate (all the product obtained in section A above). The colour of solution change gradually to orange. Add (0.4 g) charcoal. Oxidize Co(II)-complex to Co(III)-complex by bubling air through the solution using glass tube passing through rubber stopper to the bottom of the flask for two hours (using water suction). Shake the solution during bubling with air.

3. Filter the mixture (using Buchner) and wash the precipitate with (6 ml) of 40% aqueous ethanol.
4. Add hot water (60 °C) in portions to the precipitate (dextro-isomer) in the filtration funnel as quick as possible to prevent racimization until a colourless filtrate obtained. Neglect the charcoal in the filtration funnel.
5. Evaporate the filterate to 10 ml.

C .Preparation of the complex:

d-Tris-etheylenediamine cobalt (III) iodide, d-$[Co(en)_3]I_3.H_2O$

1. To the filtrate (10 ml) (prepared in section B), add (0.5 ml) concentrated ammonia solution with stirring followed by solution of (6.7 g) sodium iodide in (3 ml) hot water.
2. Cool the mixture to precipitate the triiodide as reddish orange, needle like crystalline solid and filter the precipitate (using Buchner).
3. Wash the precipitate with (4 ml) of 30% sodium iodide solution then with ethanol (2.5 ml) and finally with acetone (2.5 ml).
4. Dry the precipitate (dextro) in air.
5. Weigh the complex and calculate the % yield. In the same way the levo-isomer l-$[Co(en)_3]I_3.H_2O$ can be prepared. d-$[Co(en)_3]I_3.H_2O$

Required measurements:

1. Prepare 2% aqueous solution of the prepared complex.
2. Measure the optical rotation,α , of the plane of polarized light using a polarimeter at laboratory temperature.
3. Calculate the specific rotation [α].

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Specific rotation

$$[\alpha]^{t}_{\lambda} = \frac{\overset{\theta}{\alpha}}{C \times L}$$

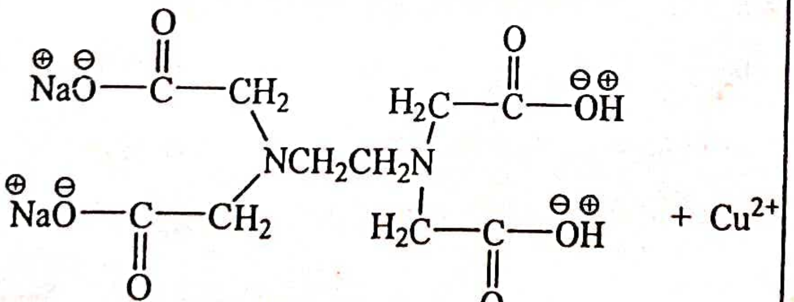
Experiment No. 9:

Study the complex formation of copper (II) and EDTA in solution

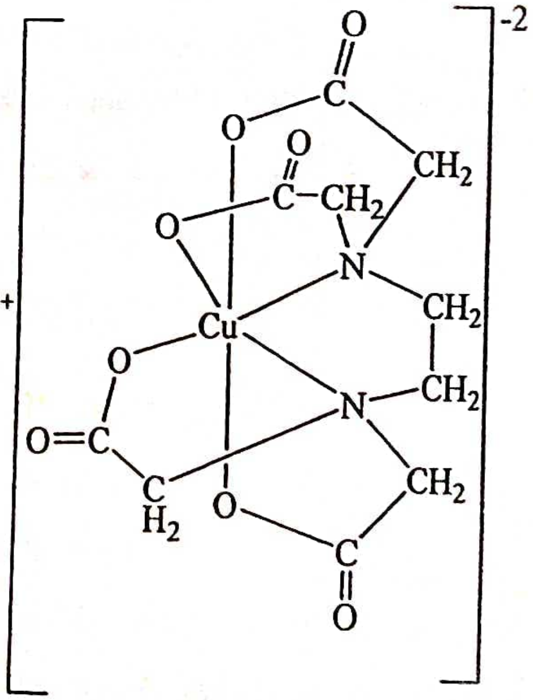
Theory:

Ethylenediaminetetraacetic acid (EDTA), a hexa-dentate ligand forms stable complexes with a wide variety of metal ions including the alkaline earth metals. This compound is used commercially as a sequestrant, a reagent that will form complexes with metal ions and in this way control their concentration in solution. For example, EDTA will very efficiently complex calcium ion and is therefore an excellent water-softening agent. It is also used as an analytical reagent.

In basic solution EDTA (H_4Y) reacts quantitatively with certain metals to produce the metal complex (Fig.1). It can therefore be used as a titrant for the volumetric determination of many metals.



EDTA-Na_2 $(H_2Y)^{2-}$
disodium salt



$[CuY]^{2-}$

$$Cu^{2+} + H_2Y^{2-} \rightleftharpoons CuY^{2-} + 2H^+$$

EDTA usually forms (1:1) (metal : ligand) complexes with transition elements acting as tetranegative hexadentate ligand coordinating through two nitrogen and four carboxyl group oxygen donor atoms, forming anionic octahedral complexes.

EDTA form very stable complexes with metal ion, which easily formed. The disodiumethylenediamine tetra acetic acid (Na_2H_2Y) salt is the most important form of EDTA and used as primary standard in metal analysis due to its solubility while the monosodium salt and the tetraacid forms are very much less soluble so it could not be used as primary standard. The tetraacid form of EDTA is weak acid. It may have Zetter-ion structure with four dissociable hydrogen ions having:

$pk_1 = 2.0$	$ka_1 = 1.02 \times 10^{-2}$
$pk_2 = 2.67$	$ka_2 = 2.14 \times 10^{-2}$
$pk_3 = 6.16$	$ka_3 = 6.92 \times 10^{-7}$
$pk_4 = 10.26$	$ka_4 = 5.5 \times 10^{-11}$

ka = dissociation constant pk = -log k

From these values it is clear that the dissociation of the first two protons is easier than the others. The four protons dissociation in basic medium (pH > 10) as follows:

$$H_4Y \xrightarrow{OH^-} H_3Y^{-1} \xrightarrow{OH^-} H_2Y^{-2} \xrightarrow{OH^-} HY^{-3} \xrightarrow{OH^-} Y^{-4}$$

Y^{4-} is the strongest chelating agents as compared with other ions of EDTA (H_3Y^-, H_2Y^{2-}, HY^{3-}) when a solution of disodiumethylenediamine - tetraacetate, (Na_2H_2Y) is added to a solution containing metallic ions, complexes are formed with libration of two equivalents of hydrogen ions:

$$M^{2+} + H_2Y^{-2} \longrightarrow [MY]^{-2} + 2H^+$$

The hydrogen ions thus set free can be titrated with standard KOH using acid-base indication or potentiometric and point (using pH –meter).

Procedure:

1. Calibrate the glass electrode of pH –meter at pH = 4 using buffer pH = 4 solution [consist of 0.05M solution of potassium hydrogen phthalate, pH = 4 at laboratory temperature.
2. Prepare the following solutions:

a. 100 ml of (0.05M) potassium hydroxide (KOH) solution.

b. 250 ml of (0.001333 M) disodium salt EDTA solution.

c. 100 ml of (0.1M) potassium chloride solution (to keep constant ionic strength of mixture).

d. 100 ml (0.004M) Cu^{+2} solution using copper sulfate pentahydrate ($CuSO_4.5H_2O$).

3. Fill the burette with potassium hydroxide solution (adjust the mark). Transfer 75 ml of EDTA solution to a suitable beaker and add to this solution 25 ml of potassium chloride solution.
4. Dip the glass electrode in the solution mixture at the beaker and record the pH of the solution from the pH-meter, titrate against KOH solution in the burette by adding gradually (0.5 ml) of KOH solution followed by shaking the mixture well and recording the pH value after each addition. Continue the KOH addition until pH ≈ 11.
5. Remove the glass electrode and rinse it with distilled water. Refill the burette with KOH solution.
6. Transfer 75 ml of EDTA solution to the beaker. Add 25 ml of copper (II) solution,

7. Titrate against KOH solution as in step 4 above by recording pH-values using pH-meter.
8. Plot the titration curves, pH versus ml of KOH added for the first titration of KOH against EDTA and the second titration of KOH against (EDTA + Cu^{2+}) solution.
9. Compaire the two titration curves and note the differences between them .
10. Find the end point (graphically) of the two titrations and calculate pk_3.

Experiment No. 10:

Preparation of spectral studies of iron (II) and nickel (II) complexes

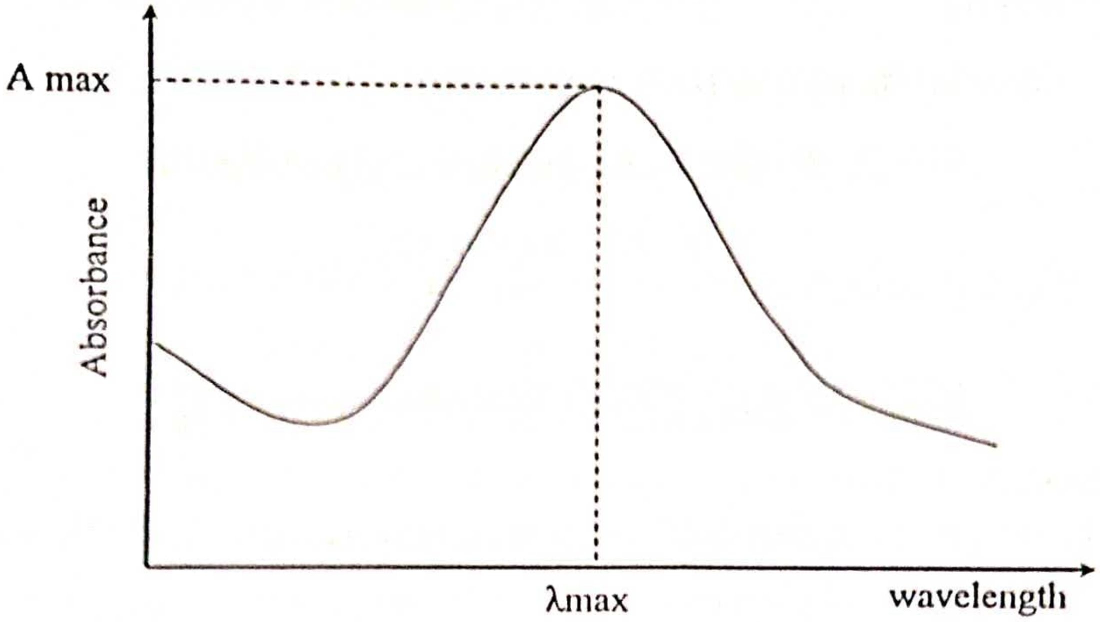
Theory:

A characteristic feature of most d-block complexes is their coloures, which arise because they absorb light in the visible region (380-700 nm) due to electronical transitions. Studies of the electronic spectra of metal complexes provide information about structure and bonding, although interpretation of the spectra is not always straight forword. Absorption arise from transitions between electronic energy levels are mainly :

a- Transitions between metal – centred orbitals possessing d-character (d-d transitions).

b- Transitions between metal – centred and ligand – centred molecular orbitals, which transfer charge from metal to ligand or ligand to metal (charge transfer bands).

Absorption bands in the electronic spectra of d-block are usually broad. They are described in terms of λmax corresponding to the absorption maximum, Amax (Fig.1). The wavelength, λmax is usually given in nm (nanometer = 10^{-9} meter), but the position of the absorption may also be reported in terms of wavenumber, $\bar{\upsilon}$ (cm^{-1}).



The molar extinction coefficient (or molar absorbtivity) ε_{max} of an absorption must also be quoted; ε_{max} indicates how intense an absorption is and is related to Amax by the equation:

$$\varepsilon_{max} = \frac{Amax}{C \times L}$$

Where C = concentration of the solution and L = the path length (in cm) of the spectrometer cell.

ε_{max} in $dm^3\ mol^{-1}\ cm^{-1}$

value of ε_{max} range from close to zero (a very weak absorption) to > 10000 $dm^3\ mol^{-1}\ cm^{-1}$ (an intense absorption).

Procedure:

A . Preparation of iron (II) complex:

Tris (1,10-phenanthroline) iron (II) perchlorate

$[Fe(1,10\text{-}phen)_3](ClO_4)_2$

(Note the poisonous nature of the compound)

1. Dissolve (0.35 g) iron (II) sulphate heptahydrate ($FeSO_4.7H_2O$) and (0.15 g) 1,10-phenanthroline in (50 ml) distilled water. After complete dissolution, filter the solution by simple filteration.
2. Add to the filtrate (4 ml) of sodium perchlorate ($NaClO_4$) solution dropwise with continuous stirring at (50 -60 °C).
3. After complete addition, heat the solution at (60 -70 °C) for one hour to complete precipitate formation.
4. Leave the solution to cool to room temperature and filter under reduced pressure (Buchner).
5. Recrystalize the precipitate by dissolving it in 15 ml hot ethanol and filter while hot (hot filtration). Evaporate to concentrate the solution by reducing the volume of solution then cool in an ice-bath and refilter under reduced pressure (use Buchner).
6. Dry the precipitate in an oven at 60°C for at lest one hour.
7. Weigh the precipitate and calculate the % yield.

B . Preparation of nickel (II) complex:

Tris (ethylendiamine) nickel (II) chloride

$[Ni(en)_3]Cl_2 2.H_2O$

1. Dissolve (6.0 g) nickel (II) chloride hexahydrate ($NiCl_2.6H_2O$) in distilled water (25 - 40 ml) in a 100 ml beaker.
2. Add (5.5 ml) of 100% ethylenediamine to the solution.
3. Filter the pink colour solution under reduced pressure (use Buchner) to remove the small amount of hydrated nickel oxide that may precipitated.
4. Evaporate by heating in water bath or heater below 100 °C to reduce the volume of solution to 10-15 ml.
5. Add to the hot solution two drops of ethylenediamine.
6. Cool the solution gradually to room temperature then in an ice-bath.
7. Filter the mixture under reduced pressure (use Buchner) and collect the pink crystals formed.
8. Dry the solid in an oven at 60 °C for at least one hour.
9. Weigh the solid and calculate the % yield.

C . Spectral measurements

Do the measurements for each complex separately

1. Weigh accurately (0.04 g) of the dry complex and dissolve it (200 ml) distilled water in a 500 ml beaker and heat to boiling temperature, to complete dissolution of the fine particles of the red solid complex.

2. Cool the solution to room temperature then transfer to 250 ml volumetric flask and complete the volume to the mark with distilled water.
3. Prepare four diluted solution of different concentrations of the complex by pipating (don't use mouth) 5, 15, 25 and 35 ml of the stock solution to four 100 ml volumetric flask, and fill to the make by distilled water.
4. Measure the spectrum of the complex over the range (300 -650 nm) of the UV-visible light, using spectrometer, of the highest dilute concentration (that contain 35 ml stock solution). Find λmax of the complex at which maximum absorbance (Amax) obtained.
5. Fix the wavelength of the spectrometer at λmax and record the absorbance (A) of the other three diluted solution (those containing 5, 15 and 25 ml stock solution).
6. Plot the absorbance (A) of the diluted solutions (recorded in step 4) versus wavelength (in nm) and find λmax graphically.
7. Plot the relation between the absorbance (A) versus concentration C (mol / liter).

   (note that the straight line passing through the origin). Find the slope of the straight line and apply Beer's –Lambert law:

   $A = \varepsilon C l$

   Where A = absorbance.

   ε = molar absorbance coefficient.

   C = concentration (molar / liter).

   l = spectrometer cell pathlength (cm)

Experiment No. 11:

Preparation and properties of chromium (III) complexes: preparation of potassium trioxalato chromate (III) trihydrate $K_3[Cr(C_2O_4)_3].3H_2O$

Theory:

Chromium (Cr) is found in group 6 (V1A) of the periodic table along with molybdenum (Mo) and tungsten (W). These metals, therefore, have six valence electrons. In the chromium atom, the 3d-orbital is half filled with five electrons and the sixth electron is present in 3s orbital. The most common ore of chromium is chromite ($FeCr_2O_4$) which can be used directly for manufacture of steel and other chromium compounds. An alloy of chromium and nickel, nichrome is used for electrical heating elements.

Chromium (III), the most stable oxidation state of chromium, forms a number of complexes, in which six monodentate ligands surrounded the cation (octahedral structure). Large number of mononuclear, octahedral Cr (III) complex are also known with bidentate ligands, having magnetic moments close to the spin-only value of 3.87 BM. The electronic spectra of octahedral (d^3) complexes contain three absorptions due to "d ⟶ d" transitions. Selected examples of octahedral chromium (III) complexes are $[Cr(acac)_3]$, $[Cr(ox)_3]^{3-}$, $[Cr(en)_3]^{3+}$, $[Cr(biphy)_3]^{3+}$, $[Cr(CN)_6]^{3-}$.

In this experiment the trioxalato – complex containing three oxalate ions acting as bidentate ligands providing six oxygen donor atoms at the corners of octahedron (Fig.1) prepared by oxidation reduction reaction:

$$Cr_2O_7^{=} + 9C_2O_4^{=} + 14H^{+} \longrightarrow 2[Cr(C_2O_4)_3]^{3-} + 6CO_2 + 7H_2O$$

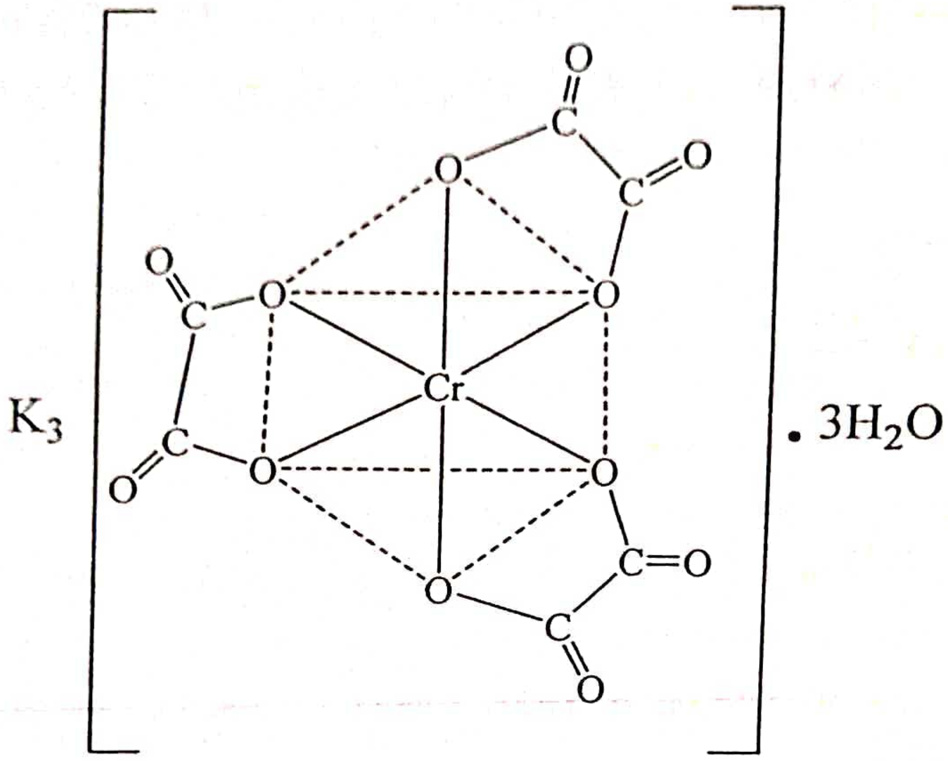


Fig.1

Procedure:

1. Dissolve (9.0 g) oxalic acid dehydrate ($H_2C_2O_4.2H_2O$) in (20 ml) warm distilled water.
2. Add, in portions, potassium dichromate ($K_2Cr_2O_7$) (3.0 g).
3. When the vigorous reaction has subsided, heat the resulting solution to boiling and dissolve in it potassium oxalate monohydrate (3.5 g).
4. Cool to room temperature, add (4 ml) ethanol and cool in an ice bath.

5. Filter off the blue-green crystals, wash with an equivolum mixture of ethanol and water (10 ml), and finally pure ethanol (3 ml).
6. Dry in air and weigh the product then calculate the % yield.
7. Prepare accurately (0.01 M) solution of the prepared dry complex in (100 ml) volumetric flask.
8. Obtain the electronic spectrum in the visible region 350-700 nm, using spectrometer.
9. Find the wavelengths λmax (for each absorption) then calculate their wavenumber (cm^{-1}) and the molar extinction coefficients of the absorption bands, using the formula:

$$A = \varepsilon C l$$

$$\text{wave number } (cm^{-1}) = \frac{107}{\lambda\text{max (nm)}}$$

ε = extinction coefficient.

C = concentration .

A = absorbance.

l = pathlength = 1.

10. Explain the observed colour of the complex in relation to its absorption spectrum.