

Lab Manual

P. Coordination Chemistry (III) 3rd Stage First Semester

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Table of Contents

Experimental Number 1:	3
Terms Related to Coordination Compounds:	3
Experimental Number 2:	6
Preparation of Tris(acetylacetonato) Iron(III): $[\text{Fe}(\text{acac})_3]$	6
Experimental Number 3:	10
Preparation of Tris(acetylacetonato) manganese(III): $[\text{Mn}(\text{acac})_3]$	10
Experimental Number 4:	13
Preparation of Bis(Acetylacetonato)Copper(II) Complex: $[\text{Cu}(\text{acac})_2]$	13
Experimental Number 5:	16
Synthesis of potassium tris(oxalato)chromate(III) trihydrate. $\text{K}_3[\text{Cr}(\text{C}_2\text{O}_4)_3] \cdot 3\text{H}_2\text{O}$	16
Experimental Number 6 and 7:	18
Synthesis cis- and trans- potassium dioxalatodiaquochromate(III) dihydrate complex	18
Cis- $\text{K}[\text{Cr}(\text{H}_2\text{O})_2(\text{C}_2\text{O}_4)_2]$ and Trans- $\text{K}[\text{Cr}(\text{H}_2\text{O})_2(\text{C}_2\text{O}_4)_2]$	18
Experimental Number 8	21
Synthesis Hexamminenickel(II) Chloride.....	21
$[\text{Ni}(\text{NH}_3)_6]\text{Cl}_2$	21
Synthesis Tris(ethylenediamine)nickel(II) chloride dihydrate $[\text{Ni}(\text{en})_3]\text{Cl}_2 \cdot 2\text{H}_2\text{O}$	21
Experimental Number 9	24
Complexometric determination of nickel in a solid sample	24

Experimental Number 1:

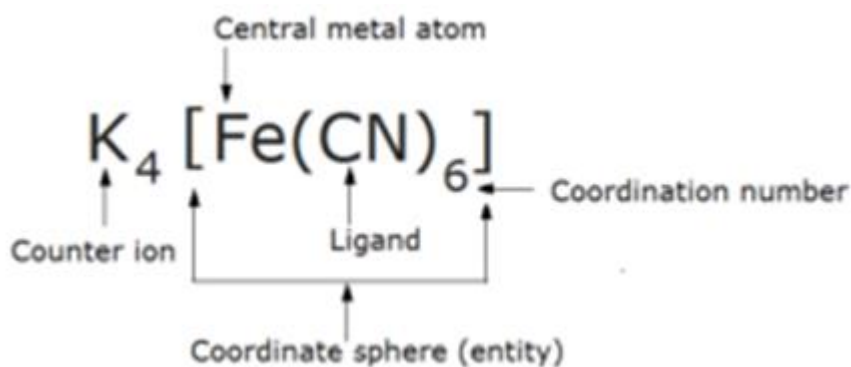
Terms Related to Coordination Compounds:

- 1. Coordination chemistry:** Coordination chemistry is the study of compounds that have a central atom (often metallic) surrounded by molecules or anions, known as ligands. The ligands are attached to the central atom by dative bonds, also known as coordinate bonds, in which both electrons in the bond are supplied by the same atom on the ligand e.g., Potassium ferrocyanide, $K_4[Fe(CN)_6]$.
- 2. Central Atom or Ion:** The atom or ion to which a fixed number of ions or groups are bound is called central atom or ion. It is also referred as Lewis acid. e.g., in $[NiCl_2(H_2O)_4]$. Ni is central metal atom. It is generally transition element or inner-transition element.
- 3. Ligands:** Ligands are electron donating species (ions or molecules) bound to the Central atom in the coordination entity. These may be charged or neutral. **Ligands are of the following types:**
 - a. Monodentate** It is a ligand, which has one donor site, i.e., the ligand bound to a metal ion through a single donor site. e.g., H_2O , NH_3 , etc.
 - b. Didentate** It is the ligand. which have two donor sites.
 - c. Polydentate** It is the ligand, which have several donor sites. e.g., $[EDTA]^{4-}$ is hexadentate ligand.
 - d. Chelating ligands** Di or polydentate ligands cause cyclisation around the metal atom which are known as

chelate , Such ligands two or more donor atoms to bind a single metal ion and are known as chelating ligands.

Ligand Type	Examples
Unidentate	$\text{H}_2\ddot{\text{O}}$: water $\ddot{\text{F}}^-$ fluoride ion $\text{:C}\equiv\text{N:}^-$ cyanide ion $[\ddot{\text{O}}-\text{H}]$ hydroxide ion :NH_3 ammonia $\ddot{\text{Cl}}^-$ chloride ion $[\text{:S}=\text{C}=\ddot{\text{N}}:]^-$ thiocyanate ion $[\ddot{\text{O}}-\text{N}=\ddot{\text{O}}:]$ nitrite ion <small>or</small>
Bidentate	$\begin{array}{c} \text{H}_2\text{C}-\text{CH}_2 \\ \quad \\ \text{H}_2\text{N} \quad \text{NH}_2 \end{array}$ ethylenediamine (en) $[\ddot{\text{O}}=\text{C}-\text{C}=\ddot{\text{O}}]^{2-}$ oxalate ion
Polydentate	$\begin{array}{c} \text{H}_2\text{C}-\text{CH}_2-\text{CH}_2-\text{CH}_2 \\ \quad \quad \\ \text{H}_2\text{N} \quad \text{NH}_2 \quad \text{NH}_2 \end{array}$ diethylenetriamine $[\ddot{\text{O}}=\text{P}(\ddot{\text{O}})-\text{O}-\text{P}(\ddot{\text{O}})-\text{O}-\text{P}(\ddot{\text{O}})=\ddot{\text{O}}]^{5-}$ triphosphate ion $[\ddot{\text{O}}=\text{C}(\ddot{\text{O}})-\text{CH}_2-\text{N}(\ddot{\text{O}})-\text{CH}_2-\text{CH}_2-\text{N}(\ddot{\text{O}})-\text{CH}_2-\text{C}(\ddot{\text{O}})=\ddot{\text{O}}]^{4-}$ ethylenediaminetetraacetate (EDTA) ion

- Coordination Number:** It is defined as the number of coordinate bonds formed by central metal atom, with the ligands e.g., in $[\text{PtCl}_6]^{2-}$, Pt has coordination number 6.
- Coordination Sphere:** The central ion and the ligands attached to it are enclosed in square bracket which is known as coordination sphere. The ionisable group written outside the bracket is known as counter ions.
- Complex ion or Coordination Entity:** It is an electrically charged species in which central metal atom or ion is surrounded by number of ions or neutral molecules.



7. **Cationic complex entity** It is the complex ion which carries positive charge. e.g., $[Pt(NH_3)_4]^{2+}$
8. **Anionic complex entity** It is the complex ion which carries negative charge. e.g., $[Fe(CN)_6]^{4-}$
9. **Oxidation Number of Central Atom:** The charge of the complex if all the ligands are removed along with the electron pairs that are shared with the central atom, is called oxidation number of central atom.

Types of Complexes:

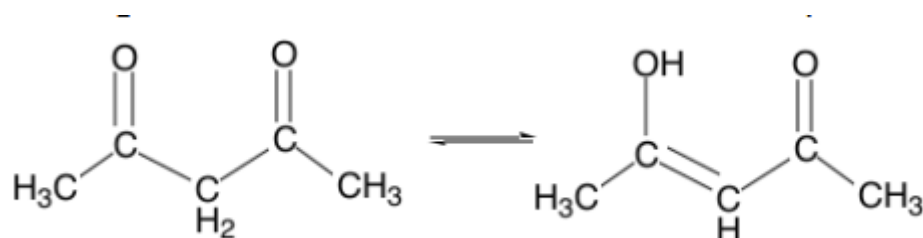
10. **Homoleptic complexes:** Complexes in which the metal atom or ion is linked to only one kind of donor atoms, are called homoleptic complexes e.g., $[Co(NH_3)_6]^{3+}$
11. **Heteroleptic complexes:** Complexes in which the metal atom or ion is linked to more than one kind of donor atoms are called heteroleptic complexes e.g., $[Co(NH_3)_4Cl_2]^+$

Experimental Number 2:

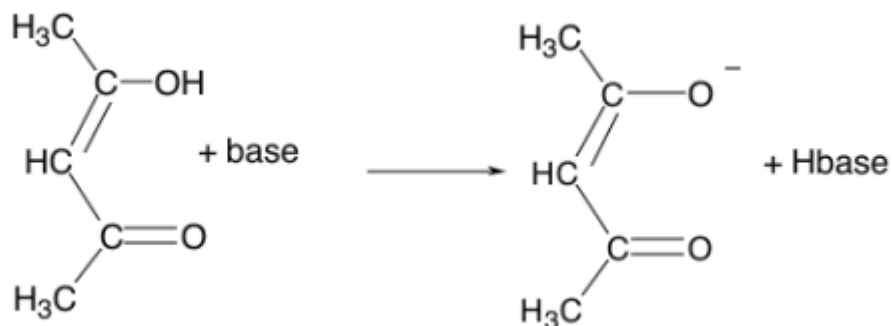
Preparation of Tris(acetylacetonato) Iron(III): [Fe(acac)₃]

The purpose of this lab was to use inorganic synthesis techniques to prepare Fe(acac)₃

In this experiment we will synthesize a metal-ligand complex using the **bidentate ligand acetylacetonone**, whose structure is shown below, with one of four metal ions (Fe, Mn, Co, Cr and Cu). The oxygens in acetylacetonone, which are double-bonded to carbons, are called ketone groups. There is another important form of this ligand, called an enol, in which a proton on the central carbon atom moves to one of the double-bonded oxygens, as shown on the right side of the figure below; although not present in significant amounts, it is the enol form of acetylacetonone that binds with metals.

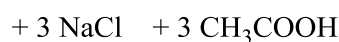
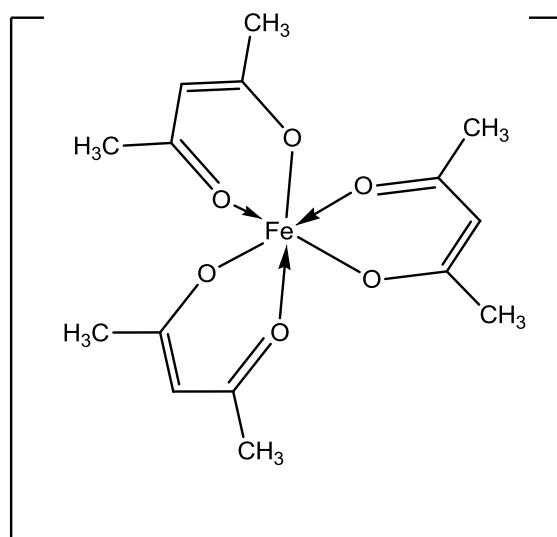
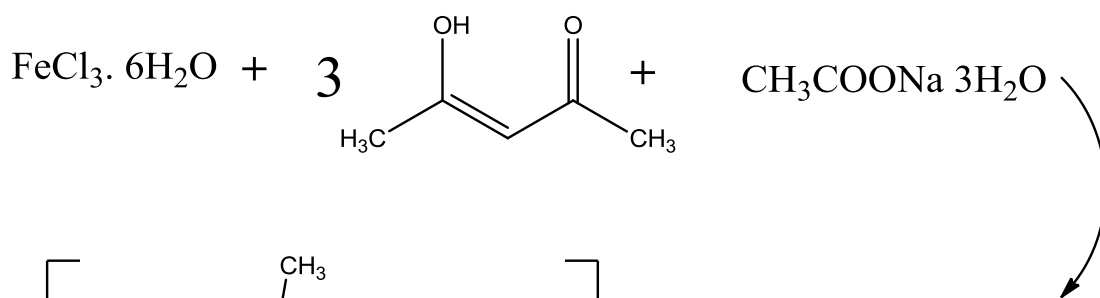


Before acetylacetonone can bind with a metal, we must first remove the hydrogen bound to the oxygen in its enol form. This is accomplished using a base, as shown below.



Chemists often adopt shorthand names for metal complexes and their ligands. The ligand acetylacetonate is abbreviated “acac⁻” when deprotonated, and “Hacac” when it is protonated. The metal–ligand complex shown to the right, therefore, is [Fe(acac)₃]

Depending on the metal ion, two or three acetylacetonate ligands are required to form a metal–ligand complex, giving the metal a coordination number of 4 or 6. In the case of Fe³⁺, for example, the metal–ligand complex, which is shown to the right, has three ligands. Because each ligand has a charge of –1, the metal–ligand complex is neutral.



Geometric shapes:

hybridization

Spin:

Magnetic propertie

Procedure:

1. Weigh 1.7 g of $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ into an Erlenmeyer flask and dissolve in 10 mL of distilled water. Add a magnetic stir bar, stir the solution, and then add a mixture of 1.9 mL of acetylacetone in 7.0 mL of methanol.
2. Add a solution containing 2 g of sodium acetate trihydrate in 10 mL of distilled water, and briefly heat the mixture on a hotplate until some of the methanol has boiled away (you should see a decrease in the volume of the reaction mixture).
3. Cool the reaction mixture to room temperature, and then place it in an ice bath for approximately 15 minutes. Filter the red crystalline solid, and wash the flask and crystals using distilled water (2 rinses of 5 mL each). Place your product in your lab drawer and allow it to dry until next week.

Questions:

1. Why the Oxygen act as a donor atom?
2. Why the sodium acetate was added to the reaction?
3. What is the hybridization and geometric Shape of $[\text{Co}(\text{acac})_3]$ complex?
4. Explain the type of spin and magnetic properties for $[\text{Co}(\text{acac})_3]$ complex?
5. What is the role of H_2O_2 to prepare $[\text{Co}(\text{acac})_3]$ when CoCO_3 is used as a starting material?
6. Calculate the percentage yield and percentage error for your product.
7. What is the preparation reaction of $[\text{Co}(\text{acac})_3]$ complex?

Experimental Number 3:

Preparation of Tris(acetylacetonato) manganese(III): [Mn(acac)₃]

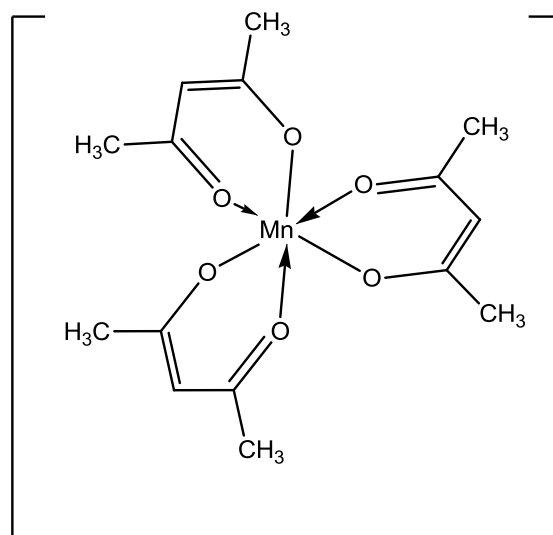
In this experiment a solution of manganese(II) chloride (MnCl₂) is oxidized with potassium permanganate in the presence of acetylacetonone giving the brown [Mn(acac)₃] solid.

The manganese complex [Mn(acac)₃] is an octahedral complex containing three bidentate ligands. Because each metal ion carries a +3 charge, and each ligand carries a -1 charge, the complex is electrically neutral. Tris(acetylacetonato)manganese(III) was prepared and used for magnetic susceptibility. The number of unpaired electrons were determined through this process to assign the geometry and to gain information on the bonding between the ligand and metal. The Tris(acetylacetonato)manganese(III) complex had four unpaired electrons. The ligand was a weak field strength thus concluding in a high spin

The three bidentate ligands are packed very efficiently around the trivalent ions of transition metal. As a result, the Mn²⁺ can be easily oxidized to form Mn(acac)₃ in the presence of acetylacetonate ions. Oxidizing agent (potassium permanganate solution) is used to oxidize the four equivalents of Mn²⁺ to become Mn³⁺ as shown in the equation below:



The formation of Mn³⁺ ions are used to react with acetylacetonate ions to produce Mn(acac)₃



Geometric shapes:

hybridization

Spin:

Magnetic properties

Procedure:

1. In a 250-mL conical flask prepare a solution of 1.32 g manganese(II) chloride tetrahydrate and 3.59 g of sodium acetate trihydrate in 50 mL water. To this solution add by pipette 5 mL of acetylacetone. Place a small magnetic stirring bar in the solution and place the flask on a magnetic stirrer in the hood.
2. To the stirred mixture add dropwise a solution of 0.28 g of potassium permanganate in 15 mL water (due to the color intensity of the permanganate solution it is difficult to determine if it has completely dissolved, therefore, stir thoroughly and check for undissolved solute).
3. After the addition of the potassium permanganate solution, stir for an additional 5 minutes. Meanwhile, prepare a solution containing 3.59 g of sodium acetate trihydrate in 15 mL of water and add this in approximately 1-mL portions to the stirred solution of crude $\text{Mn}(\text{acac})_3$.
4. Heat the reaction mixture to near boiling (hot plate) for 10 minutes and subsequently cool to room temperature. Filter the crude dark solid on a small Buchner funnel and wash with three 10-mL

portions of deionized water. Spread out the crude product on a porcelain dish and dry in an oven at 60 o C to 70 o C for at least 30 minutes

Questions:

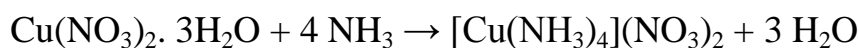
1. Why the Oxygen act as a donor atom?
2. Why the sodium acetate was added to the reaction?
3. What is the hybridization and geometric Shape of $[\text{Mn}(\text{acac})_3]$ and $[\text{Cr}(\text{acac})_3]$ complexes?
4. Explain the type of spin and magnetic properties for $[\text{Mn}(\text{acac})_3]$ and $[\text{Cr}(\text{acac})_3]$ complexes?
5. What is the role of KMnO_4 to prepare $[\text{Mn}(\text{acac})_3]$?
6. Calculate the percentage yield and percentage error for your product.
7. What is the preparation reaction of $[\text{Mn}(\text{acac})_3]$ complex?

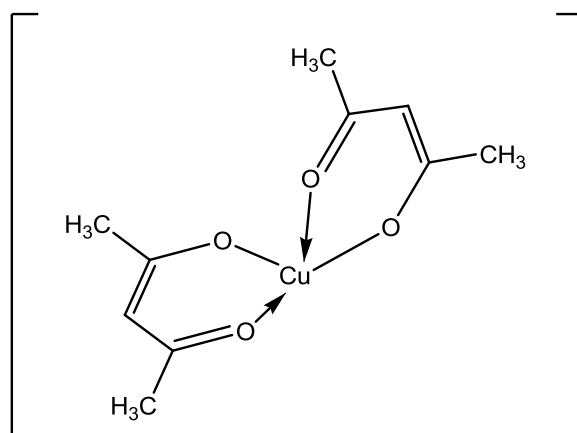
Experimental Number 4:

Preparation of Bis(Acetylacetonato)Copper(II) Complex: [Cu(acac)₂]

In this experiment, copper (II) nitrate is used as the starting material. Copper (II) nitrate is a blue crystal. When it is in hydrated form, it is very soluble in water and ethanol. Bis (acetylacetonato) copper (II) is the product formed when copper(II) nitrate reacts with ammonia and acetyl acetone. It belongs to the group of metal, acetyl acetonates. It is crystalline powder with slightly soluble in water but soluble in alcohol and blue in colour. The bis (acetylacetonato) copper (II) complex has a coordination number of 4 because there are 2 ligands bonded to the central metal ion which is the copper (II) ion.

Acetyl acetone is used in the preparation of metal acetyl acetonates for catalyst application. It able to act both as chelating agent and solvent, which it is also a competing complex. The ammonia molecule in the [Cu(NH₃)₄]²⁺ ion deprotonate the acetyl acetone as ammonia is a good leaving group. With the presence of this competing complex, the process can be done in faster rate as it speeds up the reaction.





Geometric shapes:

hybridization

Spin:

Magnetic propertie

Procedure:

1. 6g of copper (II) nitrate ($\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$) was dissolved in 60 mL of water.
2. The solution of 1:1 ammonia was slowly added with stirring until the precipitate first formed had just re-dissolved.
3. The solution was continued to stir and then 6 mL of acetyl acetone was added dropwise.
4. The crude product was filtered off by using the suction filtration.
5. Then the product was washed it well with water and drained it thoroughly. 6. The percentage yield was calculated

Questions:

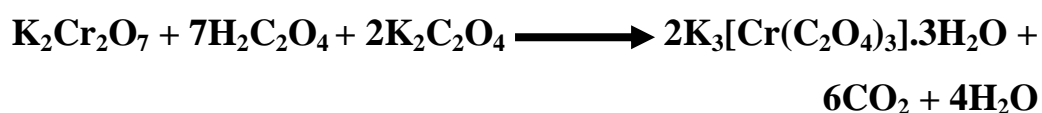
1. Why the Oxygen act as a donor atom?
2. Why the NH_3 was added to the reaction?
3. Describe the structure by mentioning the ligating atoms of the ligand and its geometry.
4. What is the hybridization of $[\text{Cu}(\text{acac})_2]$?
5. Calculate the percentage yield and percentage error for your product.

Experimental Number 5:

Synthesis of Potassium tris(oxalate)chromate(III) trihydrate.

$K_3[Cr(C_2O_4)_3].3H_2O$

Oxalic acid in the form of the di anion, behaves as a bidentate ligand with many transition metals ion. Oxalate ion is a bidentate ligand even though it contains four O atoms which have lone pairs of electrons. Oxalate(2-) is a dicarboxylic acid dianion obtained by deprotonation of both carboxy groups of oxalic acid. It has a role as a human metabolite and a plant metabolite. It is an oxalate and a dicarboxylic acid dianion. Chromium(III) , the most stable oxidation state of chromium , forms a number of complexes , in which ligands surround the cation octahedrally. There is the balance chemical equation of preparation potassium tris(oxalate)chromate(iii) trihydrate.



Procedure:

1. Make a solution of potassium oxalate (2.3g) and oxalic acid (5.5g) in water (110 ml).
2. To this solution add solid potassium dichromate (1.9g) in small portion with constant stirring.
3. Concentrate the solution nearly to dryness
4. On cooling deep green shiny crystals of potassium tris-oxalate chromium (III) of trihydrate will be formed.
5. Filter the solid crystals and leave to dry and the percentage yield.

Questions:

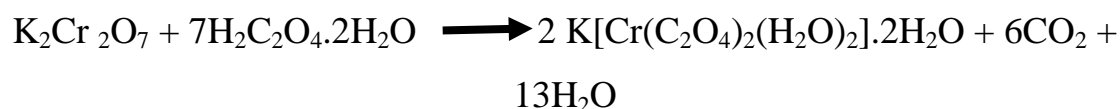
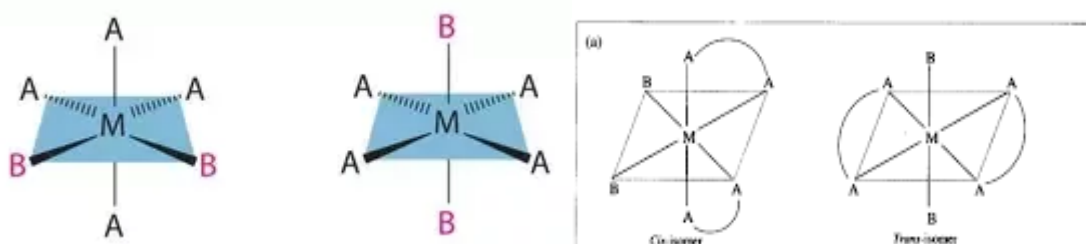
1. What are the roles of potassium oxalate and oxalic acid in this E.X.P.
2. What is the hybridization, geometric Shape, the type of spin and magnetic properties of $2K_3[Cr(C_2O_4)_3] \cdot 3H_2O$ complex?
3. What is the hybridization, geometric Shape, the type of spin and magnetic properties of $[Co(C_2O_4)_3]^{3-}$ complex ion ?
4. What is the hybridization, geometric Shape, the type of spin and magnetic properties of $K_3[Co(en)_3]Cl_3$ complex?
5. Calculate the percentage yield and percentage error for your product.

Experimental Number 6 and 7:

Synthesis cis- and trans- potassium dioxalatodiaquochromate(III) dihydrate complex

Cis- $\text{K}[\text{Cr}(\text{H}_2\text{O})_2(\text{C}_2\text{O}_4)_2]$ and Trans- $\text{K}[\text{Cr}(\text{H}_2\text{O})_2(\text{C}_2\text{O}_4)_2]$

Based on the type of geometric isomer compound or complex ions can be divided into types of cis and trans. To the complex there are two types of octahedral complexes have cis and trans forms, namely MA_4B_2 : M is an atom or ion center while A and B is a monodentate ligand. If the monodentate ligand is replaced with multidentate, for example bidentate, it will produce a complex tie ML_2B_2 . L is a bidentate ligand. Isomer structure becomes



Based on the solubility difference between the cis to trans form the two types of isomers that can be separated. Trans isomer is not soluble in

aqueous ammonia and produce light brown precipitate. Whereas the cis isomer is soluble in aqueous ammonia to produce a dark green solution which spread rapidly on filter paper.

Procedure: Cis- cis- $\text{K}[\text{Cr}(\text{H}_2\text{O})_2(\text{C}_2\text{O}_4)_2]$

1. At first, 3g of oxalic acid and 1g of $\text{K}_2\text{Cr}_2\text{O}_7$ are mixed with each other and grinded to obtain the powder form.
2. Take this mixture in the china dish and heat the content in china dish gently on a low flame by which a vigorous reaction will occur with the evolution of CO_2 and water vapour. Finally the mixture will become deep colored liquid.
3. Now without cooling the liquid, 20 mL $\text{C}_2\text{H}_5\text{OH}$ is added over this liquid and triturate the content by metallic spatula until the solid mass is formed.
4. the crystals of cis potassium dioxalatodiaquo (III) complex look black in the diffused day light which are finally weigh.

Procedure: Trans $K[Cr(H_2O)_2(C_2O_4)_2]$

1. Take 3g of oxalic acid crystal in a beaker and add small amount of water and heat to dissolve the oxalic acid crystal in water.
2. Take 1g of $K_2Cr_2O_7$ and small amount of water in a boiling tube and heat to dissolve the $K_2Cr_2O_7$ in water.
3. Now introduce the content of boiling tube in the beaker containing oxalic acid solution and cover the beaker by watch glass.
4. Now cool the dark coloured content of beaker and transfer it to a china dish. The content of china dish is kept in air for 36-48 hours by which volume is reduced to one third of the original volume.
5. filter by regular filter paper with the help of funnel which are washed by water and ethyl alcohol and finally weigh the crystals of trans isomer.

Questions:

1. Draw the structure of Cis-and Trans of $K[Cr(H_2O)_2(C_2O_4)_2]$ complexes
2. What is the role of oxalate in this e.x.p.
3. What is the different between cis and trans of $K[Cr(H_2O)_2(C_2O_4)_2]$
4. How can distinguish between cis and trans of $K[Cr(H_2O)_2(C_2O_4)_2]$
5. What is the isomer and explain type of the isomers in coordination complexes?
6. What are the different between cis and Trans procedure?
7. What is the hybridization, geometric Shape, the type of spin and magnetic properties of $[Co(C_2O_4)(H_2O)_2]^{1-}$ complex ion ?
8. Calculate the percentage yield and percentage error for your product.

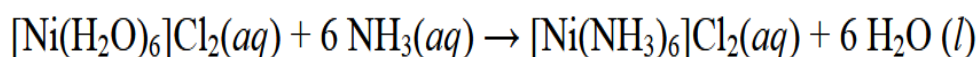
Experimental Number 8

Synthesis Hexamminenickel(II) Chloride



Synthesis Tris(ethylenediamine)nickel(II) chloride dihydrate $[\text{Ni}(\text{en})_3]\text{Cl}_2 \cdot 2\text{H}_2\text{O}$.

Part A: In aqueous solution nickel ion Ni^{2+} is surrounded by six water molecules which are actually bonded to the central metal ion. This complex is called the hexaquaonickel(II) ion. When aqueous nickel chloride is precipitated from solution, the nickel ions carry their six water molecules into the crystals and so solid nickel(II) chloride is hydrated, has the formula $[\text{Ni}(\text{H}_2\text{O})_6]\text{Cl}_2$, and is more properly called hexaquaonickel(II) chloride ammonia is added to a solution of this salt, ammonia molecules compete with the water in bonding Ni^{2+} and because the ammonia forms a stronger bond than water, the ammonia replaces the water according to:

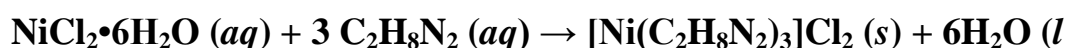


Part B: In this lab, you will combine nickel chloride hexahydrate and ethylenedi. If amine in water to produce tris(ethylenediamine)nickel chloride. When nickel chloride hexahydrate dissolves in water, it rearranges to become the hexaaquanickel cation and two chloride anions.

Adding ethylenediamine results in three ethylenediamine molecules exchanging with the six water molecules. This generally happens one

ethylenediamine molecule at a time. So, the nickel complex will pass through the light blue tetraaquaethylenediamine nickel and dark blue diaquabisethylenediamine nickel intermediates before forming the purple tris(ethylenediamine) nickel chloride product

The equation for the synthetic reaction you will carry out is:



Procedure:

The preparation of $[\text{Ni}(\text{NH}_3)_6]\text{Cl}_2$: Dissolve 3.0g of $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ in 5 mL of warm H_2O in a 125 mL Erlenmeyer flask and add 5.8mL of concentrated NH_4OH . Cool with an ice bath and observe the precipitation of large violet crystals. Add 15 mL of cold ethanol to complete the precipitation. Collect the crystals on a Büchner funnel and wash with two 5 mL portions of ethanol. Dry in air. Record the yield.

The preparation of $[\text{Ni}(\text{en})_3]\text{Cl}_2 \cdot 2\text{H}_2\text{O}$. Dissolve 6.0g of $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ in 3mL of H_2O . A little warming improves the rate of dissolution. Cool the solution in ice while adding 5.0g (5.6 mL) of ethylenediamine. Add the ethylenediamine slowly because the reaction is quite exothermic. Cool. Add 15 mL of cold ethanol to initiate crystallization. Keep cold for 10 min. The collect the product on a Büchner funnel and wash with two 5 mL portions of ethanol. Dry in air. Record the yield.

Questions

1. Calculate the actual yield, which is the mass of your crystals.
2. Calculate the theoretical yield
3. Calculate the percent yield
4. Ammonia and en are stronger ligand than water explain this statement
5. Fill the table below

Complexes	$[\text{Ni}(\text{H}_2\text{O})_6]$ Cl_2	$[\text{Ni}(\text{NH}_3)_6]$ Cl_2	$[\text{Ni}(\text{en})_3]$ Cl_2	$[\text{Ni}(\text{dmg})_2]$ Cl_2
Hybridization				
Magnetic property				
Geometric shape				
Spin				
Structure				
Type of ligand				

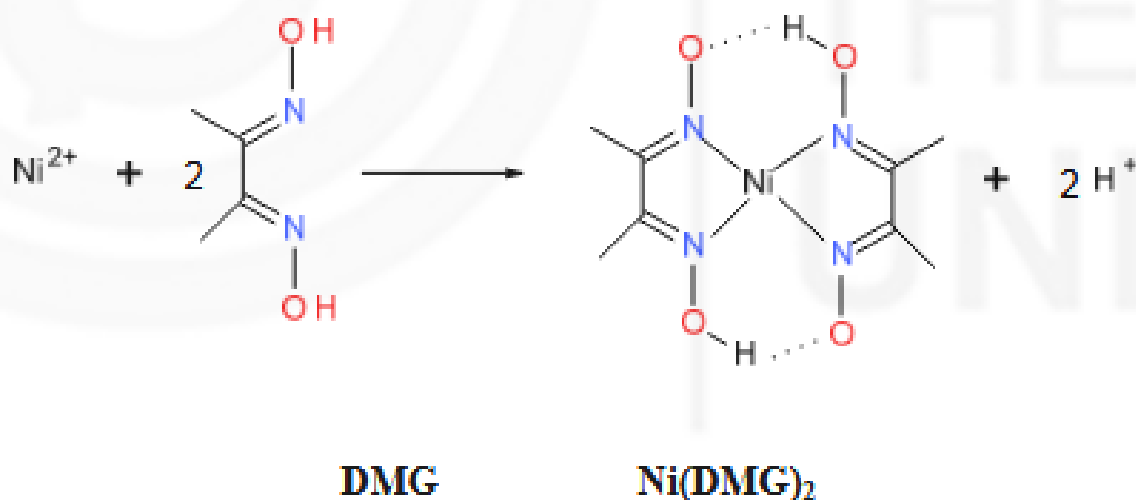
Experimental Number 9

Complexometric determination of nickel in a solid sample

Nickel(II) forms a precipitate with the organic compound dimethylglyoxime, $C_4H_6(NO)_2$. The formation of the red chelate occurs quantitatively in a solution in which the pH is buffered in the range of 5 to 9.

Although the loss of one proton occurs from one oxime group (NOH) on each of the two molecules of dimethylglyoxime, the chelation reaction occurs due to donation of the electron pairs on the four nitrogen atoms, not by electrons on the oxygen atoms. The reaction is performed in a solution buffered by either an ammonia or citrate buffer to prevent the pH of the solution from falling below 5. If the pH does become too low the equilibrium of the above reaction favors the formation of the nickel(II) ion, causing the dissolution of $Ni(DMG)_2$ back into the mother liquor.

Adding tartarate or citrate ions before the precipitation of the red nickel complex prevents interference from Cr, Fe and other metals. These anions selectively form tightly bound soluble complexes with the metals and prevent the formation of insoluble metal hydroxides in the buffered solution.



Procedure

1. Weigh (3.0) of the Nickel complexes and add (5 ml) of (6 N) HCl and dilute the mixture in to 100 mL with DW
2. Heat the solution abut 75C, and add (35 mL) of (1% DMG in alcohol) and stir well
3. Add drop wise (2M NH₄OH) solution with stirring until the precipitation occur. After that 20 drops excess of (2M NH₄OH) then heat the solution in water bath for 25 mins
4. Let the precipitate for 1h then collect it by filltartion finally wash ptt. By cool DW
5. Weight the dry product and calculate the % of Ni in sample

Sample calculations:

Gravimetric factor (G.F.) = Ni/Ni (C₈ H₁₄ O₄ N₄) = G.F

% Ni = [(wt of precipitate) (G.F.)/wt. of sample] x 100%

Question

- If you have a sample contain Ni, Fe and Cr, how can you determine the % of Ni explain the procedure and the structure of all three metals with DMG ligand