Some Experiments for B. Tech in Chemistry & Chemical Technology

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Course Name: Chemical Technology Lab II

List of probable experiments

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Expt. No. 2: Metal ion estimation: Quantitative estimation of copper (II), calcium (II) and chloride in a mixture

Expt. No.3: Co-ordination complex: Synthesis and characterization of potassium tris-oxolato chromate (III) trihydrate

Expt. No.4: Gravimetry and solution estimation: Estimation of nickel and copper in a given mixture

Expt. No.5: Colorimetry: Simultaneous determination of chromium and manganese in a solution by visible spectroscopy.

Expt. No.6: Ligand synthesis for multimetal complex: Preparation of bis-(N,N- disalicylidene ethylenediamine) μ-aquo-dicobalt

Expt. No.7: Non-metal complex: Synthesis and characterization of bis pyridine iodo nitrate

Expt. No.8: Supramolecular chemistry: Hydroquinone clathrate with sulphur-dioxide.

Expt. No.9: Acetylation of ferrocene

Expt. No.10: Unusual oxidation state: Synthesis and characterization of
a) Tris-triphenyl-phosphine copper(I) nitrate
b) Electrochemical synthesis of copper (I) dicyclopentadiene perchlorate

Expt. No.11: Soap: Preparation of soap and study of its properties.

Expt. No.12: Fertilizer: Preparation of a fertilizer and its estimation of anion

Expt. No.13: Solder as example of alloy: Preparation of alloy of lead and tin and understand alloy formation by DSC and X-ray diffraction and solder a copper wire with the prepared alloy.

Expt. No.14: Nano-chemistry: Preparation of manganese dioxide nano-particles or alumina-particles.

Expt. No.15: Industrially important compounds:
a) Disinfectant: synthesis of alum
b) High temperature superconductor: synthesis and characterization of $YBa_2Cu_3O_7$

Expt. No.16: Photosynthesis:

a) Oxidation of iso-propanol by copper(II) acetylacetonate in the presence of benzophenone by light

b) Extraction of chlorophyll from plant leaves

Expt. No17: Magnetic material: Measure the magnetic susceptibility of Mn(II), Iron(II), cobalt(II), copper(II) and zinc(II) and correlate their magnetic moment to number of unpaired electrons.

Expt. No.18: Photocatalysis / Fuel cell: Splitting of water by titanium dioxide.

Experiment No.1

Standardization of potassium permanganate by oxalic acid:

Commercially available potassium permanganate generally contains impurity. Thus it cannot be used as a primary standard. In order to make standard potassium permanganate solution it requires standardized by a primary standard.

Equivalent weight of $KMnO_4 = [\ 2\ KMnO_4 / 10\ ] = 31.61$, which is derived from the equation

$$2\ KMnO_4 = K_2O + 2\ MnO, \text{ i.e. } Mn^{+7} + 5\ e \rightarrow Mn^{+2}$$

Preparation of potassium permanganate (0.1 M):

Weigh potassium permanganate (3-3.2 g) with an ordinary balance. Take the weighed potassium permanganate in a beaker (250 ml). Add distilled water (100 ml); cover the beaker with a watch glass, heat the solution to boiling, and allow it to cool to the room temperature. Filter the solution through a funnel containing glass wool. The filtered solution should be stored in a stopper clean, glass bottle. The solution should be stored in the dark except when in use.

Standardization:

1. Rinse a clean burette (50 ml) thrice with 5 ml portions of the potassium permanganate solution. Fill up the burette with potassium permanganate solution up to the zero mark, note the upper meniscus. Examine that the jet of the burette is completely filled with the solution and no air bubble is left behind.

2. Prepare a solution of sulphuric acid (1:2): Add conc. sulphuric acid (10 ml) to water (10 ml) with stirring and cooling if necessary.

Precautions: Heat is evolved during mixing so care is to be taken. (Discuss with laboratory supervisor)

3. Pipette out standard oxalic acid (0.1 M, 10 ml) into a conical flask (250 ml). Add sulphuric acid (1: 2 in water, 10-15 ml) and then add boiling water to dilute it to about 100 ml. Now titrate the solution with potassium permanganate solution. At first add potassium permanganate solution small quantities at a time with stirring; the pink color of potassium permanganate will take some time to discharge its colour at the beginning. So initial addition should be very slow. When some potassium permanganate solution has been added, the pink color will be discharged quickly. Now add potassium permanganate solution more quickly with stirring. Near the end point when the rate of disappearance of the pink color slows down, add potassium permanganate solution drop wise with stirring until with one drop makes the whole solution pink. (The pink color persists for 30 seconds, after which the color may be discharged again).

4. Note the volume of potassium permanganate solution added. Repeat the operation thrice.

Calculations:

Let volume of $KMnO_4$ solution = V_1 ml, Strength of oxalic acid = S_1 (N)

Therefore, strength of $KMnO_4$ solution = $(10 \times S_1) / V_1$ (N)

Experiment No.2

Quantitative estimation of copper (II), calcium (II) and chloride from a mixture:

In this experiment the chloride ion is separated by precipitation with silver nitrate and estimated. Whereas copper (II) is estimated by iodometric titration and calcium by complexometric titration

Iodometric estimation of copper (II):

Copper solution oxidizes potassium iodide and liberates iodine; liberated iodine is estimated by sodium thiosulphate. The reactions involved are:

$2\,Cu^{2+} + 4I^{-} \Rightarrow 2\,Cu^{+} + 2I_2$ and $2I_2 + 2S_2O_3^{2-} \Rightarrow 2I^{-} + S_4O_6^{2-}$

Complexometric estimation of calcium (II):

During the complexometric titration of calcium (II), copper (II) ion can interfere if eriochrome black-T indicator is used. So copper (II) is reduced to copper (I) by using hydrazine hydrate and precipitated as copper thiocyanate by using ammonium thiocyanate. A calcium (II) ion is then titrated with ethylene diamine tertraceetic acid (EDTA).

$Ca^{2+} + H_2Y^{2-} \Rightarrow CaY^{2-} + 2H^{+}$, where YH_4 is EDTA

No sharp end point can be obtained for calcium (II) with eriochrome black-T indicator, so small amount of $Mg\text{-}YH_2$ should be added to get a sharp end point.

Gravimetric estimation of chloride:

Chloride can be estimated gravimetrically by precipitating it as silver chloride. An aqueous solution of chloride is generally acidified with dilute nitric acid to prevent precipitation of other silver salts.

$$AgNO_3 + Cl^{-} \Rightarrow AgCl \downarrow$$

Requirements:

1. Stock solution of Cu^{2+}, Ca^{2+} and Cl^{-} ions.
2. Sodium thiosulphate
3. Starch solution
4. Potassium iodide
5. Erichrome Black-T
7. Ammonium thiocyanate
8. Ethylene diamine tetra acetic acid
9. Hydrazine hydrate
10. Ammonia buffer
11. Silver nitrate

6. Dilute nitric acid

Procedure:

Estimation of copper (II):

Take stock solution (25 ml) in a conical flask (250 ml) and add potassium iodide (1g) to it. Iodine is liberated in the solution. Titrate the liberated iodine with standard sodium thiosulphate solution (0.1 M).

Estimation of calcium (II):

Take stock solution (25 ml) in a conical flask (250 ml). Add hydrazine hydrate (1 drop), a colourless solution will be observed. To this add a solution of ammonium thiocyanate or potassium thiocyanate (10%, 5 ml) and filter the white precipitate. Take the filtrate in a conical flask and add ammonia-ammonium chloride buffer solution (2 ml, pH =10) and add Mg-EDTA (1 ml) to the filtrate. Titrate the solution with standard EDTA (0.01 M solution) using eriochrome black-T indicator. Calculate the amount of calcium in wt / lit and also express it as ppm.

Estimation of chloride:

Take stock solution (25 ml) in a conical flask. Acidify the solution with dilute nitric acid (1M, 10 ml), and then add silver nitrate solution (0.2g / ml water). This will give a white precipitate of silver chloride. Filter the precipitate in a preweighed crucible. Place the crucible along with the precipitate in an oven (~ 50^0C) for 1hr after that cool the crucible to room temperature and weigh. Repeat the process of heating and weighing till constant weigh is obtained. Find out the weigh of silver chloride formed and find out the amount of silver.

Experiment No.3

Synthesis and characterization of potassium *tris* oxalato chromate (III) trihydrate:

The experiment is based on the following equation:

$$K_2Cr_2O_7 + 7H_2C_2O_4 + K_2C_2O_4 \xrightarrow{H_2O} K_3[Cr(C_2O_4)_3] + 6CO_2 + H_2O$$

Requirements:

1. Potassium dichromate	6. Sodium hydroxide	11. Sulphuric acid
2. Potassium oxalate monohydrate	7. Potassium permanganate	12.Silver nitrate

4. Oxalic acid dihydrate
5. Potassium iodide
8. Oxalic acid
10. Starch solution
13. Ammonoium persulphate
14. Sodium thiosulphate

Procedure:

Make a solution of potassium oxalate monohydrate (2.3 g, 12.5 mmol) and oxalic acid dihydrate (5.5 g, 43.6 mmol) in water (110-120 ml). To this solution add solid potassium dichromate (1.9 g, 6.45 mmol) in small portions with constant stirring. Concentrate the solution nearly to dryness. On cooling deep green shiny crystals of potassium *tris*-oxalate chromium (III) trihydrate will be formed. Filter the solid and dry by pressing between filter paper.

Tasks:

1. Calculate percent yield
2. Estimate chromium
3. Estimate oxalate
4. Find out the molar conductance in water
5. Record the visible spectra and interpret
6. Record IR spectra and interpret

Estimation of chromium:

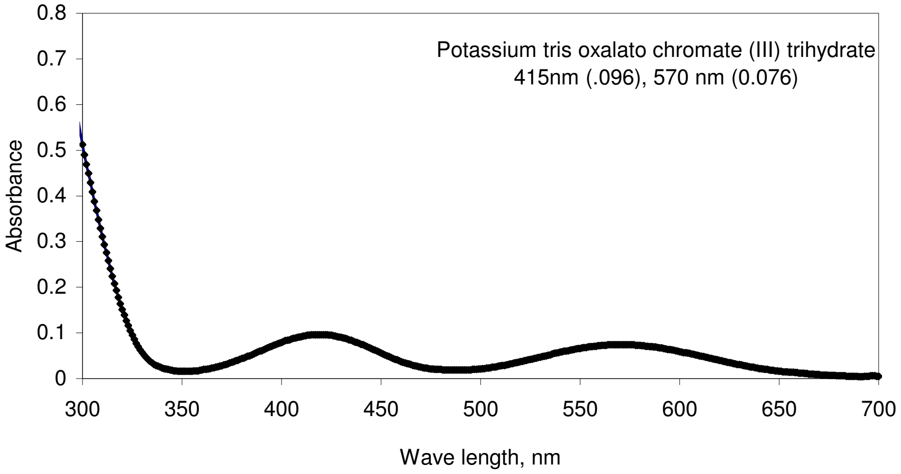
Dissolve an accurately weighed sample (in the range 0.10 – 0.15 g) of the complex in distilled water (100 ml). To this add sulphuric acid (5 N, 5 ml), ammonium persulfate (~1 g) and silver nitrate solution (0.1 M, 0.1 ml). Heat the solution under boiling condition for half an hour and the color of the solution is orange yellow at this stage. Cool the solution to room temperature and estimate the chromium iodometrically.

(***A duplicate run should be performed simultaneously)***

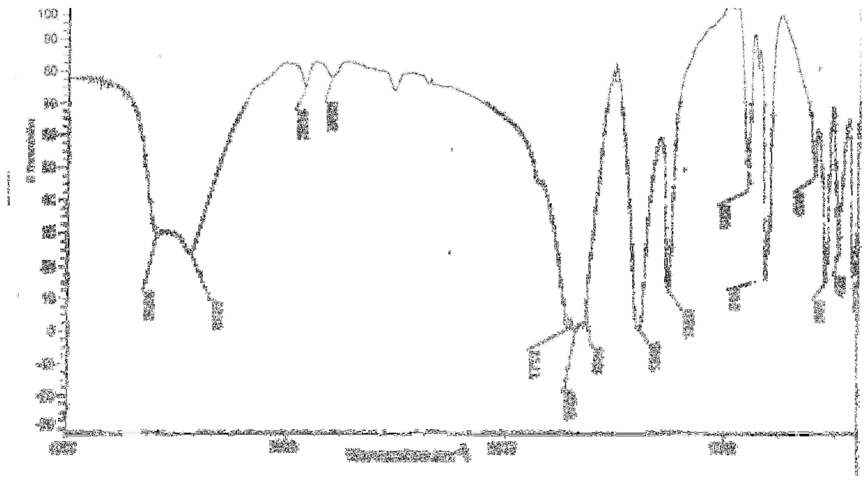
Estimation of oxalate:

Dissolve an accurately weighed sample (in the range 0.10 – 0.15 g) of the complex in 50-70 ml of distilled water. Warm the solution after adding sulphuric acid (5 N, 3 ml) and then carefully add dilute sodium hydroxide solution (in the initial stage use a concentrated solution and at the near neutral point use a dilute solution) until no more precipitation of the hydrated chromium oxide is observed (excess use of alkali should be avoided). Digest the solution hot, filter, and wash thoroughly with enough water. Combine the filtrate and washings, acidify with sulphuric acid (5 N, 15 ml), warm to 70°C and then titrate with standard potassium permanganate solution. ***(Two sets of experiments should be conducted simultaneously).***

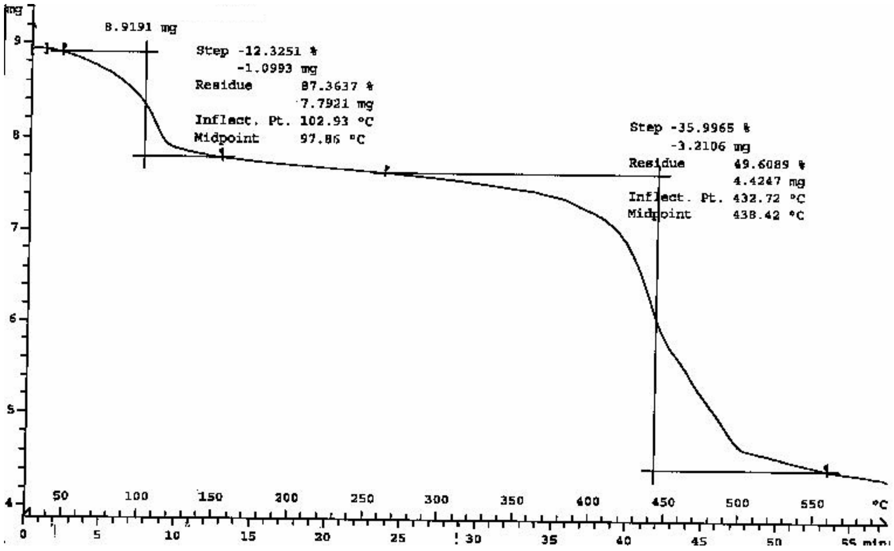
UV-Visible spectra (MeOH) of potassium *tris* oxalato chromate (III) trihydrate



FT-IR spectra (KBr, cm^{-1}) of potassium *tris* oxalato chromate (III) trihydrate



Thermogram (10^0C / min) of potassium tris oxalato chromate (III) trihydrate



Experiment No.4

Estimation of nickel and copper in a given mixture:

Principle:

In presence of nickel, copper can be estimated volumetrically (iodometrically). For estimation of nickel, the copper is to be first separated from the mixture. The separated copper can be either gravimetrically or iodometically estimated. The nickel is precipitated as nickel dimethylglyoximate and estimated gravimetrically.

Requirements:

1. Sodium Carbonate
2. Sodium thiosulphate
3. Acetic acid
4. Potassium iodide
5. Starch
6. Ammonium thiocyanate
7. Sulphurous acid
8. Methyl red
9. Nitric acid
10. Ammonia solution
11. Dimethyl glyoxime
12. Hydrochloric acid

Procedure:

(a) Volumetric estimation of copper: Pipette out the supplied solution (20 ml) into a conical flask. Now add sodium carbonate solution dropwise till slight turbidity appears. Dissolve the turbidity drop wise in dilute acetic acid so as to get a clear light blue solution. To this flask add solid potassium iodide (1 g). Keep the flask below the nozzle of the burette and run down sodium thiosulphate solution into it till a light yellow colour is obtained. Then add starch solution (2 ml) and again add more quantity of sodium thiosulphate solution till the blue colour of the solution just disappears. At this stage add ammonium thiocyanate solution (2 ml, 10%) and continue addition of sodium thiosulphate solution till the blue colour disappears. Repeat the titration to get a set of concordant readings.

Or, Gravimetric estimation of copper:

Measure out the supplied solution (25 ml) in a beaker (250 ml). To this add hydrochloric acid (1-2 ml) and a freshly prepared saturated solution of sulphurous acid (15-20 ml) (smell of sulphur dioxide in the beaker indicates the presence of sulphurous acid). Dilute the solution by adding 50 ml of distilled water. Heat the solution to boiling. Discontinue heating and to the solution add ammonium thiocyanate solution (10%, 25 ml) slowly with constant stirring. Digest the precipitate by keeping the beaker on a water bath for about 30 minutes and test for complete precipitation with one or two drops of ammonium thiocyanate solution (10%)

Filter the solution through a weighed sintered G-4 crucible. Wash the precipitate with ammonium thiocyanate solution (1%). Reserve the filtrate and washings for the estimation of nickel. Dry the precipitate by keeping the crucible in an oven at 100-105°C. Weight the crucible accurately and do the calculation.

(c) Gravimetric estimation of nickel:

Transfer the filtrate and washings obtained from the estimation of copper to a beaker. Evaporate the solution to a 40-50 ml. To this add conc nitric acid (15 ml) and conc HCl (8 ml) and evaporate the solution nearly to dryness (2 ml). Wash down the sides of the beaker with distilled water and evaporate the solution to complete dryness in order to remove excess nitric acid by heating the beaker on a water bath. Dissolve the residue in water and add conc. hydrochloric acid (1 ml) if the solution is not clear. Dilute the solution in the beaker to 75 ml with distilled water and add methyl red (2 drops). Then add dimethylglyoxime (15 ml of 1%) with constant stirring. Now continue adding 1:1 ammonia solution drop wise till the solution becomes alkaline, i.e. the colour of the solution changes from red to yellow. Digest the ppt on a steam bath and test the complete precipitation by adding few drops of dimethylglyoxime to the clear supernatant liquid. After this cool the solution for one hour and filter through a weighed G-3 crucible. Wash the precipitate many times with hot water until the washings are free from chloride and sulphate ions.

Now dry the precipitate at 110-120°C by keeping the crucible in an oven. Cool it in a dessicator and weigh the crucible. Now do the calculations.

Calculations

(a) For copper

Suppose the copper present in (let 250 ml) of the solution X gm

So, weight of copper present in 1000 ml of solution =4 X

Suppose 20 ml of $CuSO_4$ solution = V ml of 0.05 M $Na_2S_2O_3$

$$20 \text{x}\ M_{Cu}{}^{2+} = V \text{ x } 0.05\ M$$

$$M_{Cu}{}^{2+} = V \text{ x } 0.05/20\ M$$

Therefore the strength of Cu^{2+} ions in the solution =V x 0.05/20 x 63.5 g

(b) For nickel

Let weight of ppt = X g

We know Ni $(C_4H_7O_2H_2)_2 \equiv$ Ni

288.91 58.69

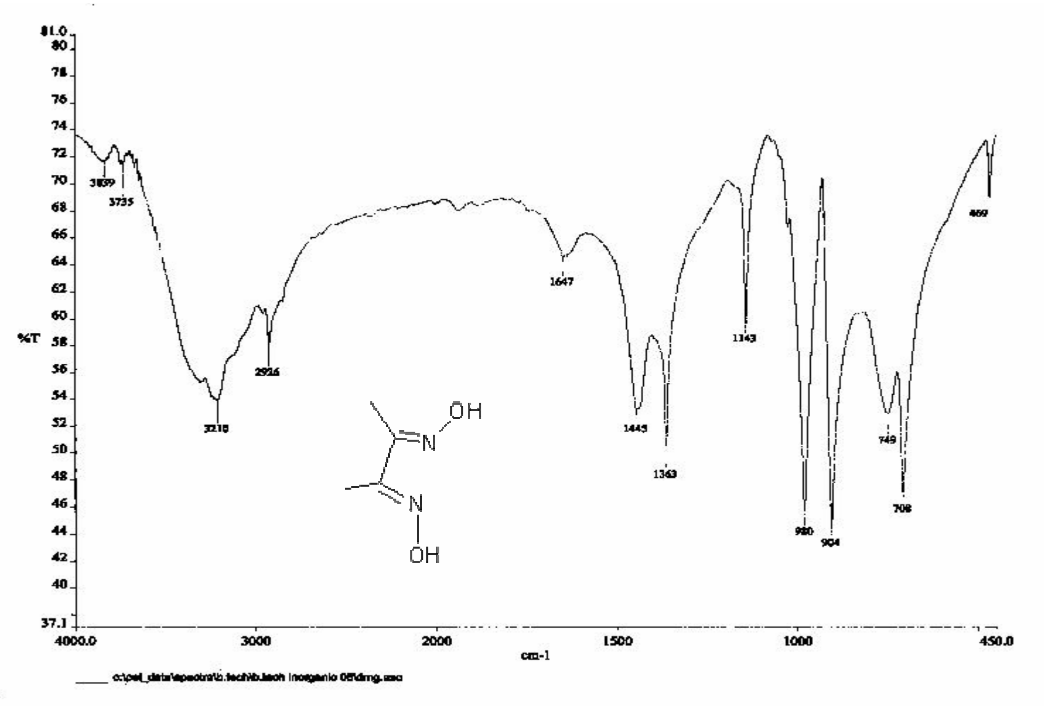
288.91 g nickeldimethylglyoximate contains = 58.69 g of nickel

So, X g of nickeldimethylglyoximate contains = 58.69/288.91 x X

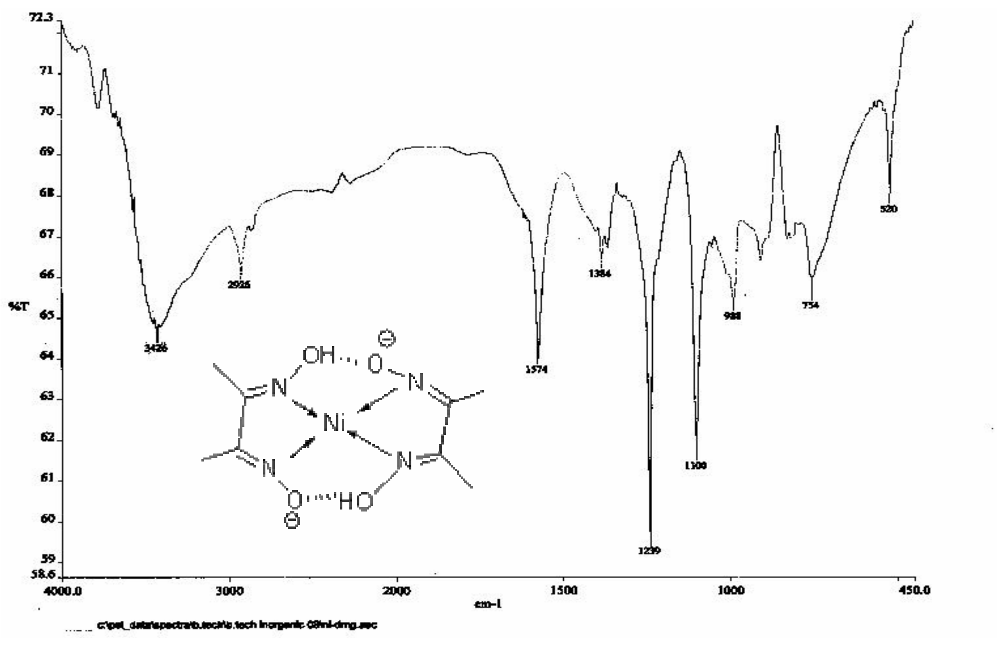
Or, 25 ml of the solution contains = 58.69/288.91 x X g of nickel

1000 ml of the given solution contains = 58.69 / 288.91 x X x 1000/25 g

FT-IR spectra (KBr, cm^{-1}) of dimethylglyoxime



FT-IR spectra (KBr, cm^{-1}) of nickel dimethylglyoxime



Experiment No.5

Simultaneous determination of chromium and manganese in a solution by visible spectroscopy:

This experiment is concerned with the simultaneous spectro-photometric determination of two solutes in a solution. The absorbance's are additive, provided there is no reaction between the two solutes. Hence we may write

$A\,\lambda_1 = \lambda_1\,A_1 + \lambda_1\,A_2$ (1) and $A\,\lambda_2 = \lambda_2\,A_1 + \lambda_2\,A_2$ (2)

Where A_1 and A_2 are the measured absorbances at the two wavelengths λ_1 and λ_2; the subscripts 1and 2 refer to the two different substances, and the subscripts λ_1 and λ_2 refer to the different wavelengths. The wavelengths are selected to coincide with the absorption maxima of the two solutes; the absorption spectra of the two solutes should not overlap appreciably, so that substance 1 absorbs strongly at wavelength λ_1 and weakly at wavelength λ_2, and substance 2 absorbs strongly at λ_2 and weakly at λ_1. Now $A = \varepsilon\,c\,l$, where ε is the molar absorption coefficient at any particular wavelength, c is the concentration (mol L^{-1}) and l is the thickness, or length, of the absorbing solution (cm). If we set l = 1 cm then

$A\,\lambda_1 = \lambda_1\,\varepsilon_1 c_1 + \lambda_1\,\varepsilon_2 c_2$ (3) and $A\,\lambda_2 = \lambda_2\,\varepsilon_1 c_1 + \lambda_2\,\varepsilon_2 c_2$ (4)

Solutions of the these simultaneous equations gives

$$c_1 = (\lambda_2\varepsilon_2\,A\,\lambda_1 - \lambda_1\varepsilon_2\,A\,\lambda_2) / (\lambda_1\varepsilon_1\,\lambda_2\varepsilon_2 - \lambda_1\varepsilon_2\,\lambda_2\varepsilon_1) \quad (5)$$

$$c_2 = (\lambda_1\varepsilon_1\,A\,\lambda_2 - \lambda_2\varepsilon_1\,A\,\lambda_1) / (\lambda_1\varepsilon_1\,\lambda_2\varepsilon_2 - \lambda_1\varepsilon_2\,\lambda_2\varepsilon_1) \quad (6)$$

The values of molar absorption coefficients ε_1 and ε_2 can be deduced from measurements of the absorbances of pure solutions of substances 1and 2. By measuring absorbance of the mixture at wavelengths λ_1 & λ_2 , the concentrations of the two components can be calculated.

Requirements:

1. 0.001 M, 0.0005 M and 0.00025 M solutions of potassium dichromate (in 1 M sulphuric acid and 0.7 M phosphoric acid)
2. 0.001 M, 0.0005 M and 0.00025M solutions of potassium permanganate (in 1 M sulphuric acid and 0.7 M phosphoric acid)

Procedure:

1. Prepare a series of solutions: Potassium dichromate 0.001 M, 0.0005 M and 0.00025 M in mixed solvent of sulphuric acid (1 M) and phosphoric acid (0.7 M).

2. Prepare a series of solutions: Potassium permanganate 0.001M, 0.0005 M and 0.00025 M in sulphuric acid (1M) and phosphoric acid (0.7 M).

3. Measure the absorbance A for each of the three solutions of potassium dichromate and also each of the three solutions of potassium permanganate at both 440 nm and 545 nm by taking 1ml solution each. Calculate ε in each case by A= εcl and record the mean values for dichromate (2) and permanganate (1) at the two wavelengths.

4. Mix potassium dichromate (0.001 M) and potassium permanganate (0.0005 M) in the following amounts shown in table 1 in 100 ml beakers. In each case total volume of solution should be 50 ml. To each of these solutions add 1.0 ml of concentrated sulphuric acid, (a set of typical results included in table I for guidance). Measure the absorbance of each of the mixtures at 440 nm. Calculate the absorbance of the mixtures from

$$A_{440} = 440\ \varepsilon_{Cr}\, c_{Cr} + 440\ \varepsilon_{Mn}\, c_{Mn}$$

$$A_{545} = 545\ \varepsilon_{Cr}\, c_{Cr} + 545\ \varepsilon_{Mn}\, c_{Mn}$$

Preparation of mixture of solutions:

$K_2Cr_2O_7$ solution (mL)	$KMnO_4$ solution (mL)	Absorbance (observed)	Absorbance (calculated)
50	0		
45	5		
40	10		
35	15		
25	25		
15	35		
5	45		
0	50		

4. Record the absorbance of the unknown solution (supplied) at 545nm and 440 nm. Calculate the concentrations of permanganate and dichromate in this solution.

Experiment No.6

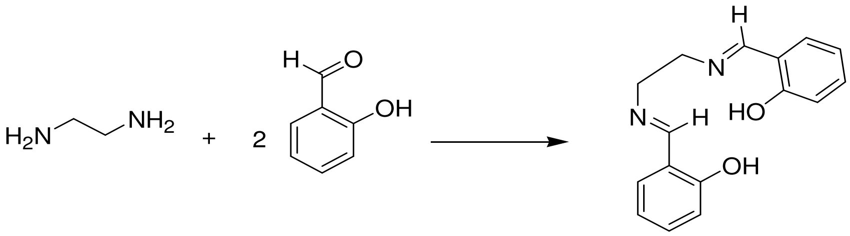
Preparation of bis(N, N' disalicylalethylene-diamine)-μ- aquadicobalt (II):

Requirements:

1. Salicylaldehydrate
2. Cobalt (II) chloride hexahydrate
3. Ethanol
4. 1-nitroso-2 naphthol
5. Ethylene diamine
6. Acetic acid
7. Methanol
8. Hydrochloric acid
9. Hydrogen peroxide
10. Sodium acetate trihydrate
11. Sodium hydroxide
12. Bis-(N, N' disalicylidine ethylene diamine)

Preparation of bis-(N,N-disalicylidene ethylenediamine):

The preparation of bis-(N,N-disalicylidene ethylenediamine) is based on the following equation:



Procedure:

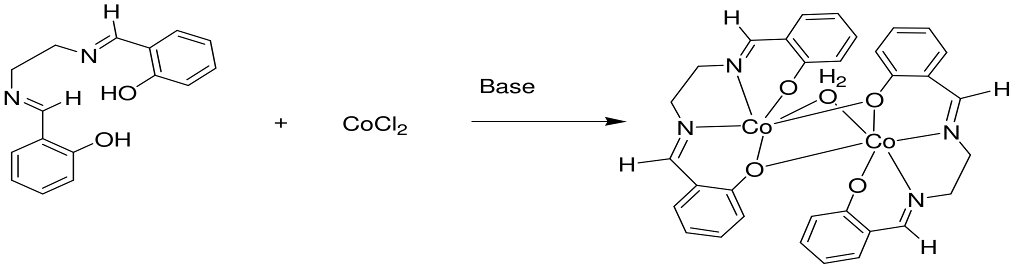
Dissolve salicylaldehyde (3.05 g, 0.025 mol) in ethanol (25 ml) in a round bottom flask. Heat the solution to ~70°C and add a solution of ethylenediamine (0.75 g, 0.0125 mol) dissolved in ethanol (3 ml). Heat the resulting yellow solution under reflux for 30 mins. Reduce the volume of the solution to half by evaporating the solvent and after cooling, filter the yellow crystalline precipitate under suction and wash with ice-cold ethanol. Recrystallize the product from methanol and dry at room temperature under vacuum. Determine the melting point and calculate the percent yield.

Tasks:

1. Calculate the percent yield
2. Determine the melting point
3. Record and interpret the IR spectra
4. Record and interpret the nmr spectra

Preparation of bis(N, N' disalicylalethylene-diamine)-μ- aquadicobalt(II):

The preparation of bis (N, N' disalicylalethylene-diamine)-μ- aquadicobalt (II) is based on the following equation:



Procedure:

Dissolve finely ground N,N'-disalicylidene ethylenediamine (1.34 g) in water (150 ml) and to this solution add sodium hydroxide (3.9 g) and sodium acetate trihydrate (5 g). Continue stirring for 10-15 minutes. While stirring is continued add a solution of cobalt (II) chloride hexahydrate (1.23 g) dissolved in hot water (25 ml). Stir the reaction mixture until it turns to a reddish-brown paste. Allow the reaction mixture to stand for at least 15 minutes. Centrifuge the reaction mixture until most of the mother liquor has been removed and a hard cake remains. Wash the precipitate three times with water (each time with about 10 ml of water). Remove the cake from the centrifuge tube and mix thoroughly with water (75 ml) so that no large particles remain and uniform slurry is obtained. The cake is further centrifuged as dry as possible. It is then broken into small pieces and dried at 100°C under reduced pressure.

Tasks:

1. Calculate the percent yield.
2. Record the IR spectra.
3. Measure the molar conductivity

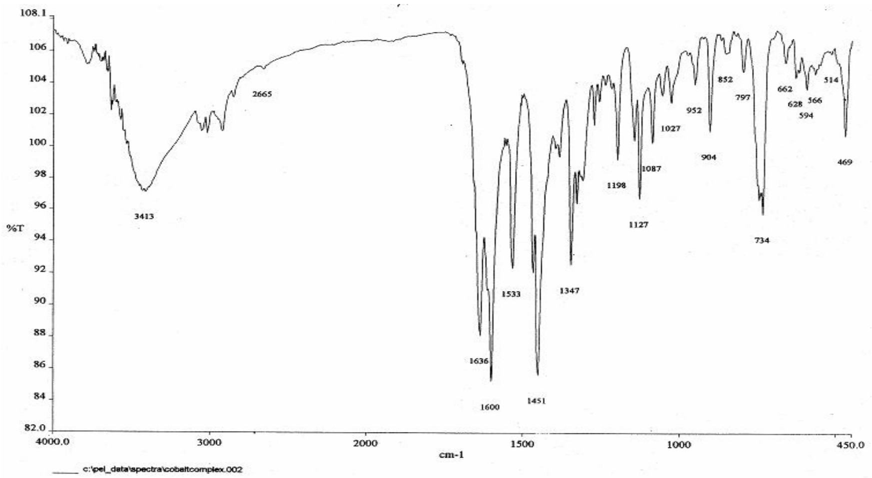
Estimation of cobalt:

Take an accurately weighed amount (range 0.1 to 0.2 g) of the cobalt (II) complex in minimum amount of hot dilute hydrochloric acid (1:1). Treat the solution with sodium hydroxide (5%) in order to get cobalt (II) hydroxide. Use of excess alkali is to be avoided. Centrifuge the precipitate and wash carefully with distilled water. Filter the precipitate and oxidize it to cobalt (III) by treating with a small amount of hydrogen peroxide (1 ml, 30% v/v) solution. Remove the excess peroxide by boiling for nearly 30 min. Dissolve the precipitate in warm acetic acid and dilute it to 200 ml with water. Add 1-nitroso-2-naphthol reagent drop wise to the warm solution with stirring. A red-brown precipitate of $Co(C_{10}H_6O_2N)_3$ will appear. Filter the precipitate through a pre-weighed sintered glass crucible and dry in an oven at ~110°C and weigh. (Repeat the weighing, until the value is constant). Find out the percentage of cobalt.

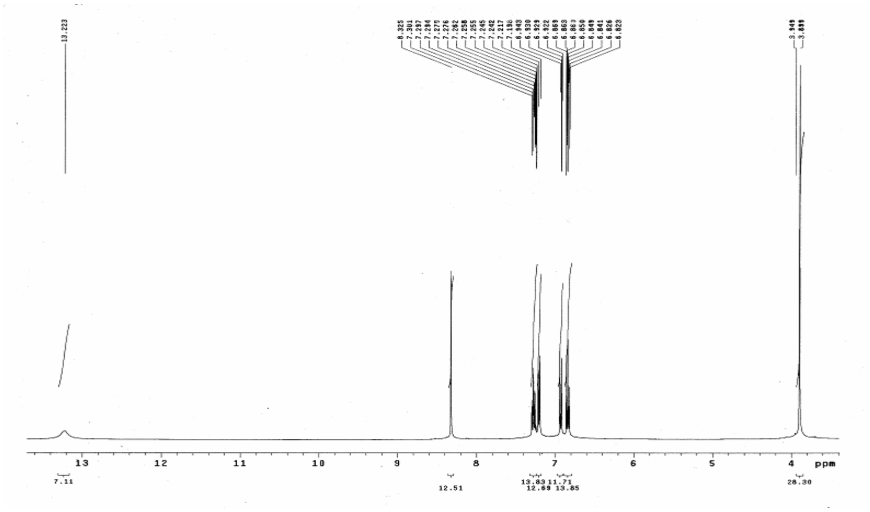
Preparation of the reagent

Dissolve 1-nitroso-2-napthol in glacial acetic acid (4g / 100 ml) and then add hot distilled water (100 ml). The cold filtered solution should be used immediately.

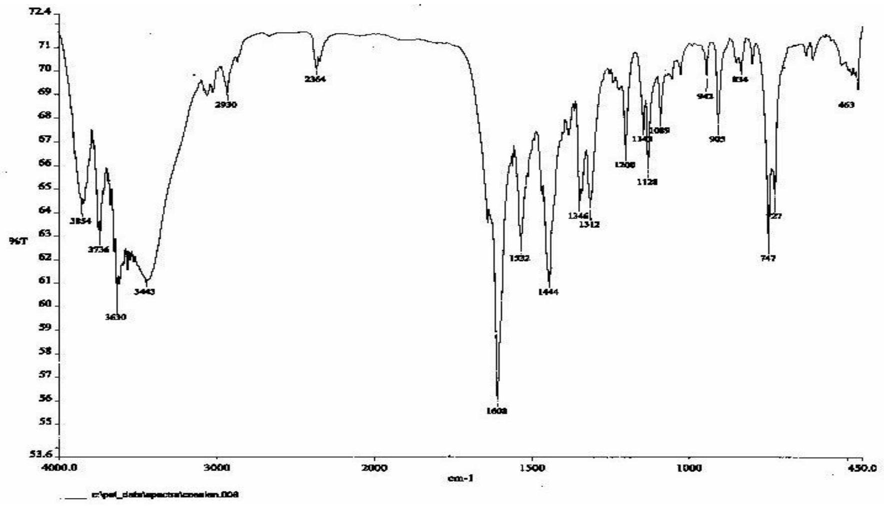
FT-IR spectra (KBr, cm^{-1}) of bis-(N,N-disalicylidene ethylenediamine)



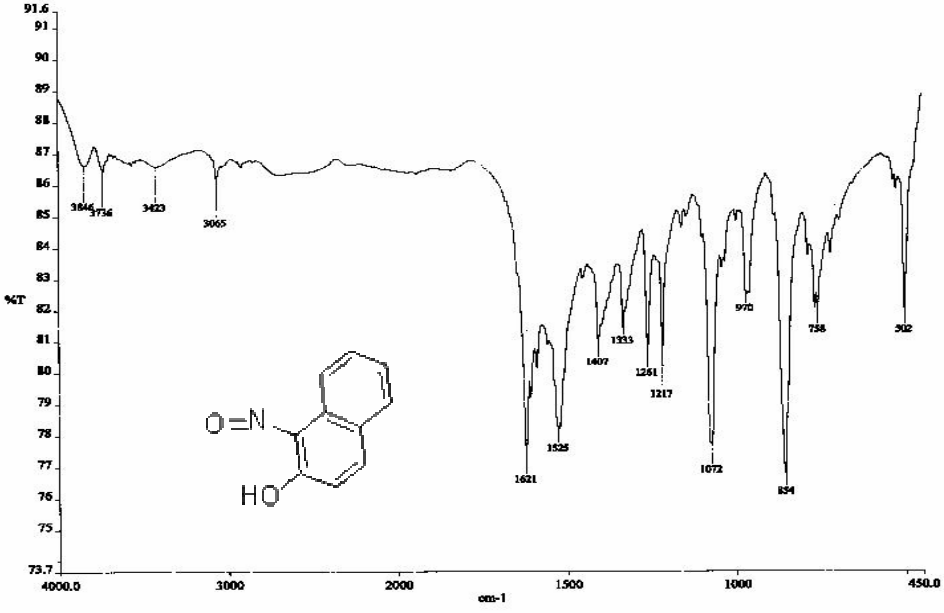
^{1}HNMR spectra (400 MHz, $CDCl_3$, ppm) of bis-(N,N-disalicylidene ethylenediamine)



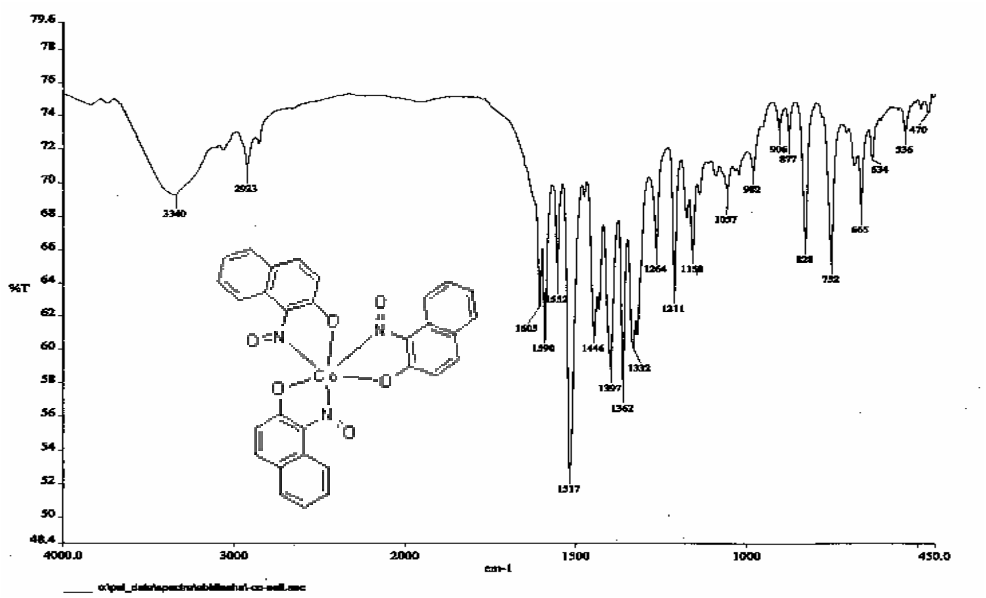
FT-IR spectra (KBr, cm^{-1}) of bis (N, N' disalicylalethylene-diamine)-μ- aquadicobalt (II)



FT-IR spectra (KBr, cm^{-1}) of 1-nitroso-2-napthol



FT-IR spectra (KBr, cm^{-1}) of tris- 1- nitroso- 2- naphthalato cobalt (III)



Experiment No.7

Synthesis and characterization of *bis*-pyridine iodo nitrate:

The experiment is based on the following reaction:

$$AgNO_3 + py + I_2 \longrightarrow I(py)_2(NO_3) + AgI$$

where py = pyridine

Requirements:

1. Silver nitrate	2. Pyridine	3. Iodine
2. Chloroform	5. Diethylether	

Procedure:

Dissolve silver nitrate (1.7 g) in pyridine (5 ml) and add iodine (2.5 g) in chloroform (50 ml). The solution is allowed to stand for half an hour to get a yellow precipitate. Filtered off the precipitate and wash it with diethylether (25 ml). Collect the filtrate and evaporate on water bath to obtain the product. Calculate the yield and estimate iodine in the complex iodometrically. Record the IR and interpret the spectra.

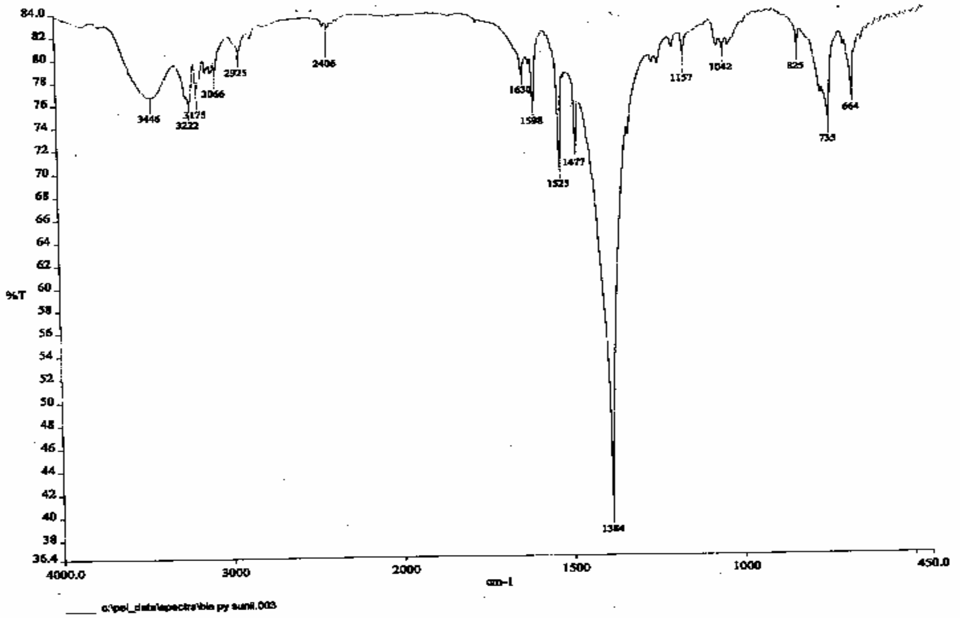
Iodide tests:

Take two small portions of product in two test tubes and add hydrochloric acid (6 ml, 0.1 M) and sodium hydroxide (6 ml, 0.1 M) to each of the tubes. Now add potassium iodide solution (1 ml, 0.1 M) to each of the test tube. Note your observations and conclusions about stability of I^+ in acidic and basic solutions.

Tasks:

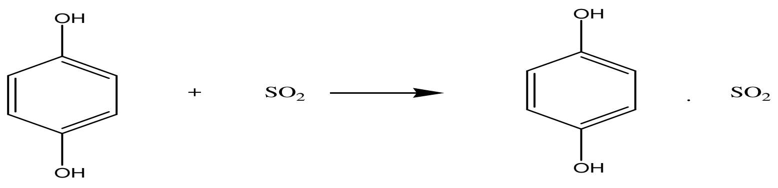
1. Estimate iodine
2. Record IR and interpret the spectra.
3. Calculate the percent yield

FT-IR spectra (KBr, cm^{-1}) of bis-pyridine iodo nitrate



Experiment No.8

Hydroquinone has extensive hydrogen bonded structure and form different types of clathrate compounds. Sulphur dioxide can also form clathrate with the hydrogen bonded network of hydroquinone. A 1:1 molecular complex is formed by passing of SO_2 to hydroquinone solution as per the following equation:



Requirements:

1. Hydroquinone 2. Hydrochloric acid
3. Sodium metabisulphite 4. Sulphur dioxide

Hydroquinone clathrate with sulphur dioxide:

Prepare a saturated solution of hydroquinone (5 g in 50 ml water) heated to 50^0C and to this solution pass a slow stream of sulphur dioxide gas in fume cupboard for 30 minutes. Yellow precipitate will be obtained. Filter the precipitate through buchner funnel and dry it by filter paper pressing.

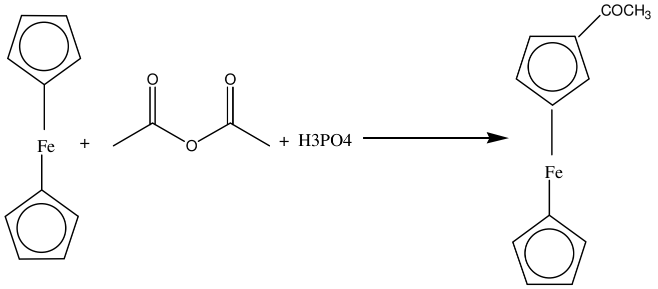
Note: Sulphur dioxide gas is produced by reaction of sodium metabisulphite and hydrochloric acid.

Tasks:

1. Calculate the yield
2. Record the IR spectra

Experiment No.9

The experiment is based on the following equation:



Requirements:

1. Ferrocene 2. Calcium chloride

3. Phosphoric acid
4. Acetic anhydride
5. Sodium bicarbonate

Acetylation of ferrocene:

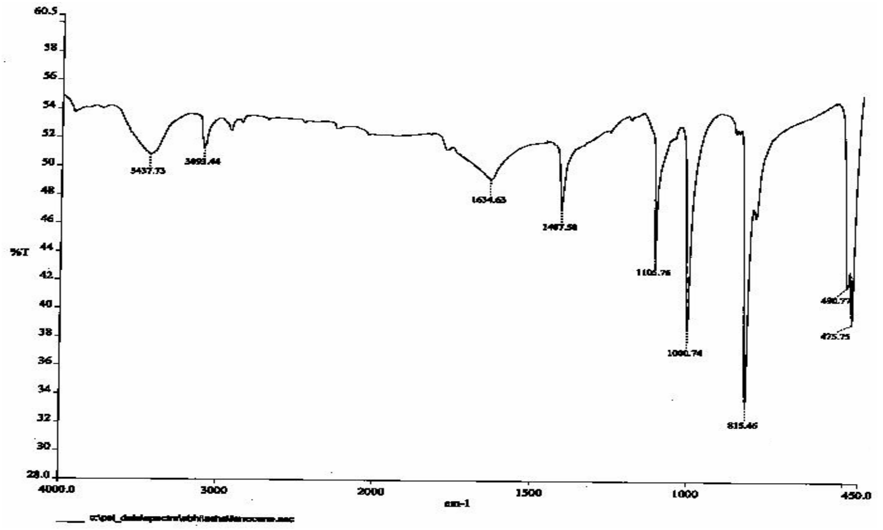
Procedure:

Take ferrocene (1.5 g, 8 mmol) and acetic anhydride (5 ml 5.25 g, 87 mmol) in an oven dried, medium sized flask. To this add phosphoric acid (1 ml) with constant stirring. The solution is protected with guard tube containing anhydrous calcium chloride. Heat the reaction mixture in a steam bath for 15 minutes and then pour on to about 20 g of ice taken in large beaker. Stir the solution and when the ice melts neutralize by adding solid sodium bicarbonate until carbon dioxide cease to evolve. Cool in an ice bath for 30 minutes to ensure complete precipitation of the ferrocene from the solution. Filter the solid in a buchner funnel under suction. Wash it with cold water until the filtrate is pale orange. Dry the solid in air for 10 minutes. The solid contains ferrocene and monoacetyl ferrocene. The mixture is to be purified by column chromatography using silica gel and petroleum ether eluent.

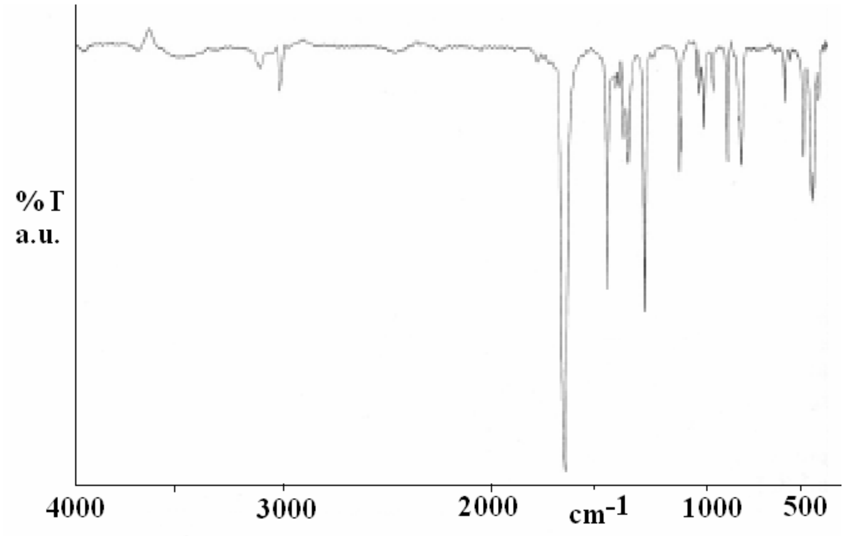
Tasks:

1. Record IR spectra
2. Calculate the yield

FT-IR spectra (KBr, cm^{-1}) of ferrocene



FT-IR spectra (KBr, cm^{-1}) of acetyl ferrocene



Experiment No.10

a. Synthesis and characterization of *tris*-triphenylphosphine copper (I) nitrate:

Requirements:

1. Copper nitrate pentahydrate
2. Ethanol
3. Triphenyl phosphine
4. Nitric acid
5. Sodium hydroxide
6. Sulphuric acid
7. Sodium thiosulphate
8. Potassium iodide
9. Starch

Procedure:

Dissolve a solution of copper nitrate trihydrate (0.365 g, 1.5 mmol) in minimum amount of ethanol and in another beaker dissolve triphenylphosphine (1.965 g, 7.5 mmol) in 30 ml ethanol (warm the solution to dissolve). Mix the two solutions, reflux for half an hour and cool. Filter the white crystalline solid and wash with ethanol. (If necessary recrystallize from methanol)

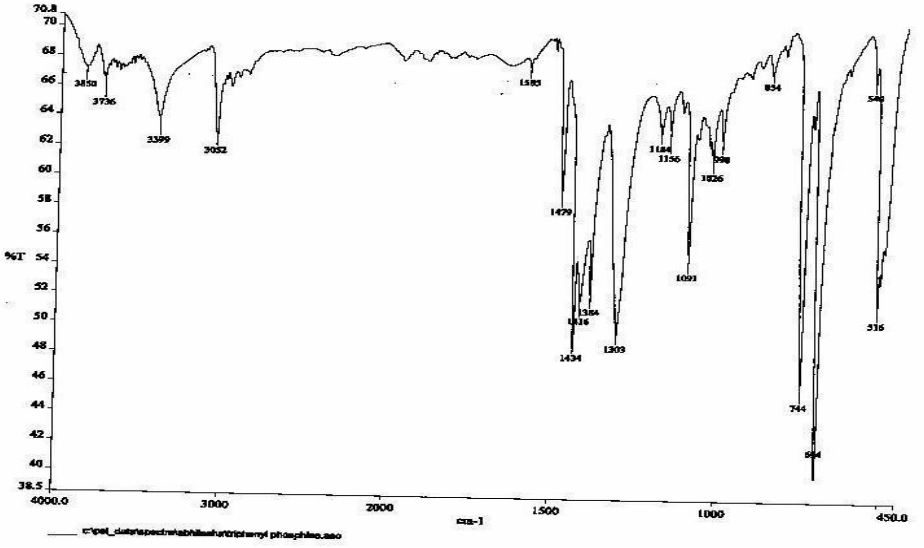
Tasks:

1. Calculate percent yield
2. Estimate copper

Estimation of copper:

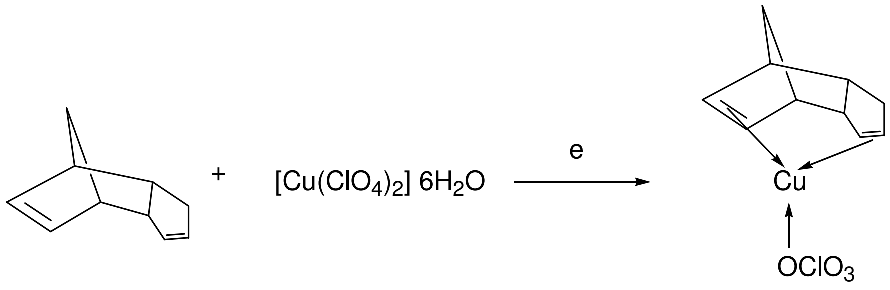
Decompose an accurately weighed (0.10 - 0.15 g) copper complex, by heating with minimum volume of concentrated nitric acid. To the resulting oily mass, add water and filter. Treat the filtrate with dilute alkali to precipitate copper hydroxide. Filter solution and reject the mother liquor. Wash copper hydroxide with water and dissolve it with dilute sulphuric acid (10 ml, 1M). The resulting solution contains copper (II) sulphate (aq.), estimate copper iodometrically.

FT-IR spectra (KBr, cm^{-1}) of *tris*-triphenylphosphine copper (I) nitrate



b. Electrochemical synthesis of mono-dicyclopentadiene copper(I) perchlorate

The experiment is based on the following equation



Requirements

1. Copper(II) perchlorate hexahydrate 2. Dicyclopentadiene 3. Copper plate

4. DC power supply unit

Procedure

Take a solution of copper (II) perchlorate hexahydrate (0.74 g, 2 mmol) in acetonitrile (50 ml) in a beaker (100 ml). Add dicyclopentadiene (0.29 g, 2.1 mmol) to this solution. Take two copper plates of 0.1 cm x 2 cm x 6 cm dimension and place them vertically in parallel position (about 3 cm apart) in the beaker. Connect the copper plates by copper wire with the help of steel clips to a direct current power supply unit. Set the voltage to 10 V and current 0.1 amp and pass current through the solution for 6 hrs. (stop passing current if the solution gets heated and on cooling once again pass electricity) One of the electrodes will dissolve in the solution and a white crystalline compound will be deposited on the other copper plate. Take out the white precipitates by decanting the solvent.

(Caution: Perchlorate salts are potentially explosives; donot treat them with protic solvent in basic medium or donot heat the perchlorate salts to high temperature)

Tasks:

1. Record the IR spectra of the compound and interpret.
2. Estimate copper in the complex

Experiment No.11

Study on synthesis and properties of soap

Introduction:

Soap are salts of fatty acids; consists of long chain hydrocarbon attached to carboxylic acid group on one end which is bonded generally to an alkali metal ion, sodium or potassium. The structure of a soap molecule is shown below:

CH_3-$(CH_2)_n$-COO^- Na^+ where n = is an integer generally greater than 10.

Preparation of soap

Requirements:

1. Sodium hydroxide
2. Sodium chloride
3. Ethanol
4. Boiling chips
5. Erlenmeyer flask
6. Beakers
7. Measuring cylinder
8. Funnel
9. Tissue paper
10. Glass rod
11. Watch glass

Procedure:

Dissolve sodium hydroxide, (2 g) in a mixed solvent of ethanol (10 ml) and distilled water (10 ml), in a beaker (100 ml) by continuous stirring. Place vegetable ghee (2 g) in a flask (125 ml) and add the sodium hydroxide solution to this. Add also a few boiling chips and cover the flask with a watch glass and heat it over a water bath. As the water in the water bath boils, lower the reaction flask to the bottom of the bath to make contact with the boiling water surface. Allow the reaction to proceed for at least 30 minutes.

In the mean time prepare a sodium chloride solution by dissolving about 15 g of sodium chloride, in 80 ml of water in a beaker (500 ml). Add ice about 25 g to this solution.

After 30 minutes, pour the hot reaction mixture into the salt solution. Stir the resulting mixture in the beaker for about five minutes. Decant the water and wash the collected soap twice with l0 ml portions of ice-cold water. Dry the soap by pressing over tissue paper.

Record an IR of ghee and soap and interpret the spectra.

Study on properties of soap

Emulsifying properties:

Soaps are capable of emulsifying or dispersing oils and similar water-insoluble substances. To prove this:

1. Place 0.5 ml of oil (cooking oil, or household lubricating oil) each in two separate test tubes.
2. Add 5 ml of water to one tube, 5 ml of soap solution (0.05 g of soap in 5ml water) to another tube. Shake each tube vigorously for about one minute.
3. Write your inference.

Alkalinity

Test the alikalinity of your soap solution (used for emulsification purpose) with red litmus paper.

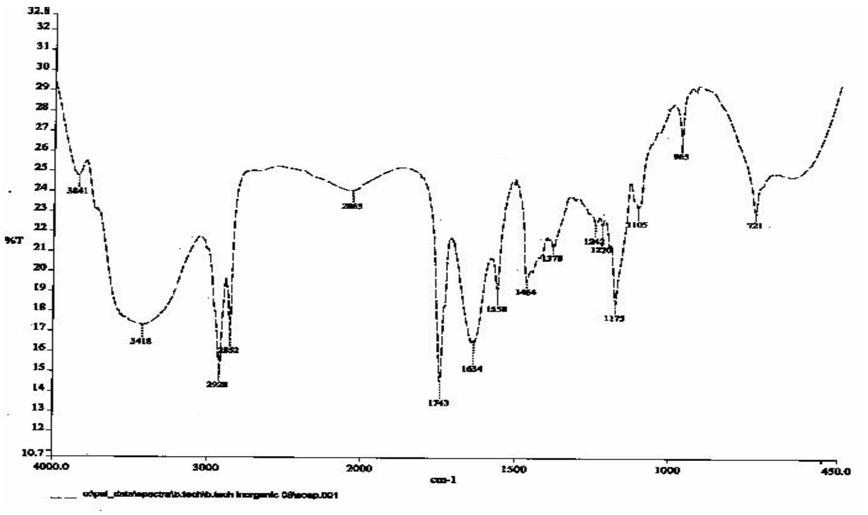
1. Prepare a soap solution by dissolving soap (0.05 g) in hot water and; add 1 drop of phenolphthalein solution.
2. Titrate the soap solution with standard hydrochloric acid (0.01 M) and infer the results obtained.

Reaction of soap with mineral Acid

1. Prepare a soap solution by dissolving soap (0.05 g) in hot water (5 ml) in a conical flask.
2. Add hydrochloric acid (10 ml, 0.1 M) drop-wise. After addition of hydrochloric acid check the pH of the solution and write the inference.

3. Titrate the solution with NaOH (0.1 M) using phenolphthalein indicator and infer the results.

FT-IR Spectra (KBr, cm^{-1}) of soap from vegetable ghee



Experiment No.12

Preparation of a fertilizer:

Producing fertilizers is very important. This experiment involves preparation of ammonium phosphate. Ammonium phosphate is a popular and effective fertilizer.

$3\ NH_4OH + H_3PO_4 \rightarrow (NH_4)_3\ PO_4 + 3\ H_2O$

Requirements:

1. Phosphoric acid
2. Ammonia
3. Indicator paper

Procedure:

Take 20 ml of phosphoric acid in a 100 ml beaker. Add ammonia solution to the phosphoric acid in portions at a time with stirring until the p^H reaches 7 or slightly above (test with indicator paper). Evaporate the solution to one fifth of its original volume and cool (while heating care should be taken so that the solution do not bump out). On cooling white crystalline solid is formed. Filter off the crystals and dry.

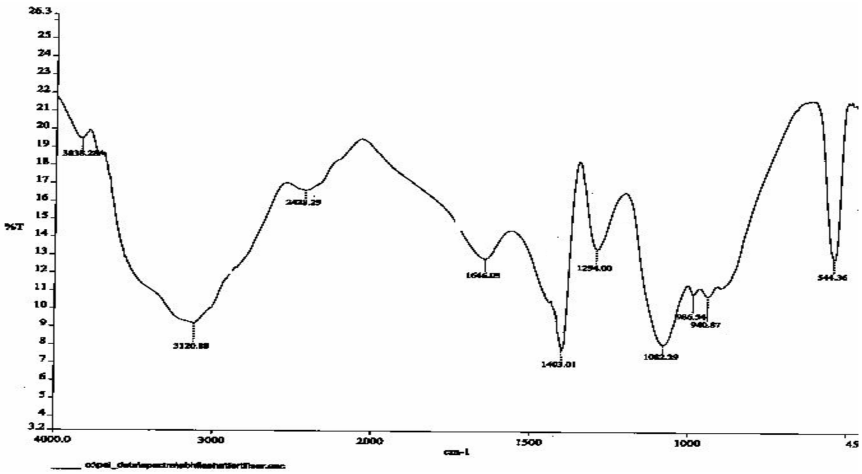
Estimation of phosphate

Take ammonium phosphate (0.2 g, 1.34 mmol) in water (10 ml). Add 1 drop of methyl red indicator followed by ammonia buffer (10 ml, pH 10). Yellow colour appears. To this solution add magnesium sulfate heptahydrate (0.984 g, 4.02 mmol) and stir. Allow the solution to stand one hour. Filter the solution to conical flask (100 ml) and titrate the filtrate with standard EDTA (0.05 M) solution. Calculate the amount of phosphate by substracting the precipitate as magnesium phosphate.

Tasks:

1. Record the IR spectra
2. Calculate the NP value of the fertilizer

FT-IR spectra (KBr, cm^{-1}) of ammonium phosphate



Experiment No.13

Preparation of alloy of lead and tin:

Requirements:

1. Lead metal 2. Tin metal

3. Copper wire 4. Copper plate

5. Solder

Procedure:

Place lead metal (1 g) in a silica crucible and to this add tin metal (1 g). Melt the metals over silica crucible over a flame. Cool the melt and remove the solid from the crucible. Repeat the experiment with 1.5 g of lead and 0.5 g of tin and also with 0.5 g lead and 1.5 g of tin.

Record the DSC and powder XRD of the three samples and infer the results.

Soldier a copper wire over a copper plate by prepared alloy. Try the same with pure lead and tin. Infer the result.

Experiment No.14

Preparation of manganese dioxide nano-particles:

Requirements:

1. Manganese sulphate monohydrate
2. Potassium permanganate
3. Sodium hydroxide

Procedure:

Prepare the following solutions

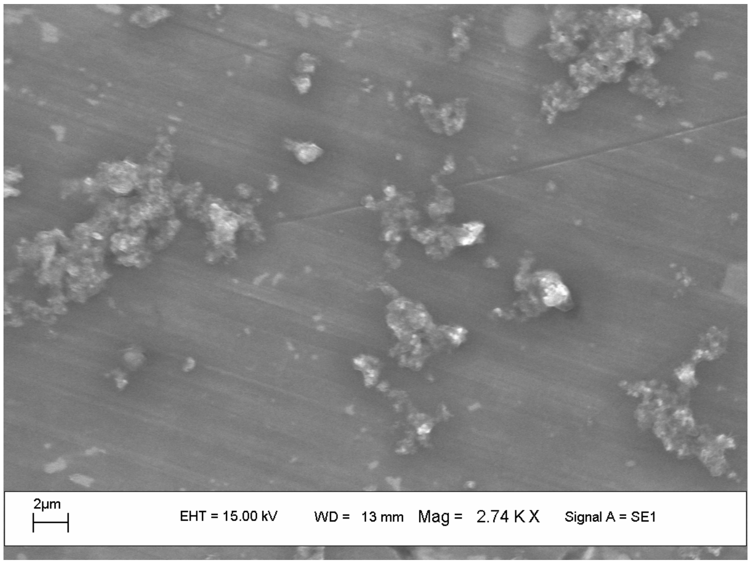
1. Manganese sulfate monohydrate, (1.69 g, 10 mmol) in water (3 ml).

2. Sodium hydroxide (2.5 mol, 2.4 ml).

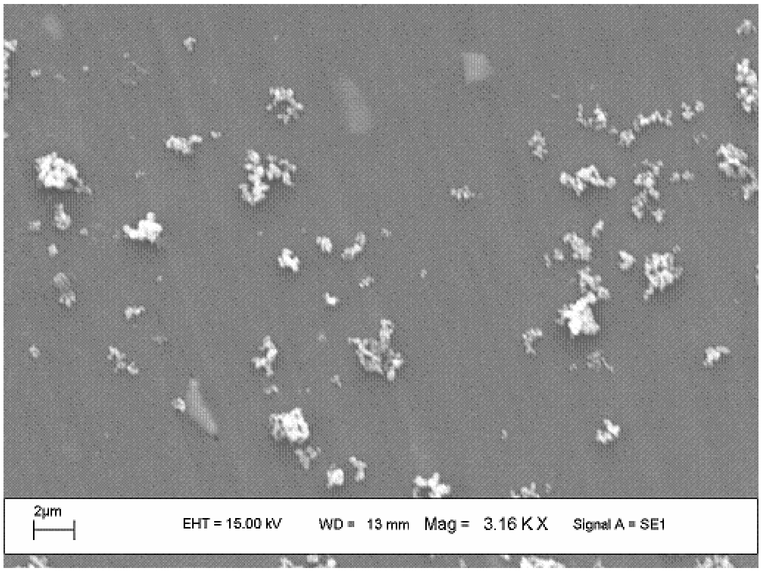
Add simultaneously these two solutions to a hot solution of potassium permanganate (1.9 g, 1.0 mmol) in water (15 ml). Stir the solution for 1hr and isolate the brown precipitate of manganese dioxide by filtration. Dry the product in a dessicator under vacuum and grind finely. Record the powder X-ray diffraction and SEM.

Repeat the experiment by adding cetyltrimethyl ammonium bromide (CTAB, 0.5 g) to manganese solution (Step 1).

Scanning electron microscopic view of manganese dioxide nano-particles



Scanning electron microscopic view of manganese dioxide nano-particles (prepared by adding CTAB)



Experiment No.15

a. Preparation of potash alum:

Theory:

Alum is one of the most widely used industrial chemicals mainly due to the usefulness in the pulp and paper industry as well as in water and wastewater treatment plants. It is effective for a broad range of treatment problems because it can function as a coagulant, flocculent, precipitant and emulsion breaker. As a coagulant and flocculent, alum removes turbidity, suspended solids and colloidal colours and clarifies potable process and waste water.

The chemical reaction in the formation of potash alum is as shown below:

Aluminum rapidly reacts with hot aqueous KOH to give $KAl(OH)_4$. By reacting $KAl(OH)_4$ with sulphuric acid, potash alum is obtained

$$2Al + 2\ KOH + 6H_20 \longrightarrow 2\ KAl(OH)_4 + 3\ H_2O$$

$$2\ KAl(OH)_4 + 4\ H_2SO_4 \longrightarrow K_2SO_4\ Al_2SO_4.\ 24\ H_2O$$

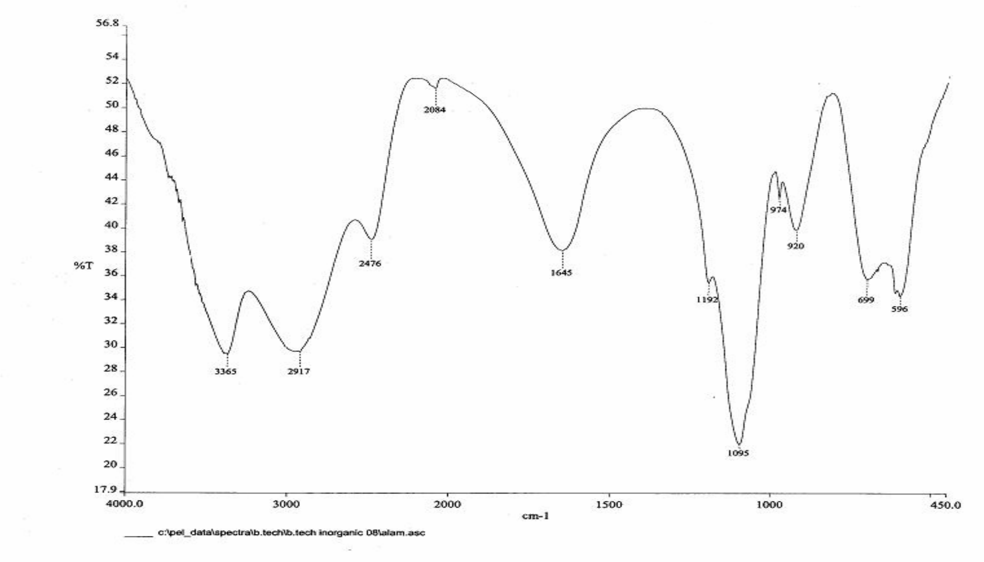
Requirements:

1. Aluminum foil
2. Potassium hydroxide
3. Sulphuric acid
4. Ethanol
5. Sodium hydroxide

Procedure:

Take aluminum scrap (0.5 g) and cut into very small pieces. Place it in a beaker (250 ml) and carefully add potassium hydroxide solution (1.75 g in 15 ml water). Heat the beaker gently and cover it with a watch glass. Continue heating till all the aluminum has dissolved and have an ash-colored solution. (Do not heat to dryness). Filter the warm solution carefully through a thin layer of cotton or glasswool. Cool the solution and add slowly sulphuric acid (15 ml, 6 M) with stirring. The solution should give some solid precipitate after the addition of the acid. Heat the solution to dissolve the solids. If the solution is still not clear, filter again while it is warm. Cool the clear solution in an ice bath for 20 min to get the alum crystals. Filter the solution by using a filter paper, dry and determine the yield. Recrystallize the product from water and determine melting point of the crystalline alum.

FT-IR spectra (KBr, cm^{-1}) of potash alum



b. Preparation of a super conductor:

Requirements:

1. Yttrium hydroxide
2. Barium peroxide
3. Cupric oxide
4. Aluminium oxide

Procedure:

Take yttrium hydroxide (0.60 g) and transfer them in a small beaker (make sure the beaker is dry). Weigh separately equivalent amounts of barium peroxide and cupric oxide transferring and place them in the beaker.

Mix the three materials in a mortar. Make a pallet of the mixture and place in an alundum (a form of Al_2O_3) boat and heat it in a furnace. The furnace is heated to 930^0C over a period of about 8-12 hours, held at 930°C for 12-16 hours, allow to cool to 500°C and hold there for 12-16 hours. Finally the furnace is turned off and allowed to cool the mixture to room temperature. The cooled pellets are stored in a dessicator.

The finished pellet should be dark gray to black.

The overall reaction (with only the metal ions balanced) is given by:

Y_2O_3 + 2 BaO_2 + 3 CuO ------> $YBa_2Cu_3O_7$

Meissner effect

Using the plastic forceps, place a superconductor pellet in a cup containing liquid nitrogen. When the pellet cools below the critical temperature, levitate a magnet above the superconductor. Touch the pellet gently with a forcep and the pellet starts to spin.

Experiment No.16

a. Photochemical oxidation of iso-propanol by copper (II) acetylacetonate:

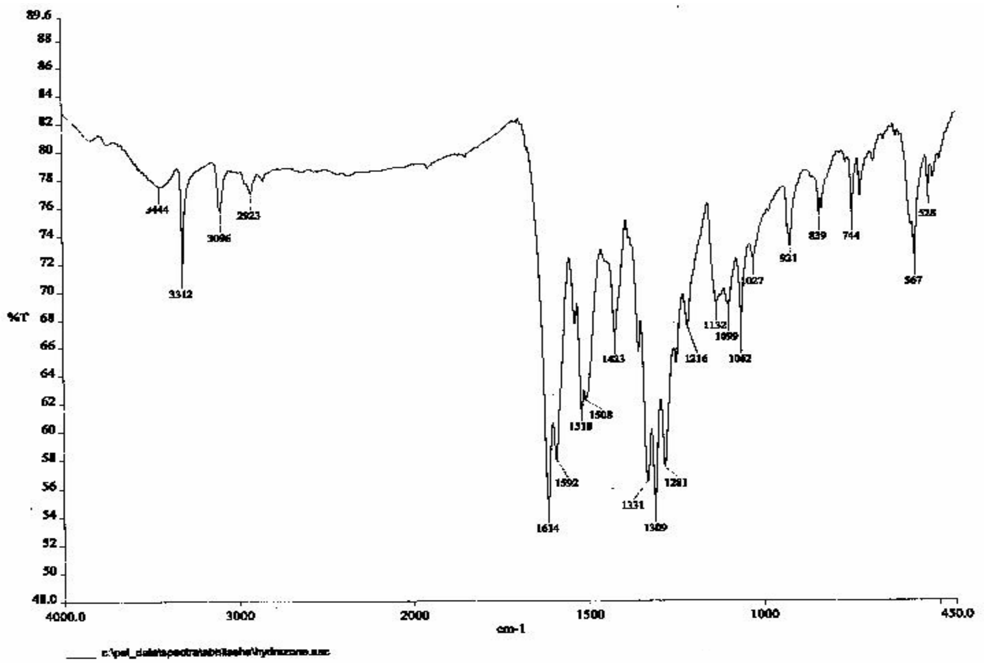
Requirements:

1. Copper (II) acetylacetonate
2. Benzophenone
3. Isopropyl alcohol
4. Ethanol
5. Acetone
6. 2,4-dinitro phenyl hydrazine

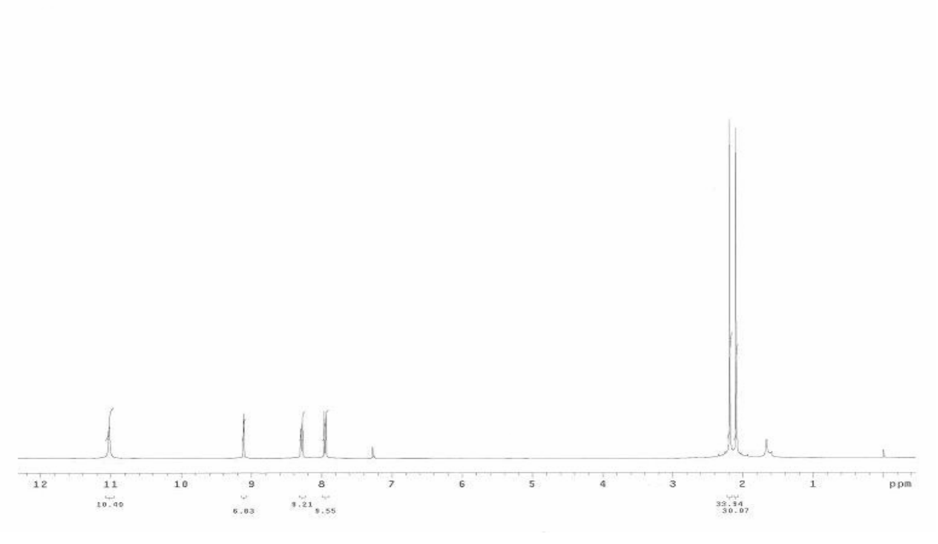
Procedure:

Take a solution of copper (II) acetylacetonate (0.05 g, 0.19 mmol) and benzophenone (0.112 g, 0.64 mmol) in isopropyl alcohol (100 ml) in a quartz tube and photolysed the solution for 1 hr by using a lamp (630 nm). After I hr copper suspension will be formed and filter off the copper formed. Add 2,4-dinitro phenyl hydrazine solution to the filtrate. A yellow precipitate will be obtained. Filter and recrystallise the solid from ethanol / water. Characterize it as 2,4-dinitro phenyl hydrazone derivative of acetone. (m.p. 124-126.5^{0}C)

FT-IR spectra (KBr, cm^{-1}) of N - (2, 4-dinitro phenyl) - N'- isopropylidene hydrazine



^{1}HNMR spectra (400 MHz, CDCl$_3$, ppm) of N - (2,4-dinitro phenyl) - N'- isopropylidene hydrazine



b. Extraction of chlorophyll from plant leaves

Chlorophyll the green magnesium containing pigment of a leaves can be extracted by isopropanol.

Requirements

1. Plant leaves 2. Isopropyl alcohol

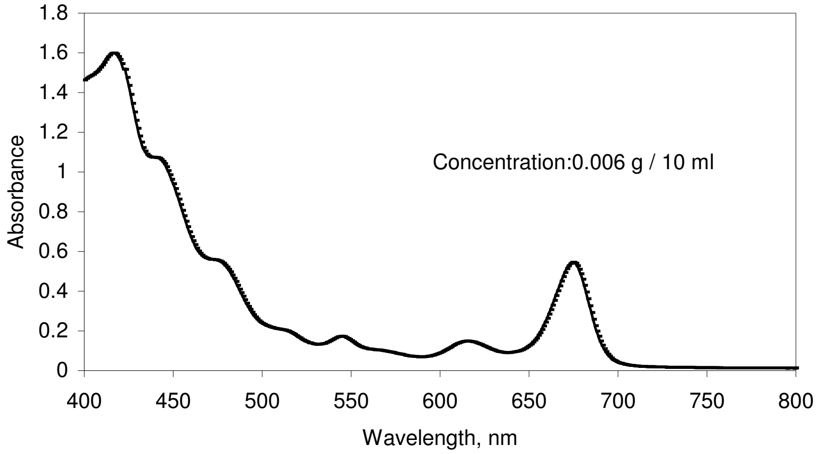
Procedure

Take finely torn plant leaves (20 g) in a beaker (500 ml). Add isopropyl alcohol (aprox 250 ml) to soak the leaves. Wrap the beaker with carbon paper. Allow the mixture to stand for overnight. Verify that the isopropyl alcohol solution is green. Filter the solution containing the leaves and evaporate the green filtrate to dryness in a rotary flash evaporator. Transfer the sample to a sample tube after dissolving it in a minimum amount of isopropyl alcohol. Remove the solvent under reduced pressure or on a water bath. Store the crude chlorophyll in dark.

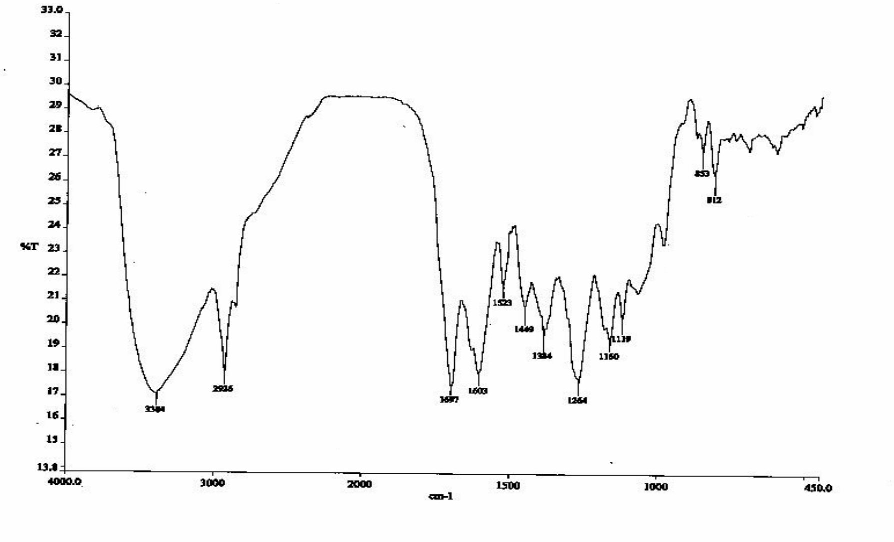
Tasks:

4. Record the visible spectra and interpret
5. Record the IR spectra and interpret.
6. Estimate magnesium.

UV-Visible spectra (MeOH) of crude chlorophyll



FT-IR spectra (KBr, cm^{-1}) of crude chlorophyll



Experiment No.17

Magnetic susceptibility:

This experiment is to understand the magnetic moment of a series of transition metal salts. The experiment is also to find out deviation from spin only value of magnetic moment of the salts which is given by equation

$\mu = \sqrt{n(n+2)}$ BM

where n is the no of unpaired electrons.

Requirements:

1. Magnetic balance
2. Capillary
3. Manganese (II) chloride
4. Cobalt (II) chloride
5. Nickel (II) chloride
6. Copper (II) chloride
7. Zinc (II) chloride

Procedure:

Take finely ground manganese (II) chloride, cobalt (II) chloride, nickel (II) chloride, copper (II) chloride and zinc (II) chloride in five separate capillary tubes. Record their magnetic susceptibility in a magnetic balance.

Magnetic moment measurement:

Insert an empty preweighed sample tube to the tube guide of the instrument, measure the magnetic deflection caused by the tube (R_0). Take weighed amount of the finely ground solid sample in a sample tube. Insert the sample tube with the sample to the tube guide and record magnetic deflection caused by the tube (R). Calculate the mass susceptibility, X_g using equation given below:

$$X_g = \frac{C_{bal}\, l\, (R - R_0)}{10^9 m}$$

where,

l = Sample length (cm)
m= Sample mass (g)
R= Reading for tube plus sample
R_0= Empty tube reading
C_{bal}= Balance calibration constant

The molar susceptibility,

$$X_m = X_g M$$

where, M= molecular formula weight of the substance

For compounds containing a paramagnetic ion, X_M will be less than susceptibility / g atom of the paramagnetic ion, X_A, because of the diamagnetic contribution of the other groups or ligand present. Since magnetic moments are additive, X_A can be obtained from X_M by the addition of the appropriate corrections.

$$XA = XM - \sum_{I} X_I$$

where, $\sum X_I$ = diamagnetic correction.

The effective magnetic moment (μ_M) is calculated from the equation below

$$\mu = 2.84\, X_M\, T$$