S.Y.B.SC. INORGANIC CHEMISTRY

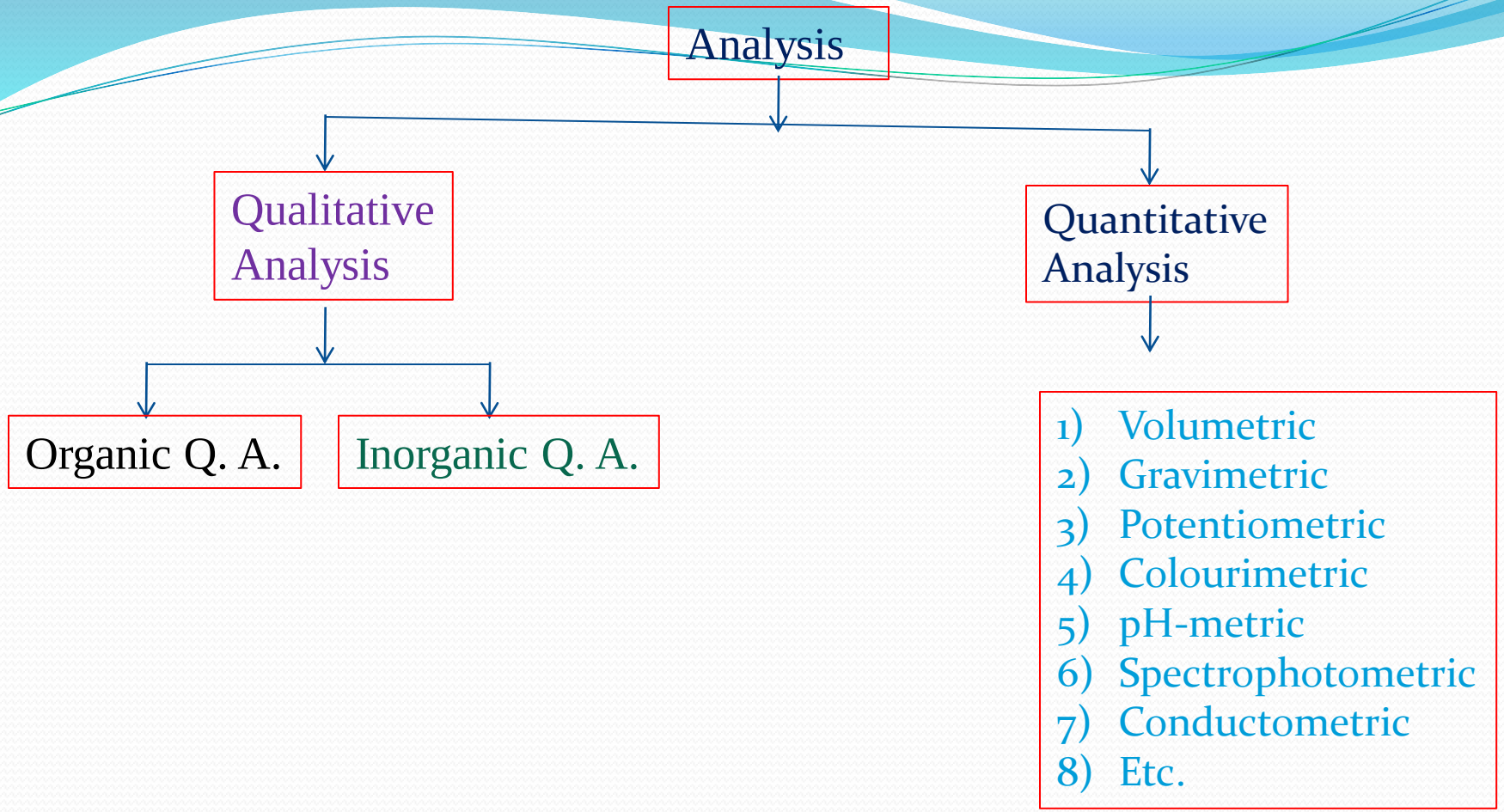
PRACTICAL SEM-II

ONLINE LECTURE NO. 1

GRAVIMETRIC ANALYSIS

DATE:- 11, MAY 2021

TIME: (12.30 P.M.)


Analysis
Qualitative Analysis
Quantitative Analysis
Organic Q. A.
Inorganic Q. A.
1) Volumetric
2) Gravimetric
3) Potentiometric
4) Colourimetric
5) pH-metric
6) Spectrophotometric
7) Conductometric
8) Etc.

In gravimetric analysis, the amount of substance is determined by reacting it with Other substance of known chemical composition and then can be readily isolated, purified and weighed.

Scheme: -

Weighing of sample → Preparation of solution → Separation of interfering components (impurities) → Establishment of suitable experimental conditions (either by control of pH by adding buffer or change of Oxidation State, addition of masking agent) → Addition of suitable organic or inorganic precipitant to hot and dilute solution of sample → Digestion of the ppt → Separation of precipitate by filtration → Washing of precipitate to purify it from the adjoined water soluble impurities → Drying and ignition of the precipitate to bring it to a constant weight → Weighting of Residue → Calculation.

Conditions of precipitation: →

1) Precipitation must be carried out from hot solution.
2) Precipitation should be carried out in dilute solution.
3) Precipitation should be carried out in presence of a suitable reagent.
4) The precipitant should be added very slowly with thorough stirring of solution.
5) Precipitation must be carried out with excess of precipitant.
6) The precipitation should be followed by digestion or ageing.
7) The precipitate should be wash with hot and dilute solution of a suitable electrolyte.

To estimate Ni as Ni-DMG

[C] GRAVIMETRIC ANALYSIS

Experiment No.1

Aim

Estimation of nickel as nickel dimethyl glyoxime gravimetrically (By counterpoise filter paper method)

Theory

Nickel is precipitated as nickel dimethylglyoxime by adding 1% alcoholic solution of dimethylglyoxime (DMG) to an ammonical solution of nickel salt. $Ni(DMG)_2$ a scarlet precipitate is obtained. It is digested, filttered (through No. 40) washed with hot water, dried at 100°C in a oven and weighed.

Preparation of solutions

1) Ni^{++} stock solution
   30 g of nickel sulphate ($NiSO_4.7H_2O$) dissolved in distilled water and dilute to one litre
   (Distribute 8-12 ml of stock solution of nickel sulphate to the students)
2) 1% DMG solution
   1 g of DMG salt dissolved in 100 ml ethyl alcohol (if need, heat it in hot water both)
3) (1:1) ammonia
   100 ml ammonia + 100 ml of water

Apparatus

Beakers (250 ml) lebelled as A, B, glass rod (rubber-tipped), wash bottle, hot water bath, whatman filter paper No. 40, filter stand with funnel, oven or metal cone.

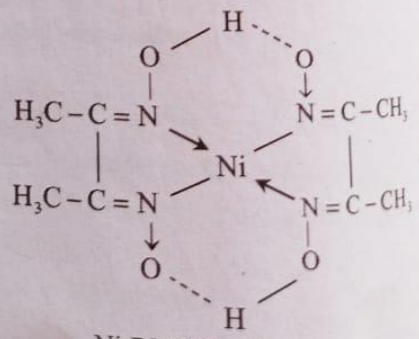
Chemicals

1) Ni^{2+} stock solution ($NiSO_4.7H_2O$).
2) 1% Dimethyl glyoxime (about 25-30 ml)
3) Aqueous ammonia (1:1)

Reaction

$$Ni^{+2} + 2\ \underset{\underset{CH_3-C=NOH}{|}}{\overset{CH_3-C=NOH}{|}}\ + 2OH^- \longrightarrow$$

DMG



Ni-DMG Scarlet red ppt.

Procedure

A) Preparation of the solution

1) Ni^{2+} solution given in a 100 ml volumetric flask is diluated with distilled water upto the mark and transfers it in a beaker (A).

2) Pipette out exactly 50 ml solution from beaker (A) and transfer it in a beaker (B), add to it 50 ml distilled water. Heat the solution.

B) Precipitation

1) To this hot solution (beaker B) add drop wise 1% alcoholic solution of DMG (25-30 ml) with constant stirring.

2) Take 25-30ml ammonia (1:1) in a beaker. Add aqueous ammonia (1:1) drop wise, with constant stirring, till formation of the precipitation / complex (scarlet red) is complete. Add 2ml excess of ammonia.

C) Digestion

1) Digest the scarlet red ppt. on hot water bath (low flame) about 30 minute. When the precipitated settled, test the completion of the precipitate by adding DMG to the supernant liquid. (There is no red turbidity), cool the solution. Digestion is done to increase particle size for easier filtration

D) Filtration of precipitate by using Whatman paper No. 40 and washing of the precipitate.

1) Weigh Whatman filter paper (W_1), filter the cold solution (beaker B) through filter paper. Initially transfer supernant solution and then transfer the precipitate to the filter paper. Wash the beaker with small quantity of water and transfer the ppt. and washing carefully into the filter paper. Clear filtrate should be collected in another beaker.

2) Wash the residue with distilled water till free from SO_4^{2-} ions (test with $Ba(NO_3)_2$ till no white ppt) and Cl^- ions (test with $AgNO_3$ till no white ppt)

E) Drying of the residue

1) Place the metal cone on tripod stand with wire gauze. Now, place the funnel along with filter paper and heat it carefully till residue is completely dry. (or mark the funnel with T. No. and place it in preheated oven at 110°C for half an hour). Cool the funnel with residue in desicator).

F) Weighing of the residue

1) Weigh the residue along with filter paper (W_2), Repeat the drying again for 10-12 min. Cool, weigh till constant weight. (Take care filter paper should not turn black due to drying).

Observation table

Obs. No.	Description	Symbol	Weight
01	Weight of the filter paper (whatman paper NO. 40)	W_1	..... g
02	Weight of the filter paper + residue. After complete drying.	W_2	..... g
03	Weight of the residue (Ni-DMG)	$W_2 - W_1 = A$ g	..... g

Calculations

50 ml of diluted solution of nickel sulphate = A g of Ni-DMG

$\therefore$ 100 ml diluated solution (given) = $(A \times 2)$

= B

From stoichiometric equation

1 Ni-DMG = 1 $NiSO_4.7H_2O$

= 1 Ni^{+2}

$\therefore$ 288 g of Ni-DMG $\equiv$ 58 g of Ni^{+2}

B g Ni-DMG $\equiv \left(\frac{58}{288}\right) \times B$ g of Ni^{+2}

= $0.2013 \times B$

= C g of Ni^{2+}

Result

Sr. No.	Description	Symbol	Results
1	50 ml of dilute solution contain …….. g of Ni-DMG	A	……g
2	Amount of the nickel in the given solution	C	……g

Experiment No. 2

Aim

Estimation of barium as barium sulphate gravimetrically (By ignition method)

Theory

In the estimation of barium from $BaCl_2.2H_2O$ solution, hot 2N H_2SO_4 is used as a precipitating reagent. Where $BaSO_4$, white crystalline precipitate is obtained, NH_4Cl is added to avoid the formation of Colloidal precipitate of $BaCl_2$.

The precipitate is digested, filtered, washed, dried and ignited and weighed as $BaSO_4$.

Reaction

$$Ba^{2+} + SO_4^{2-} \longrightarrow BaSO_4 \downarrow$$

$$BaCl_2 + H_2SO_4 \longrightarrow BaSO_4 \downarrow + 2HCl$$

white ppt

Preparation of Solutions

1) Ba^{2+} solution
   50 g of barium chloride ($BaCl_2.2H_2O$) + 5 ml conc. HCl, dissolve in distilled water and dilute to one litre with water.
2) 2N H_2SO_4
   60 ml conc. H_2SO_4, add carefully to distilled water and make the volume one litre

Apparatus

Beakers labeled as A, B, glass rod, wash bottle, burette, silica crucible + lid, desiccators, pipe-clay tringle, pair of tongs, filter stand with funnel, whatman paper No. 42, glazed paper.

Chemicals

1) Ba^{2+} solution ($BaCl_2.2H_2O$).
2) 2N H_2SO_4 (precipitating reagent)
3) Solid NH_4Cl

Procedure

A) Preparation of the solution

1) Dilute the given solution of Ba^{2+} ion ($BaCl_2.2H_2O$) in a 100 ml. volumetric flask with distilled water upto the mark and transfer it in beaker (A).
2) Pipette out exactly 50 ml solution from beaker A and transfer it into a beaker (B). Add to it 50 ml distilled water, 2ml conc. HCl and about one gram of solid NH_4Cl. Heat the solution to boil and keep it on asbestos sheet.

B) Precipitation

1) Take about 50 ml 2N H_2SO_4 in a beaker and heat it.
2) Add drop wise this solution to beaker B solution with constant stirring till white ppt. of $BaSO_4$ is completed. Let the precipitated settle, then add 2N H_2SO_4 from the side of the beaker. No white turbidity to the supernant liquid indicates precipitation is completed, add 2ml H_2SO_4 excess.

C) Digestion

1) Digest the white precipitate on hot water bath at low flame for about 30-40 minutes. The size of the crystals is increased due to digestion.

D) Filtration of precipitate by using Whatman paper No. 42 and washing of the precipitate.

1) Filter the precipitate from beaker B through Whatman paper No. 42 carefully (first supernant liquid, then precipitate). Wash the beaker and transfer washing also to the filter paper.
2) Wash the residue with hot distilled water using wash bottle. Residue should be free from SO_4^{2-} ions (test with $Ba(NO_3)_2$, no white ppt.), and Cl^- ions (test with $AgNO_3$, no white ppt.)

E) Drying of the residue

Dry the filter paper containing residue on metal-cone or in an oven at about 120°C.

F) Ignition of the residue

1) Take a previously cleaned silica crucible and lid and weigh it (W_1)
2) Transfer the residue from filter paper on a glazed paper and cover it with clean funnel.
3) Burn the filter paper carefully over the crucible and collect all the matter in the crucible.
4) Place the crucible + lid, on pipe-clay triangle placed on a tripod stand.
5) Heat it carefully so that it will turn to white ash and cool it.
6) Now, transfer the residue from the glazed paper into crucible carefully.
7) Heat it for 5 minutes on low flame and then for 30 minutes on high flame.
8) Cool it and keep it in the desiccator.

G) Weighing of residue

1) Weigh the (crucible + lid + residue) say W_2. Repeat the heating, cooling and weighing twice or thrice to ensure constant weight (W_2).

Observation table

Obs. No.	Description	Symbol	Weight
01	Weight of empty crucible + lid	W_1	..... g
02	Weight of crucible + lid + residue ($BaSO_4$) on heating.	W_2	..... g
03	Weight of residue ($BaSO_4$)	$W_2 - W_1$	..... g

Calculations

50 ml of diluted solution of $BaCl_2.2H_2O$ = A g of $BaSO_4$

$\therefore$ 100 ml diluated solution of $BaCl_2.2H_2O$ = (A × 2) g of

= B g of $BaSO_4$

From stoichiometric equation

$$1\ BaSO_4 \equiv 1\ BaCl_2.2H_2O$$
$$\equiv 1\ Ba^{+2}$$
$$233.36\ g\ BaSO_4 \equiv 244.36\ g\ of\ BaCl_2.2H_2O$$
$$\equiv 137\ g\ of\ Ba^{+2}$$

233.36 g of $BaSO_4$ gives 137 g of Ba^{+2}

233.36 g of $BaSO_4$ contains $\equiv$ 207.29 g of Ba^{+2}

$$B\ g\ of\ BaSO_4 \equiv \left(\frac{137}{233.36}\right) \times B\ g\ of\ Ba^{+2}$$
$$= 0.5870 \times B^{+2}\ g\ of\ Ba^{+2}$$
$$= C\ g\ Ba^{+2}$$

Result

Sr. No.	Description	Symbol	Results
1	50 ml of dilute solution contain A g of $BaSO_4$	A	.....g
2	Amount of barium in the given solution (B) g	C	.....g

Experiment No. 3

Aim

Estimation of lead as lead chromate $PbCrO_4$ (By Gooch crucible method)

Theory

Lead is estimated as lead chromate gravimetrically. It is precipitating completely by adding hot potassium chromate solution to the hot and dilute soluble salt of lead solution in presence of buffer solution of acetic acid and sodium acetate. It prevents the hydrolysis of lead nitrate and dissolution of lead chromate in nitric acid which is formed in the reaction. Hence, the residue should be washed with 4% sodium acetate till free from chromate ions.

The residue is filtered through previously weighed Gooch crucible. It is then dried upto 120^0C about an hour, and weighed as $PbCrO_4$.

Reaction

$$Pb(NO_3)_2 + K_2CrO_4 \longrightarrow PbCrO_4 + 2KNO_3$$

Yellow ppt.

$$Pb + KCrO4 \rightarrow PbCrO4$$