

RPS DEGREE COLLEGE

BALANA (MAHENDERGARH)-123029



Lab Manual

(Chemistry 3rd & 4th Semester)

Department of Chemistry

INORGANIC CHEMISTRY

Semester 3rd

Volumetric Analysis

1. Determination of acetic acid in commercial vinegar using NaOH
2. Determination of alkali content - antacid tablet using HCl
3. Estimation of calcium content in chalk as calcium oxalate by permanganometry.
4. Estimation of hardness of water by EDTA
5. Estimation of ferrous and ferric by dichromate method.
6. Estimation of copper using thiosulphate.
7. Determination of chloride ion by Mohr's Method & Volhard's method.

Semester 4th

Gravimetric Analysis

1. Aluminum as oxinate
2. Mg as $\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$
3. Ba as BaSO_4
4. Nickel as (dimethyl glyoxime)
5. Copper as thiocyanate

EXPERIMENT:-1

AIM: Determination of acetic acid in commercial vinegar using NaOH

CHEMICAL REQUIRED:-

1. ~ 2 g KHC₈H₄O₄
2. phenolphthalein indicator solution
3. Distilled water
4. Sample of commercial grade vinegar

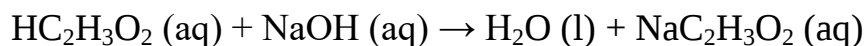
APPARATUS REQUIRED:-

50mL beaker , 100 mL volumetric flask (with cap), 50 mL burette , 250 mL Erlenmeyer flask , 25 mL pipette, Titration apparatus, scales

PROCEDURE:-

Preparation of the Potassium Hydrogen Phthalate Standard

For the titration of the vinegar in this experiment the following specific reaction will be used to calculate the acetic acid content of the vinegar sample:



1. Weigh precisely (on an electronic balance) ~ 1.5 grams of pure potassium hydrogen phthalate (KHC₈H₄O₄) into a 50mL beaker.
2. Dissolve the acid in ~50 mL of water and transfer carefully into a 100 mL volumetric flask.
3. Rinse the beaker several times with small portions of water to ensure quantitative transfer.
4. Make the volume up to the mark with distilled water, cap the flask and mix thoroughly.

This solution will be used to standardize a solution of sodium hydroxide.

Standardization of the Sodium Hydroxide Solution

Potassium hydrogen phthalate, the primary standard, reacts with sodium hydroxide.

1. Select a clean 50 mL burette, rinse it with a small portion of the sodium hydroxide solution, and fill it to just below the zero mark.
2. Read and record the initial volume to the nearest 0.01 mL.
3. Rinse a 250 mL Erlenmeyer flask with distilled water to make sure it is clean.
4. Pipette exactly 25.00 mL of the potassium hydrogen phthalate solution into the flask.
5. Add three drops of phenolphthalein indicator and titrate with the sodium hydroxide solution until a permanent colour change is first detected. The palest pink colour denotes the end point of this reaction. The colour should persist throughout the entire solution when swirled for at least 10 seconds. The colour will fade slowly on standing.
6. Repeat the titration on a second 25.00 mL aliquot of the primary standard solution. If the titration volumes do not agree within ± 0.1 mL continue to repeat the titration.
7. Report your two best titrations.

The Determination of Acetic Acid in Vinegar

The acetic acid (CH_3COOH) concentration in commercial vinegar may be easily determined by titrating a suitable sample of the vinegar with the standardized sodium hydroxide solution.

1. Pipette exactly 10.00 mL of the commercial vinegar sample into a 250 mL Erlenmeyer flask and add ~5 mL of distilled water.
2. Using three drops of phenolphthalein indicator, titrate the acetic acid with the standard base to a pale pink equivalence point. Record the burette readings.
3. Repeat the titration at least once more using a fresh aliquot of vinegar. Results should agree within ± 0.2 mL or additional titrations are required.
4. Report your two best titrations.

OBSERVATION:-

Enter your results in the table below:

Report Sheet for Assay of Acetic Acid Content of Vinegar

Data

Mass of empty 50 mL beaker: _____ g Mass of beaker and KHP:
_____ g

Standardization of NaOH

Titration Number	Initial Burette Volume (mL)	Final Burette Volume (mL)	Volume of NaOH used (mL)
1			
2			
Average Volume(mL)			

Titration of Vinegar

Titration Number	Initial Burette Volume (mL)	Final Burette Volume (mL)	Volume of NaOH used (mL)
1			
2			
Average Volume(mL)			

CALCULATIONS:-

Show your work:

1. Concentration of the KHP solution: _____ M
2. Concentration of the NaOH solution: _____ M

3. Concentration of acetic acid in the vinegar solution: _____ M

EXPERIMENT:-2

AIM: Estimation of calcium content in chalk as calcium oxalate by permanganometry.

APPARATUS REQUIRED:-

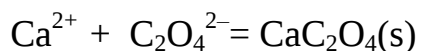
volumetric flask, 250-mL beakers, hotplate, Erlenmeyer flask, filter paper.

CHEMICAL REQUIRED:-

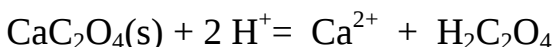
sulfuric acid, ammonium oxalate, ammonia, methyl red indicator, calcium nitrate

Theory:

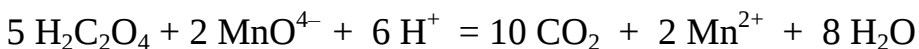
A classical and widely applied method for determination of calcium, suitable for biological material, is permanganometric titration method. The goal of this task is to determine calcium previously precipitated as calcium oxalate and quantitatively separated from solution by filtration:



The precipitate is subsequently dissolved in sulfuric acid:



The liberated oxalic acid is titrated with a standard solution of potassium permanganate:



This method is convenient for selective determination of calcium in the presence of magnesium.

Procedure

1. Fill the 100-mL volumetric flask containing the sample to the mark.

2. Prepare 100 mL of sulfuric acid with a concentration of ca. 3 mol/L starting from concentrated sulfuric acid (96%, density 1.84 g/mL). Be cautious when working with sulfuric acid.
3. Measure out two 25.00-mL aliquots of the diluted solution of the sample into 250-mL beakers using a pipette. To each of them add 10 mL of 3 mol/L sulfuric acid solution and 50 mL of water.

Hot solutions of acid are to be handled in the following steps. Take care while doing so!

4. Cover each beaker with a watch glass and carefully heat their contents on a hotplate until the solutions become hot, but not hot enough to start boiling.
5. Remove the beakers from the hotplate and carefully add solid ammonium oxalate (1.5 g) to each of them. Stir their contents until most of the ammonium oxalate has dissolved.
6. Add 5-10 drops of the methyl red indicator to each beaker, and while the solutions are still hot, increase their pH values by adding ammonia (1:1) while constantly stirring until the first color change from pink to orange. If a greater quantity of ammonia is added, the color of the solution becomes yellow. If this were to happen, add a drop of the previously prepared 3 mol/L sulfuric acid to lower the pH, and then repeat the pH adjustment with ammonia.
7. Leave the solution to stand for at least one hour for the calcium oxalate to precipitate quantitatively. Do not stir the solution during this period.
8. Filter off a majority of the solution into a 250-mL Erlenmeyer flask. Rinse the watch glass as well.
9. Determine whether the filtrate contains traces of calcium ions by transferring a drop of it onto a dark surface and adding a drop of 0.25 mol/L ammonium oxalate solution. If it becomes turbid or a precipitate forms, this means that the precipitation of calcium oxalate did not proceed quantitatively. Potential causes include inadequately adjusted solution pH or insufficient precipitation time. In this case, these parameters should be adjusted more carefully in the next aliquot trial. You should decide whether to continue working with the first aliquot.

10. If there are no calcium ions left in the filtrate, quantitatively transfer all of the precipitate onto the funnel.
11. Rinse the precipitate four times with 10 mL of water. Discard the filtrates.
12. Rinse the precipitate one more time with 10 mL of water. Test this filtrate for traces of oxalate by adding a saturated solution of calcium nitrate to a drop of the filtrate placed on a dark surface. If it becomes turbid or a white precipitate forms, carry on rinsing and testing the filtrate until you obtain one that does not contain any oxalate.
13. When the precipitate is free of oxalate, discard the filtrates.
14. Transfer the filter paper with the precipitate into a 250-mL beaker, and add 25 mL of 3 mol/L sulfuric acid solution and expose to ultrasound until complete dissolution. Decant the resulting solution into an Erlenmeyer flask, and rinse the filter paper with 25 mL water also by exposing the contents of the beaker to ultrasound. Rinse the filter paper once more with 25 mL of water, without the aid of the ultrasonic bath.
15. Heat the solution in the Erlenmeyer flask at temperature above 60 °C (white fumes must be present above the solution), and titrate the solution using a standard solution of potassium permanganate, while maintaining the temperature above 60 °C during the titration.

OBSERVATION & CALCULATIONS:-

Consumed volumes of the titrant are:

V1 = _____ mL

V2 = _____ mL

V3 = _____ mL

Determine the mass of calcium in the 100-mL sample.

The relative atomic mass of calcium is 40.08.

The concentration of the standard solution of KMnO_4 is 0.0206 mol/L.

EXPERIMENT:-3

1. **AIM:-**To Determine the percentage purity of the given sample of $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ and also determine the percentage of magnesium in it. Provided $\frac{N}{20}$ EDTA solution.(Estimation of hardness of water by EDTA)

APPARATUS REQUIRED:-

Burette, watch glass, pipette, titrating flask, beaker

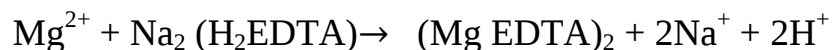
CHEMICAL REQUIRED:-

- i. $\frac{N}{20}$ Solution of $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$
- ii. $\frac{N}{20}$ EDTA (Ethylene diamine tetra-acetic acid)
- iii. Eriochrome black T indicator

THEORY:-

It is used to measure the % purity and % of Mg. for it take approximately $\frac{N}{20}$ solution of $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ is prepared by weighing 1.54 g and dissolving in 250 ml distilled water then it is titrated against $\frac{N}{20}$ solution of EDTA using Eriochrome black T indicator.

CHEMICAL REACTION:-



PROCEDURE:-

1. Preparation of approximately $\frac{N}{20}$ $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$

- i. Firstly weigh out 1.54 g of $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ accurately on a watch glass. Transfer it into 250 ml beaker and add about 50ml of distilled water.
- ii. Dissolve the solid by stirring.
- iii. Now transfer the solution into a 250 ml graduated flask.
- iv. Wash out the beaker and make up the volume by dilution with distilled water to the etched mark.

2. Titration of $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ solution against $\frac{N}{20}$ EDTA.

- i. Rinse and fill the burette with $\frac{N}{20}$ EDTA solution.
- ii. Now pipette out 20 ml of prepared $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ solution into a titration flask.
- iii. Add about 2 ml of buffer solution, 30 ml of distilled water and 2-3 drops of eriochrome black T indicator.
- iv. Now run the EDTA solution from the burette into the flask dropwise with constant shaking.
- v. Go on adding the EDTA solution till the colour changes from red to sky blue.
- vi. Repeat the titration to get a set of 3 constant reading.

OBSERVATION:-

Weight of empty watch glass = w g

Weight of watch glass + $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ = (w+1.5375)g

$\therefore$ Weight of $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ = 1.5375 g

Volume of solution made = 250ml

Volume of $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ solution taken each time = 20ml

S.No.	Initial reading of burette	Final reading of burette	Vol. of EDTA solution used in ml.
1.			
2.			
3.			
4.			

Concordant volume = Vml (say)

CALCULATIONS:-

Using normality equation

$$N_1 \times V_1 = N_2 \times V_2$$

(MgSO₄.7H₂O solution) (EDTA solution)

$$N_1, \text{ i.e., normality of MgSO}_4.7\text{H}_2\text{O solution} = \frac{N_2 V_2}{V_1}$$

$$= \frac{N}{20} \times \frac{V}{20} \times \frac{V}{400}$$

$$\therefore \text{Strength of MgSO}_4.7\text{H}_2\text{O} = \frac{V}{400} \times 123 = x \text{ g/litre (say)}$$

(where eq. wt. of MgSO₄.7H₂O is 123)

$$\text{Strength of sample (given)} = 6.15 \text{ g/litre}$$

$$\therefore 6.15 \text{ gm of the sample contains pure}$$

$$\text{MgSO}_4.7\text{H}_2\text{O} = x \text{ g}$$

$$100 \text{ gm will contain} = \frac{x}{6.15} \times 100 = y$$

$$\therefore \text{Percentage purity of MgSO}_4.7\text{H}_2\text{O sample} = y$$

$$\text{Now normality of Mg}^{+2} \text{ ions} = \text{Normality of MgSO}_4.7\text{H}_2\text{O}$$

$$\therefore \text{Strength of Mg} = \text{Normality} \times \text{Eq. wt.}$$

$$= \frac{V}{400} \times 24 = z \text{ g/litre}$$

$$\therefore 6.15 \text{ gm of MgSO}_4.7\text{H}_2\text{O contain} = z \text{ g of mg}$$

$$100 \text{ gm of MgSO}_4.7\text{H}_2\text{O will contain} = \frac{z}{6.15} \times 100 = a$$

$$\therefore \text{Percentage of Mg in the MgSO}_4.7\text{H}_2\text{O sample} =$$

a

RESULT:-

$$\% \text{ purity of } \text{MgSO}_4 \cdot 7\text{H}_2\text{O} \text{ sample} = y$$

$$\% \text{ of Mg in sample} = a$$

PRECATUTIONS:-

- i. EDTA solution should be taken in the burette because in the titration flask, the proper pH of the solution has to be maintained.
- ii. To avoid the high reactivity of the reagent with other ions, the pH of solution should be controlled by using buffer solution.
- iii. In order to avoid co-precipitation, adsorption etc. precipitation should not occur during the titration.

EXPERIMENT – 4

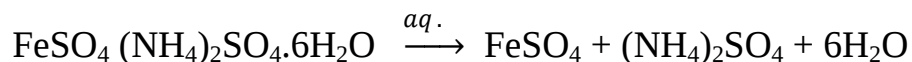
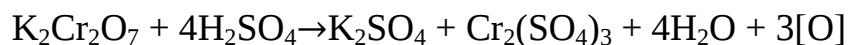
AIM:-Determine the percentage purity of given sample of Mohr's salt, 20 gm of which are present per litre of solution. Provided solid $\text{K}_2\text{Cr}_2\text{O}_7$.

APPARATUS REQUIRED:-

Burette, beaker, conical flask, glass rod

CHEMICAL REQUIRED:-

Mohr's salt, $\text{K}_2\text{Cr}_2\text{O}_7$, N-phenyl-anthracitic acid

CHEMICAL EQUATIONS:-

Indicator: N-phenyl anthranilic acid.

End point: Green to violet red.

Titration of mohr's salt against $\text{K}_2\text{Cr}_2\text{O}_7$

PROCEDURE:-

- i. Rinse and fill the burette with $\text{K}_2\text{Cr}_2\text{O}_7$ solution.

- ii. Pipette out 20ml of mohl's salt solution. Into titration flask and add about 100ml of $2\text{NH}_2\text{SO}_4$.
- iii. Add 5-10 drops of N-phenyl anthranilic acid.
- iv. Add $\text{K}_2\text{Cr}_2\text{O}_7$ solution dropwise till the colour changes from green to violet wet.
- v. Repeat the titration to get a set of three concordant readings.

OBSERVATIONS:-

Weight of empty watch glass = wg.

Weight of watch glass + solid $\text{K}_2\text{Cr}_2\text{O}_7$ = (w+0.6125)g

$\therefore$ Weight of solid $\text{K}_2\text{Cr}_2\text{O}_7$ = 0.6125g

Volume of solution made = 250ml

$\therefore$ Normality of $\text{K}_2\text{Cr}_2\text{O}_7$ solution = $\frac{\text{Strength}}{\text{Eq. wt.}} = \frac{0.6125}{49} = \frac{1}{20}$

Volume of Mohr's salt solution taken each time = 20ml

S.No.	Initial reading	Final reading	Vol. Of $\text{K}_2\text{Cr}_2\text{O}_7$ solution in ml
1.			
2.			
3.			
4.			

Concordant volume = V ml

CALCULATIONS:-

Using normality equation

$$N_1 \times V_1 = N_2 \times V_2$$

(Mohr's salt) ($\text{K}_2\text{Cr}_2\text{O}_7$ solution)

$$N_1 \times 20 = \frac{N}{20} \times V$$

$$N_1, \text{ i.e., normality of Mohr's salt solution } = \frac{V}{400}$$

$$\therefore \text{ Strength of Mohr's salt solution } = \text{ Normality } \times \text{ Eq. Wt.}$$

$$= \frac{V}{400} \times 392 = x \text{ g/litre (say)}$$

$$\therefore \text{ percentage purity of Mohr's salt } = \frac{x}{20} \times 100$$

RESULT:-

$$\% \text{ purity of Mohr's salt } = \frac{x}{20} \times 100$$

PRECAUTIONS:-

- i. Burette should be rinsed with $K_2Cr_2O_7$ solution before filling in the solution. Titration flask should be washed with distilled water after each titration.
- ii. $K_2Cr_2O_7$ solution is always to be taken in the burette and its upper meniscus is to be considered while noting the initial and final readings.

EXPERIMENT – 5

AIM:- To evaluate the value of x in $CuSO_4 \cdot xH_2O$, provided $\frac{N}{20}$ hypo solution.

(Estimation of copper using thiosulphate.)

APPARATUS REQUIRED:-

Burette, conical flask, Beaker, glass rod

CHEMICAL REQUIRED:-

$Na_2S_2O_3$, KI, starch solution

THEORY:-

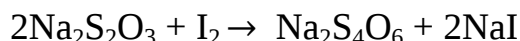
Cupric ions can be determined iodometrically. The cupric salt solution is treated with an excess of pot. Iodide. The I_2 thus liberated is titrated with a standard solution of hypo using starch solution as indicator. The value of x can be calculated by using the following relation.

$$\text{Theoretical mol. Wt. Of } CuSO_4 \cdot xH_2O = \text{Observed mol. Wt.}$$

$$159.5 + 18x = \text{Observed mol. Wt.}$$

$$X = \frac{\text{Observed mol. wt.} - 159.5}{18}$$

CHEMICAL EQUATIONS: -



Indicator: Freshly prepared starch solution.

End point: Blue to colourless with white ppt. (Hypo in burette)

PROCEDURE:-

A. Preparation of $\text{CuSO}_4 \cdot x\text{H}_2\text{O}$ solution

- i. Weight accurately 3.35g of given $\text{CuSO}_4 \cdot x\text{H}_2\text{O}$ crystals.
- ii. Dissolve them in 50ml of distilled water in a beaker.
- iii. Transfer this into 250ml graduated flask and make the volume to the etched mark by adding more of distilled water.
- iv. Stopper and shake the contents thoroughly.

B. Titration of $\text{CuSO}_4 \cdot x\text{H}_2\text{O}$ solution against hypo

- i. Rinse and fill the burette with hypo solution.
- ii. Pipette out 20ml of copper sulphate solution into the titration flask and add to it a few drops of dilute solution of sodium carbonate until a slight turbidity is obtained. Add dil. Acetic acid dropwise until the turbidity disappears.
- iii. Add about one gram of solid KI and dilute the solution with about 40ml of distilled water.
- iv. Titrate the displaced I_2 with hypo solution run from the burette.
- v. Add 2ml freshly prepared starch solution towards the end.
- vi. At the end point, the blue colour sharply changes into a white ppt. Of Cu_2I_2 .
- vii. Repeat the titration to get a set of three concordant readings.

OBSERVATIONS:-

Weight of empty watch glass = w g

Weight of watch glass + copper sulphate = (w + 3.35)g

∴ weight of copper sulphate crystals = 3.350 g

Volume of solution made = 250ml

∴ Weight of hydrated copper sulphate/litre = $\frac{3.350}{250} \times 1000 = 13.40$ g

Volume of copper sulphate solution taken each time = 20ml

S.No.	Initial reading	Final reading	Vol. Of K ₂ Cr ₂ O ₇ solution in ml
1.			
2.			
3.			
4.			

Concordant volume = V ml

CALCULATION:-

Using normality equation

$$\begin{array}{cc} N_1 \times V_1 = N_2 \times V_2 \\ (\text{CuSO}_4 \cdot x\text{H}_2\text{O}) & (\text{Hypo}) \end{array}$$

$$N_1 \times 20 = \frac{N}{20} \times V$$

$$N_1, \text{ i.e., normality of copper sulphate solution} = \frac{V}{400}$$

$$\therefore \text{Eq. Wt. Of copper sulphate} = \frac{\text{Strengt h}}{\text{Normality}}$$

$$= \frac{13.40}{V} \times 400 = E \text{ (say)}$$

$$\text{As eq. Wt. Of CuSO}_4 \cdot x\text{H}_2\text{O} = \text{Mol. Wt. Of CuSO}_4 \cdot x\text{H}_2\text{O}$$

$$\therefore \text{Mol. Wt. Of CuSO}_4 \cdot x\text{H}_2\text{O} = E$$

Theoretical mol. Wt. = Observed mol. Wt.

Or

$$159.5 + 18x = E$$

$$\therefore x = \frac{E - 159.5}{18}$$

The value of x is to be changed into the nearest whole number since the water molecules can never be in fractions.

PRECAUTIONS:-

- i. The indicator should be freshly prepared since on keeping, it is spoiled on account of bacterial attack.
- ii. When I_2 is liberated in the titration flask, the indicator must be added towards the end when the liquid becomes lemon yellow. If the indicator is added in the beginning, it will result into a permanent deep blue colour.

RESULT:-

The value of x in $CuSO_4 \cdot xH_2O$ is five _ _ _ _

EXPERIMENT:-6

Aim: Determination of chloride ion by Mohr's Method & Volhard's method.

CHEMICAL REQUIRED:-

Silver nitrate, Potassium thiocyanate, Potassium permanganate, Ferric ammonium sulfate

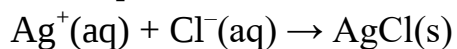
APPARATUS REQUIRED:-

boiling chips, 500 mL volumetric flask, 10 mL and 100 mL measuring cylinders, conical flasks, Bunsen burner, tripod and gauze burette and stand 50 mL pipette (if possible)

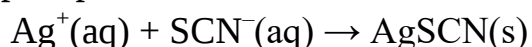
THEORY:-

This method uses a back titration with potassium thiocyanate to determine the concentration of chloride ions in a solution. Before the titration an excess volume of a silver nitrate solution is added to the solution containing chloride ions,

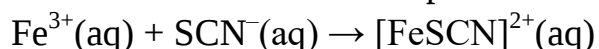
forming a precipitate of silver chloride. The term ‘excess’ is used as the moles of silver nitrate added are known to exceed the moles of sodium chloride present in the sample so that all the chloride ions present will react.



The indicator Fe^{3+} (ferric ion) is then added and the solution is titrated with the potassium thiocyanate solution. The titrate remains pale yellow as the excess (unreacted) silver ions react with the thiocyanate ions to form a silver thiocyanate precipitate.



Once all the silver ions have reacted, the slightest excess of thiocyanate reacts with Fe^{3+} to form a dark red complex.



The concentration of chloride ions is determined by subtracting the titration findings of the moles of silver ions that reacted with the thiocyanate from the total moles of silver nitrate added to the solution. This method is used when the pH of the solution, after the sample has been prepared, is acidic. If the pH is neutral or basic, Mohr’s method or the gravimetric method should be used. The method is illustrated below by using the procedure to determine the concentration of chloride (from sodium chloride) in cheese.

PROCEDURE:-

Sample Preparation

The salt sodium chloride is added during the manufacture of cheddar cheese. In this method, the cheese is ‘digested’ to release this salt to obtain the concentration of chloride ions. To carry out this digestion, the cheese is reacted with nitric acid and potassium permanganate. The chloride ions are then ‘free’ to form a precipitate with the added silver ions.

1. Cut or grate the cheese into fine pieces and accurately weigh about 6 g into a 500 mL conical flask.
2. Precisely add 50 mL of 0.1 mol L⁻¹ silver nitrate solution (by pipette if possible), 20 mL of concentrated nitric acid, (very carefully – see safety notes), 100 mL of distilled water and a few boiling chips, and heat the solution to boiling in a fumehood.

3. As the solution boils add 5 mL of 5% potassium permanganate solution. This addition will cause a very smelly reaction so done in the fumehood. Keep boiling until the purple colour disappears, then add another 5 mL of potassium permanganate solution. Continue this process until 30 mL of potassium permanganate solution has been added and the cheese particles are completely digested (or as close as possible). To find out when digestion is complete, remove the flask from heat and allow it to stand for a few moments. Undigested cheese particles will float upon the surface of the clear liquid, while the white precipitate of silver chloride will sink to the bottom. If there is still too much undigested cheese, the boiling and addition of 5 mL of potassium permanganate should be continued, checking each time until there is a satisfactory level of digestion.
4. Cool the solution and filter it. Wash the solid residue with a few mL of distilled water.
5. Make the filtrate up to 500 mL in a volumetric flask.

Titration

1. Use a volumetric cylinder to measure 100 mL of the cheese extract solution (be as precise as possible) and pour it into a conical flask.
2. Add 1 mL of saturated ferric ammonium sulfate solution as indicator.
3. Titrate the unreacted silver ions with the 0.1 mol L⁻¹ potassium thiocyanate solution. The end point is the first appearance of a dark red colour due to the ferric thiocyanate complex (figure 1).
4. Repeat the titration with 100 mL samples of the cheese extract solution until you obtain concordant results (titres agreeing within 0.1 mL).

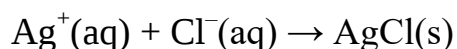
Result Calculations

1. Determine the average volume of potassium thiocyanate used from your concordant titres.
2. Calculate the moles of potassium thiocyanate used.
3. Use the equation of the reaction between silver ions and thiocyanate ions $\text{Ag}^+(\text{aq}) + \text{SCN}^-(\text{aq}) \rightarrow \text{AgSCN}(\text{s})$ to calculate the moles of unreacted silver nitrate

in 100 mL of cheese extract, and multiply the figure by five to determine the total moles of unreacted silver nitrate (the excess) in the 500 mL volumetric flask.

4. Calculate the moles of silver nitrate in the 50 mL of solution that was added during the sample preparation to the cheese.

5. Calculate the total moles of silver nitrate that reacted with the salt from the cheese by subtracting the moles of unreacted silver nitrate (the excess) from the total moles of silver nitrate added to the cheese. 6. Use the equation of the reaction between the silver ions and the chloride ions to calculate the moles of sodium chloride in the sample of cheese.



7. Calculate the concentration of sodium chloride in the cheese as grams of salt per 100 g cheese (% salt).

Semester 4th

EXPERIMENT:-1

AIM:-Determination of Aluminum as aluminum 8-Hydroxy quinoline or oxinate.

CHEMICAL REQUIRED:-

Potash alum, concentrated hydrochloric acid, Acetic acid, Ammonium acetate, 8-Hydroxyquinoline, Distilled water

APPARATUS REQUIRED:-

Beaker 250 ml, Bunsen burner, Desiccator, Filtration apparatus, Glass rod, Pair of tongs, Rubber policeman, Sintered glass crucible, Tripod stand, Wash bottle, Watch glass, Water bath

PROCEDURE:-

1. Weigh out accurately about 0.5 g of potash alum from a weighing bottle into a 400 ml beaker.
2. Dissolve in 20-25 ml of distilled water. Alternatively, take 25 ml of the solution prepared by our counsellor.
3. Add 1 ml of conc. HCl and dilute the solution to 150 ml. Add 5-6 ml of 8-hydroxyquinoline (oxine) reagent (a 10% solution in 20% acetic acid) and heat to 70-80°C. Slowly add 25 ml of 2M ammonium acetate solution dropwise and with constant stirring to ensure complete precipitation. If the supernatant liquid is yellow to orange in color, it means enough 8-hydroxyquinoline has been added.
4. Heat the contents on a boiling water bath for half an hour so that the precipitate becomes granular and of easily filterable form. Allow to cool.
5. Filter the precipitate through a constant weight sintered glass crucible of porosity G4. Wash the precipitate first with hot water and then with cold water. Continue washing till the filtrate is almost colourless.
6. Dry the precipitate at 130-140°C in an electric drying oven. Cool in a desiccator and weigh as $\text{Al}(\text{C}_9\text{H}_6\text{NO})_3$.
7. Repeat the process of heating, cooling and weighing till the weight becomes constant.

Observations:-

Weight of potash alum

- i) Weight of empty weighing bottle =----- g
- ii) ii) Weight of weighing bottle + potash alum =----- g
- iii) iii) Weight of weighing bottle after transferring= -----g potash alum

Weight of sintered crucible

- I) 1st weight of sintered crucible = -----g
- II) 2nd weight of sintefed crucible = -----g

Weight of sintered glass crucible + precipitate

- 1. 1st weight of crucible + precipitate = -----g
- 2. 2nd weight of crucible + precipitate =-----g

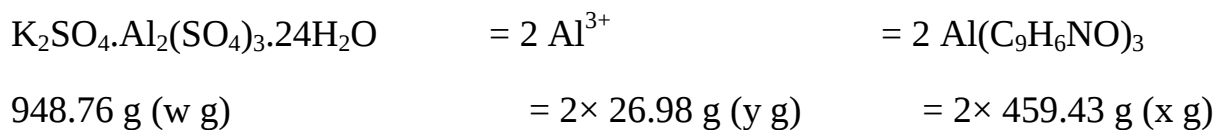
Calculations:

Calculate the percentage of aluminium ions in potash alum as follows:

Weight of potash alum taken for experiment = ii) - iii) g = w g

Weight of $\text{Al}(\text{C}_9\text{H}_6\text{NO})_3$ formed = vii) - v) g = x g

You know that one mole of potash alum ($\text{K}_2\text{SO}_4 \cdot \text{Al}_2(\text{SO}_4)_3 \cdot 24\text{H}_2\text{O}$) contains two moles of aluminium ions which are converted into two moles of aluminium (III) 8-hydroxyquinolate. Thus,



Hence, weight of Al^{3+} ions in x g of $\text{Al}(\text{C}_9\text{H}_6\text{NO})_3$,

$$y = \frac{26.98}{459.43} \times x \text{ g}$$

Now this y g of Al^{3+} ions are present in w g of the potash alum taken for analysis.
Hence, percentage of Al^{3+} ions in potash alum

$$\% \text{Al}^{3+} = \frac{y}{w} \times 100$$

Compare this value with the theoretical percentage of Al^{3+} ions in potash alum.

If you have performed the experiment carefully, the experimental and the calculated values should agree within 0.5% with each other. Following a similar procedure, you can calculate the percentage of Al^{3+} ions in any other substance.

Result:

You can report your result in any one of the following forms as instructed by your counsellor.

Weight of $Al(C_9H_6NO)_3$ formed = -----g

Percentage of aluminium ions in potash alum = ----%

EXPERIMENT:-2

Aim:- To estimate the amount of barium in the whole of the given solution of barium chloride.

CHEMICAL REQUIRED:-

barium chloride, 2N HCl, 4N H_2SO_4 ,

APPARATUS REQUIRED:-

250 mL beaker , 100 mL flask, 20 mL pipette, ashless filter paper, Bunsen burner

Theory: -

The given barium chloride solution is made up to a definite volume. A measured volume of it is then treated with dilute sulphuric acid and then treated with dilute sulphuric acid and barium precipitated as barium sulphate.



The precipitated barium sulphate is separated and weighed. The mass of Barium in the whole of the given solution is calculated knowing that 233.36 g of barium sulphate contains 137.36 g of barium.

Procedure: -

1. The given barium chloride solution is made up to 100mL in a standard flask.
2. 20mL of solution is pipetted into a 250 mL beaker.
3. About 5mL 2N HCl is added and diluted to 150mL with distilled water.
4. The solution is heated to boiling and a hot solution of 4N H_2SO_4 (10 - 15mL) is added drop by drop with constant stirring, till the precipitation is complete.
5. The solution containing the precipitate is heated in a water bath for 5 minutes.
6. The precipitate is allowed to stand for an hour.
7. The clear solution is decanted through an ashless filter paper (whatman No. 40).
8. The precipitate is washed with hot distilled water to free sulphate ions.
9. The particles adhering to the sides of the beaker and glass rod are removed by a policeman.
10. Finally the precipitate is washed once again.
11. The dried filter paper is folded and placed in a crucible which has been previously weighed.
12. The filter paper with the precipitate is first incinerated on a Bunsen burner by a low flame and then transferred to an electric burner.
13. The crucible is transferred to desiccator and cooled.
14. When cold, the crucible is weighed.
15. Heating, cooling and weighing are repeated till concordant values are obtained.

Calculations:

Mass of crucible + liquid = a g

Mass of crucible + liquid + Barium sulphate = b g

Mass of Barium sulphate = (b-a) g.

233.36 of barium sulphate contain 137.36 g of barium.

Mass of barium in (b-a) g of Bariumsulphate =

Therefore, Mass of Barium in the whole of the given solution = -----g

RESULT:

Mass of Barium in the whole of the given solution = ----- g.

EXPERIMENT:-3

Aim:- Find out gravimetrically the percentage purity of $\text{NiSO}_4 \cdot 7\text{H}_2\text{O}$, 20.0g of which has been dissolved per litre.

CHEMICAL REQUIRED:-

1. One % dimethyl glyoxime solution in alcohol
2. Aqueous ammonia solution
3. Solution of nickel salt

APPARATUS REQUIRED:-

Pipette, glass rod, watch glass, beaker, sintered glass crucible

PROCEDURE:-

1. Take a 400 ml beaker add into it 20 ml of given nickel salt solution with the help of pipette.

2. Add 5 ml of HCl and dilute the mixture by adding nearly 200 ml distilled water. Heat the solution to 70 – 80°C
3. Place the beaker on an asbestos sheet and add slowly 30 ml of 1% alcoholic DMG solution with constant stirring.
4. After it adds immediately ammonia solution with constant stirring till it gives ammoniacal smell.
5. Now keep the beaker undisturbed for 45 minutes approx. Then digest the precipitates on a water bath for 30 minutes.
6. Cool the solution and check for complete precipitation by adding two drops of DMG.
7. Now filter the precipitates using G-3 crucible using a suction pump.
8. Wash the precipitates with small lots cold water several times.
9. Dry the crucible containing precipitates in an electric oven to constant weight.

OBSERVATION:-

Vol. of given solution taken for precipitation = 20 ml

Strength of given nickel sulphate solution = 20.0 gms/litre

Weight of sintered glass crucible before experiment i.e., empty = W_1 gms

Weight of sintered glass crucible and nickel dimethyl glyoxime = W_2 gms

Weight nickel dimethyl glyoxime formed = $W_2 - W_1 = W$ gms

CALCULATIONS:-

- I. From 20 ml of given solution weight of nickel dimethyl glyoxime formed = W gms

From 1 ml of given solution weight of nickel dimethyl glyoxime formed
 $= \frac{W}{20}$

From 1000 ml of given solution weight of nickel dimethyl glyoxime formed
 $= \frac{W}{20} \times 1000 = 50 W \text{ gms}$

- II. $\text{Ni}(\text{C}_4\text{H}_7\text{O}_2\text{N}_2)_2 = \text{NiSO}_4 \cdot 7\text{H}_2\text{O}$

288.7 gms of nickel dimethyl glyoxime is obtained from $\text{NiSO}_4 \cdot 7\text{H}_2\text{O} =$
 280.7 gms

1 gm of nickel dimethyl glyoxime is obtained from $\text{NiSO}_4 \cdot 7\text{H}_2\text{O} = \frac{280.7}{288.7}$

50 w gms of nickel dimethyl glyoxime is obtained from $\text{NiSO}_4 \cdot 7\text{H}_2\text{O}$
$$= \frac{280.7}{288.7} \times 50 \text{ W} = a \text{ g/litre (say)}$$

III. In 20.0 gms of impure sample of $\text{NiSO}_4 \cdot 7\text{H}_2\text{O}$, actual amount of $\text{NiSO}_4 \cdot 7\text{H}_2\text{O}$ present = a g

In 1 gm of impure sample of $\text{NiSO}_4 \cdot 7\text{H}_2\text{O}$ actual amount of $\text{NiSO}_4 \cdot 7\text{H}_2\text{O}$ present = $\frac{a}{20}$

In 100gms of impure sample of $\text{NiSO}_4 \cdot 7\text{H}_2\text{O}$ present = $\frac{a}{20} \times 100 = 5a$

RESULT:-

Percentage of purity of $\text{NiSO}_4 \cdot 7\text{H}_2\text{O}$

Sample = 5a%

PRECAUTIONS:-

1. Excess addition of precipitant is avoided, so that precipitant may not get separated along with the precipitates.
2. To avoid decomposition of precipitates the drying should not be carried out above 120°C .

EXPERIMENT- 4

AIM: - Find out gravimetrically the percentage of Cu in given solution of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$, 20g of which has been dissolved per litre.

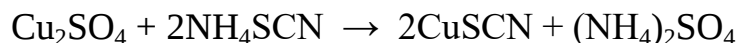
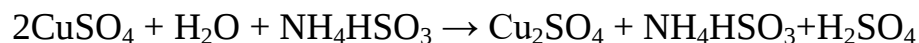
APPARATUS REQUIRED:-

Beaker, watch glass, pipette, glassrod.

CHEMICAL REQUIRED:-

1. Ammonium thiocyanate solution.
2. Saturated solution of NH_4HSO_3 .
3. Solution of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$.

REACTIONS:-



OBJECTIVE:-

In this experiment firstly, cupric salt is reduced to cuprous salt either by using sulphurous acid or ammonium bisulphate solution. Then cuprous ions are precipitated by ammonium thiocyanate (NH_4SCN). After then precipitates formed are filtered, washed dried and weighed using sintered glass crucible. From the weight of precipitates formed percentage of copper is determined.

PROCEDURE:-

1. Take 20 ml of given solution of copper sulphate in a clean 400 ml beaker.
2. Add few drops of HCl, followed by addition of 25 ml of 10% NH_4HSO_3 Solution.
3. Dilute the above solution by adding 150 ml of distilled water. Boil the solution.
4. Now put the beaker on an asbestos sheet, and then add drop wise 10% ammonium thiocyanate solution with constant stirring till the supernatant solution becomes colourless. (To check for complete precipitation add few drops more of ammonium thiocyanate).
5. Cover the beaker and allow the contents to stand for 2-3 hours preferably overnight.
6. Filter the precipitates using G-4 crucible, wash the precipitates using 1% cold dilute solution of NH_4HSO_3 till the filtrate is free from SCN^- ions.
7. Finally wash the precipitates with 20% alcohol to remove NH_4SCN .
8. Heat the crucible in oven at $110^\circ - 120^\circ\text{C}$ to constant weight.

OBSERVATIONS:-

Volume of given solution = 20ml

Strength of given Copper sulphate solution = 20 g/litre

Weight of sintered glass crucible Before experiment i.e., empty = $W_1\text{g}$

Weight of sintered glass crucible

and $\text{Cu}_2(\text{SCN})_2 = W_2\text{g}$

Weight of $\text{Cu}_2(\text{SCN})_2$ formed = $w_2 - w_1 = W\text{g}$

CALCULATIONS:-

I. From 20 ml of given solution weight $\text{Cu}_2(\text{SCN})_2$ formed = Wg

$$\text{From 1 ml of given solution weight} = \frac{W}{20}$$

$$\text{From 1000 ml given solution weight} = \frac{W}{20} \times 1000 = 50 W \text{ gm}$$

II. $\text{Cu}_2(\text{SCN})_2 = 2 \text{ cu}$

$$243 \text{ gm } \text{Cu}_2(\text{SCN})_2 \text{ is formed from copper} = 127 \text{ gm}$$

$$1 \text{ gm } \text{Cu}_2(\text{SCN})_2 \text{ is formed from copper} = \frac{127}{243}$$

$$50w \text{ gm of } \text{Cu}_2(\text{SCN})_2 \text{ is formed from copper} = \frac{127}{243} \times 50 w = a \text{ gm} - \text{litre (say)}$$

III. In 20.0n gms of copper sulphate, actual amount of Cu present = a gms

In 1 gms of copper sulphate,

$$\text{Actual amount of Cu present} = \frac{a}{20}$$

In 100 gms of copper sulphate,

$$\text{Actual amount of Cu present} + \frac{a}{20} \times 100 = 5a$$

RESULT:-

$$\text{Percentage of Cu} = 5a$$

PRECAUTIONS:-

1. Precipitate i.e. NH_4SCN should not added in excess to avoid solubility of $\text{Cu}_2(\text{SCN})_2$ as complex ion
2. Washing of the precipitates is to be done with dilute solution of NH_4HSO_3 to avoid oxidation of Cu(I) to Cu (II)