BGS SCIENCE ACADEMY & RESEARCH CENTER
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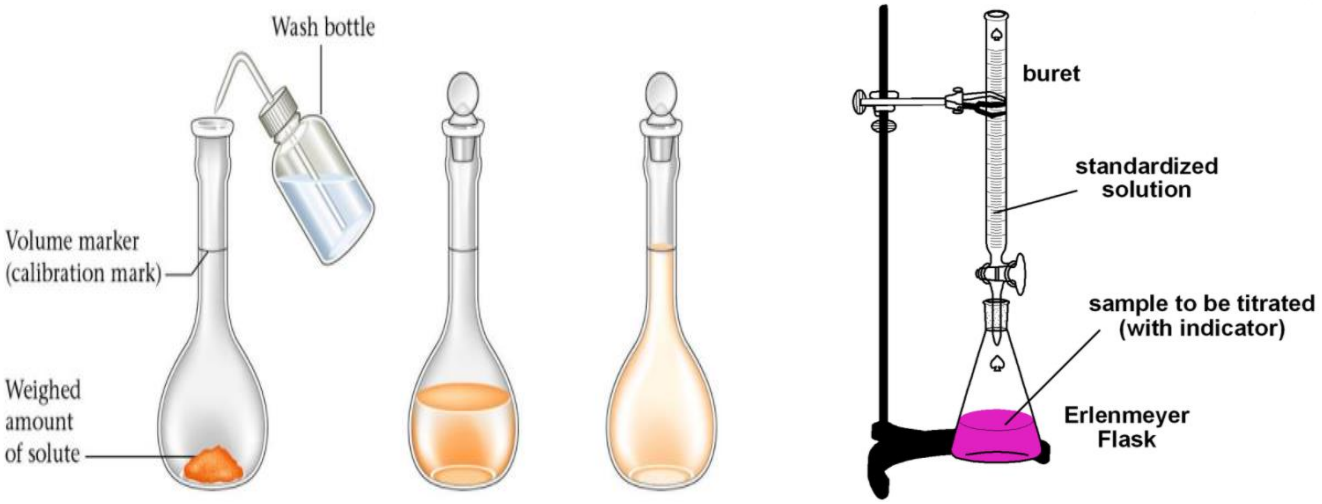
I Semester B.Sc.,
Chemistry Laboratory Manual



CONTENTS

OBSERVATIONS

Laboratory temperature: ……^{0}C

Density of water at laboratory temperature (d): ……g/cm^{3}

Tabulations:

Glassware	Volume(**cm^{3}**) = v	Mass(g) = m	Calibrated volume(**cm^{3}**) = m/d
Pipette	10		
	20		
	25		
Standard flask	25		
	50		
Burette	10		
	50		

EXPERIMENT NO. 1

AIM: **CALIBRATION OF GLASS WARES:**

1. **PIPETTE**
2. **BURETTE**
3. **VOLUMETRIC FLASK**

Calibration of commercially available glass wares like pipettes, burettes and volumetric flasks has to be performed for accuracy of the qualitative measurements. The above said types of volumetric glass wares are calibrated by finding the exact weight of the volumes they measure out by using an accurate analytical balance. Wipe the exterior of weighing beaker perfectly before inserting on the pan of the analytical balance.

The results are tabulated and the exact volume of each type of glass ware is recorded by dividing the mass of water measured out by the density of water (d) at the laboratory temperature.

OBSERVATIONS

REACTION:

$$2KMnO_4 + 5Na_2C_2O_4 + 8H_2SO_4 \rightarrow K_2SO_4 + 2MnSO_4 + 5Na_2SO_4 + 8H_2O + 10CO_2$$

PART –I: Preparation of standard sodium oxalate solution:

1. Mass of empty weighing bottle + sodium oxalate crystals (m_1) = ... g
2. Mass of empty weighing bottle (m_2) =..... g
3. Mass of sodium oxalate (m_1-m_2) = ... g

Normality of sodium oxalate = $\frac{(m_1 - m_2) \times 4}{67}$

=… ..... N

PART-II: Estimation of $KMnO_4$:

Trial no.	1	2	3
Final burette reading			
Initial burette reading			
Vol. of $KMno_4$ run down in cm^3			

$(V_2 N_2)_{KMnO4} = (V_1 N_{1)})_{Na_2C_2O_4}$

Normality of $KMnO_4$ $(N_2) = (V_1 N_1)_{Na_2C_2O_4} / (V_2)_{KMnO4}$

Normality of $KMnO_4$ (N_2) = 25 x $N_1 / (V_2)_{KMnO4}$

= ..... N

Mass of $KMnO_4$ crystals / liter of its solution: = N_2 X gm. equivalent weight of $KMnO_4$ (31.6)

Mass of $KMnO_4$ crystals present in 250 cm^3 of its solution = N_2 X 31.6 / 4 = ……

=… ..... g

EXPERIMENT NO. 02

AIM: ESTIMATION OF POTASSIUM PERMANGANATE USING STANDARD SODIUM OXALATE SOLUTION

PROCEDURE:

Part I: Preparation of standard solution of sodium oxalate

1.675 g of sodium oxalate crystals is accurately weighed and dissolved in distilled water and the solution is made up to the mark in 250 cm^3 standard flask. It is mixed well for uniform concentration. The normality of sodium oxalate is calculated.

Part II: Estimation of Potassium permanganate using std.sodium oxalate solution.

25 cm^3 of the made up sodium oxalate solution is pipetted out into a clean conical flask. Two test tubes of dilute sulphuric acid are added to it. The contents of the flask are heated on a Bunsen burner nearly to boiling. The hot solution is titrated against potassium permanganate solution taken in the burette slowly with drop wise addition until a permanent pale pink colour is obtained. The titration is repeated for concordant values.

RESULT:

Mass of potassium permanganate crystals present in 250 cm^3 of given solution is.....g.

OBSERVATIONS

REACTIONS:

$$K_2Cr_2O_7 + 7H_2SO_4 + 6FeSO_4 \rightarrow K_2SO_4 + Cr_2(SO_4)_3 + 3Fe_2(SO_4)_3 + 7H_2O$$

$$2K_3Fe(CN)_6 + 3FeSO_4 \rightarrow Fe_3[Fe(CN)_6]_2 + 3K_2SO_4$$

Part I: Preparation of standard potassium dichromate solution

1. Mass of the empty weighing bottle + potassium dichromate crystals(m_1) = …….g
2. Mass of the empty weighing bottle with the remaining (m_2) =..... g
3. Mass of potassium dichromate crystals transferred (m_1-m_2) =..... g

Normality of $K_2Cr_2O_7$ (N_1) = (m_1-m_2)x4**/**49 = ... 4**/**49
=..... N

Part II: Estimation of ferrous ammonium sulphate Burette:

0.1N $K_2Cr_2O_7$ solution
Conical flask: 25 cm^3 of FAS soln.+ 2 test tubes of dil.H_2SO_4 + 1t.t of water
Indicator: Potassium ferricyanide on paraffin paper
End point: A drop of solution tested fails to turn deep blue

Trial no.	1	2	3
Final burette reading			
Initial burette reading			
Vol. of $K_2Cr_2O_7$ run down in cm^3			

$(V_2N_2)_{FAS} = (V_1N_1)_{K_2Cr_2O_7}$

Normality of FAS (N_2) = ... N_1**/** 25

Mass of **Ferrous ammonium sulphate** crystals in 250 cm^3 of its solution

= N_2 x gram eq.wt of FAS **/**4

=.....392**/**4
=.....g

EXPERIMENT NO. 03

AIM: **ESTIMATION OF FERROUS AMMONIUM SULPHATE USING STANDARD POTASSIUM DICHROMATE SOLUTION ANDPOTASSIUM FERRICYANIDE AS EXTERNAL INDICATOR**

PROCEDURE:

Part I: **Preparation of standard solution of Potassium dichromate**

1.225 g of potassium dichromate crystals is accurately weighed and dissolved in distilled water and the solution is made up to the mark in 250 cm^3 standard flask. It is mixed well for uniform concentration. The normality of $K_2Cr_2O_7$ is calculated.

Part II: Estimation of Ferrous ammonium sulphate using standard potassium dichromate solution.

The given ferrous ammonium sulphate solution in 250 cm^3 standard flask is made up to the mark and shaken well. 25 cm^3 of the made up solution is pipetted out into a clean conical flask. Two test tubes of dil.sulphuric acid, one test tube of distilled water are added to it. The resulting mixture is titrated versus the standard potassium dichromate solution using potassium ferricyanide as external indicator. The end point is the disappearance of the blue colour, when a drop of the solution is brought in contact with the indicator droplet placed on a paraffin paper.The titration is repeated for concordant values.

RESULT: Mass of Ferrous ammonium sulphate crystals present in 250 cm^3 of given solution is …… g

OBSERVATIONS

REACTION:

$$K_2Cr_2O_7 + 7H_2SO_4 + 6FeSO_4 \rightarrow K_2SO_4 + Cr_2(SO_4)_3 + 3Fe_2(SO_4)_3 + 7H_2O$$

Part I: Preparation of standard potassium dichromate solution

1. Mass of the empty weighing bottle + potassium dichromate crystals (m_1) = ……..g
2. Mass of the empty weighing bottle with the remaining (m_2) =..... g
3. Mass of potassium dichromate crystals transferred (m_1-m_2) = …… g

Normality of $K_2Cr_2O_7$ (N_1) = (m_1-m_2) x 4**/**49 = ... 4**/**49

=..... N

Part II: Estimation of ferrous ammonium sulphate Burette:

0.1N $K_2Cr_2O_7$ solution
Conical flask: 25 cm^3 of FAS soln.+ 2test tubes of dil.H_2SO_4+ 1t.t of water + ½ t.t H_3PO_4
Indicator: Diphenyl amine
End point: Green to violet

Trial no.	1	2	3
Final burette reading			
Initial burette reading			
Vol. of $K_2Cr_2O_7$ run down in cm^3			

$(V_2N_2)_{FAS} = (V_1N_1)_{K_2Cr_2O_7}$

Normality of FAS (N_2) = ... N_1/ 25

=

Mass of **ferrous ammonium sulphate** crystals in 250 cm^3 of its solution

= N_2 x gram eq.wt of FAS **/** 4

=..... 392/4

=… ..... g

EXPERIMENT NO. 4

AIM: **ESTIMATION OF FERROUS AMMONIUM SULPHATE USING STANDARD POTASSIUM DICHROMATE SOLUTION AND DIPHENYL AMINE AS INTERNAL INDICATOR**

PROCEDURE:

Part I: **Preparation of standard solution of Potassium dichromate**

1.225 g of potassium dichromate crystals is accurately weighed and dissolved in distilled water and the solution is made up to the mark in 250 cm^3 standard flask. It is mixed well for uniform concentration. The normality of $K_2Cr_2O_7$ is calculated.

Part II: Estimation of Ferrous ammonium sulphate using standard potassium dichromate solution.

The given ferrous ammonium sulphate solution in 250 cm^3 standard flask is made up to the mark and shaken well. 25 cm^3 of the made up solution is pipetted out into a clean conical flask. Two test tubes of dil.sulphuric acid, ½ test tube of phosphoric acid, one test tube of distilled water and 2-3 drops of diphenyl amine indicator are added. The resulting mixture is titrated versus the prepared standard potassium dichromate solution till the appearance of deep violet colour.The titration is repeated for concordant values.

RESULT: Mass of Ferrous ammonium sulphate crystals present in 250 cm^3 of given solution is …… g

OBSERVATIONS

REACTIONS:

$K_2Cr_2O_7 + 7H_2SO_4 + 6KI \rightarrow 4K_2SO_4 + Cr_2(SO_4)_3 + 3I_2 + 7H_2O$

$2Na_2S_2O_3 + I_2 \rightarrow 2NaI + Na_2S_4O_6$(Colourless)

Part I: Preparation of standard potassium dichromate solution

1. Mass of the empty weighing bottle + potassium dichromate crystals(m_1) = ... g
2. Mass of the empty weighing bottle with the remaining (m_2)……. = ... g
3. Mass of potassium dichromate crystals transferred (m_1-m_2)..... = ... g

Normality of $K_2Cr_2O_7$ (N_1) = (m_1-m_2)x4/49 = ... 4/49

=..... N

Part II: Estimation of Sodium thiosulphate using standard potassium dichromate solution.

Burette: $Na_2S_2O_3$ solution

Conical flask: 25 cm^3 of $K_2Cr_2O_7$ soln.+1**/**4test tube of con.HCl + 1t.t of KI

Indicator: Freshly prepared starch

End point: Disappearance of blue colour

Trial no.	1	2	3
Final burette reading			
Initial burette reading			
Vol.of $Na_2S_2O_3$ run down in cm^3			

$(V_2N_2)_{Na_2S_2O_3} = (V_1N_1)_{K_2Cr_2O_7}$

Normality of Sod.thiosulphate (N_2) = 25 x N_1**/** V_2

= .....N

Mass of sodium thiosulphate crystals in 250 cm^3 of its solution= N_2 x gm eq.wt of $Na_2S_2O_3$/4

= 248/4

=…..... g

EXPERIMENT NO. 05

AIM: **ESTIMATION OF SODIUM THIOSULPHATE USING STANDARD POTASSIUM DICHROMATE SOLUTION**

PRINCIPLE: Potassium dichromate liberates iodine from KI quantitatively. Iodine liberated in the reaction is the measure of the concentration of potassium dichromate used, it is titrated against hypo solution using starch an internal indicator near the end point.

PROCEDURE:

Part I: **Preparation of standard solution of potassium dichromate:**

1.225 g of potassium dichromate crystals is accurately weighed and dissolved in distilled water and the solution is made up to the mark in 250 cm^3 standard flask. It is mixed well for uniform concentration. The normality of $K_2Cr_2O_7$ is calculated.

Part II: Estimation of Sodium thiosulphate using standard potassium dichromate

The given sodium thiosulphate solution is made up to the mark using distilled water and shaken well. 25 cm^3 of the prepared potassium dichromate solution solution is pipetted out into a clean conical flask. One test tube of KI is added followed by adding 1/4 test tube of con.HCl and a test tube of distilled water. The reaction mixture is shaken and kept aside for one minute. The liberated iodine solution is titrated against the made up sodium thiosulphate solution taken in the burette till a pale yellow colour is obtained 2-3 drops of the freshly prepared starch indicator is added, the solution turns blue. The blue solution is titrated against the same hypo solution till green colour is obtained or blue colour disappears. The titration is repeated for concordant values.

RESULT:

Mass of Sodium thiosulphate crystals present in 250 cm^3 of given solution is……g.

OBSERVATIONS

REACTIONS:

$K_2Cr_2O_7 + 7H_2SO_4 + 6KI \rightarrow 4K_2SO_4 + Cr_2(SO_4)_3 + 3I_2 + 7H_2O$

$2Na_2S_2O_3 + I_2 \rightarrow 2NaI + Na_2S_4O_6$ **(Colourless)**

Part I: Preparation of standard potassium dichromate solution and standardization of using standard potassium dichromate solution

1. Mass of the empty weighing bottle + potassium dichromate crystals(m_1) = …….g
2. Mass of the empty weighing bottle with the remaining (m_2)……. =..... g
3. Mass of potassium dichromate crystals transferred (m_1-m_2)..... =..... g

Normality of $K_2Cr_2O_7$ (N_1) = (m_1-m_2)x4/49 = ...4/49

=..... N

Burette: $Na_2S_2O_3$ solution

Conical flask: 25 cm^3 of $K_2Cr_2O_7$ soln.+ 1/4 test tube of con.HCl + 1t.t of KI

Indicator: Freshly prepared starch

End point: Disappearance of blue colour

Trial no.	1	2	3
Final burette reading			
Initial burette reading			
Vol.of $Na_2S_2O_3$ run down in cm^3			

$(V_2N_2)_{Na_2S_2O_3} = (V_1N_1)_{K_2Cr_2O_7}$

Normality of Sod.thiosulphate (N_2) = 25 x N_1**/** V_2

Part II: Estimation of Iodine

Burette: Standardised $Na_2S_2O_3$ solution

Conical flask: 25 cm^3 Iodine soln.+1test tube of water + 1t.t of KI

Indicator: Freshly prepared starch

End point: Disappearance of blue colour

Trial no.	1	2	3
Final burette reading			
Initial burette reading			
Vol.of $Na_2S_2O_3$ run down in cm^3			

$(V_3N_3)_{I_2} = (V_2N_2)_{Na_2S_2O_3}$

Normality of Iodine (N_3) = …… x N_1/ 25 = ... N

Mass of Iodine crystals in 250 cm^3 of its solution= N_2 x g.eq.wt of I_2**/**4 = ...127/4

=.....g

EXPERIMENT NO.06

AIM: **ESTIMATION OF IODINE USING SODIUM THIOSULPHATE AND STANDARD POTASSIUM DICHROMATE SOLUTION**

PRINCIPLE: Potassium dichromate liberates iodine from KI quantitatively. The liberated iodine is measure of the concentration of potassium dichromate used, it is titrated against hypo solution using starch an internal indicator at the end point.

PROCEDURE:

Part I: **Preparation of standard solution of potassium dichromate and standardization of sodium thiosulphate solution**

1.225 g of potassium dichromate crystals is accurately weighed and dissolved in distilled water and the solution is made up to the mark in 250 cm^3 standard flask. It is mixed well for uniform concentration. The normality of $K_2Cr_2O_7$ is calculated.25 cm^3 of the prepared potassium dichromate solution solution is pipetted out into a clean conical flask. One test tube of KI is added followed by adding 1/4 test tube of con.HCl and a test tube of distilled water. The reaction mixture is shaken and kept aside for one minute. The liberated iodine solution is titrated against the given sodium- thiosulphate solution taken in the burette till a pale yellow colour is obtained. 2-3 drops of the freshly prepared starch indicator is added, the solution turns blue. The blue solution is titrated against the same hypo solution till green colour is obtained or blue colour disappears. The titration is repeated for concordant values.

Part II: Estimation of Iodine:

25 cm^3 of the given iodine solution is pipetted out into a clean conical flask followed by one test tube of KI and one test tube of water. The solution is titrated versus the standardized sodium thiosulphate solution using starch as an indicator till blue colour disappears near the end point. The titration is repeated for concordant values.

RESULT: Mass of Iodine crystals present in 250 cm^3 of given solution =……. g.

OBSERVATIONS

REACTIONS:

$$CaOCl_2 + 2CH_3COOH + 2KI \rightarrow CaCl_2 + 3CH_3COOK + 3I_2 + H_2O$$

$$2Na_2S_2O_3 + I_2 \rightarrow 2NaI + Na_2S_4O_6 \text{(Colourless)}$$

Burette: 0.1N $Na_2S_2O_3$ solution
Conical flask: 25 cm^3 of bleaching powder soln.+1/ 4test tube of glacial.acetic.acid +1/2t.t of KI + one test tube of distilled water
Indicator: Freshly prepared starch
End point: Disappearance of blue colour

Trial no.	1	2	3
Final burette reading			
Initial burette reading			
Vol.of $Na_2S_2O_3$ run down in cm^3			

$(V \times N)_{\text{bleaching powder}} = (V \times N)_{\text{hypo}}$ Normality

of bleaching powder = 0.1x…./25

= .....N

Mass or amount of chlorine in 250cm^3 = 35.5x N/4 = 35.5 x ...../4
=… ..... g(w)

The mass of bleaching powder dissolved is ‘m’ g

Hence percentage of available chlorine in the bleaching powder = w x 100/m

= ..... x 100/m
=…....(z)

EXPERIMENT NO. 07

AIM: **DETERMINATION OF THE PERCENTAGE OF AVAILABLE CHLORINE IN THEGIVEN SAMPLE OF BLEACHING POWDER**

PRINCIPLE: Available chlorine in a sample of bleaching powder is estimated volumetrically. A known mass of bleaching powder as a suspension of water is treated with excess of potassium iodide solution. The solution is then acidified with a strong solution of acetic acid. The liberated iodine is titrated against standard sodium thiosulphate solution using starch as indicator.

PROCEDURE: About 2g of bleaching powder is weighed and dissolved in concentrated hydrochloric acid and the solution is made up to 250 ml using distilled water in a standard flask. 25 cm^3 of the made up solution is pipetted out into a clean conical flask followed by one test tube of distilled water, half test tube of 10% KI solution and ¼ test tube of glacial acetic acid. The liberated iodine is titrated against standard 0.1N sodium thiosulphate solution taken in the burette till a pale yellow colour is obtained. 2-3 drops of freshly prepared starch indicator is added. The resulting blue solution obtained is titrated against the same hypo solution till the blue colour just discharges. The titration is repeated for concordant values.

RESULT : The percentage of the available chlorine in the given sample of bleaching powder is……..

OBSERVATIONS

REACTION: $MnO_2 + C_2O_4^{2-} + 4H^+ \rightarrow Mn^{2+} + 2H_2O + 2CO_2$

Part I: Standardization of potassium permanganate

Burette: $KMnO_4$ solution

Conical flask: 25 cm^3 of 0.1N sodium oxalatesoln.+2 test tubes of dil.H_2SO_4

Indicator: Self

End point: Pale pink colour

Trial no.	1	2	3
Final burette reading			
Initial burette reading			
Vol. of $KMnO_4$ run down in cm^3(X)			

X =. .....cm^3

$(V_2N_2)_{KMnO_4} = (V_1N_1)\ Na_2Cr_2O_4$

Normality of $KMnO_4$ (N_2) = 25 x 0.1**/** V_2

N_2 =… ..... N

Part II: Estimation of Manganese dioxide

Burette: Standardized $KMnO_4$ solution

Conical flask: 0.2g of pyrolusite + 50 cm3 of 0.1N $Na_2C_2O_4$ + 3 test tubes of dil.H_2SO_4

Indicator: Self

End point: Pale pink

Trial no.	1	2	3
Final burette reading			
Initial burette reading			
Vol.of $KMnO_4$ run down in cm^3 (Y)			

Y=. ..... cm^3

1 cm^3 of 1N $KMnO_4$ = 1 cm3 of sodium oxalate = 1m.eq. of MnO_2

1 cm^3 of 1N $KMnO_4$ = 0.04346g of MnO_2

(2X-Y) cm^3 of 0.1N $KMnO_4$ = 0.04346 x 0.1 x (2X-Y)g

( ) cm^3 of 0.1N $KMnO_4$ = 0.04346 x 0.1 x ( ) =… ..... g(Z)

Percentage of MnO_2 in pyrolusite is = Z x100/0.2 = …….. x 100/0.2 =…….

EXPERIMENT NO. 08

AIM:DETERMINATION OF THE PERCENTAGE OF MANGANESE DIOXIDE IN PYROLUSITE ORE USING STANDARD POTASSIUMPERMANGANATE SOLUTION

PRINCIPLE: A known mass of pyrolusite ore is dissolved in acidified standard sodium oxalate solution in hot condition. Manganese dioxide in the ore reacts with sodium oxalate, the unreacted sodium oxalate is titrated against standard $KMnO_4$ solution till the end point. By calculating the reacted concentration of sodium oxalate, the percentage of MnO_2 in the pyrolusite can be calculated.

PROCEDURE:

Part- I:Standardization of potassium permanganate

25 cm^3 of 0.1N sodium oxalate solution is pipetted out into a clean conicalflask. Two test tubes of dilute sulphuric acid are added to it. The contents of the flask are heated on a Bunsen burner nearly to boiling. The hot solution is titrated against potassium permanganate solution taken in the burette slowly with dropwise addition until a permanent pale pink colour is obtained. The titration is repeated for concordant values.

Part-II: Estimation of MnO_2 in Pyrolusite:

About 0.2g of dry pyrolusite ore is taken in a clean conical flask. 50 cm^3 of 0.1N sodium oxalate is pipetted out into the flask. Three test tubes of 4N H_2SO_4 are also added into the same flask. The flask is covered with a funnel and digested for 30 mins. till all the ore particles dissolve completely. The funnel is washed with dil.H_2SO_4 and the washings are transferred into the flask. The solution is filtered, the filtrate is heated nearly to boiling. The hot solution is titrated against the standardized $KMnO_4$ solution till a permanent pale pink colour is obtained. The titration is repeated for concordant values.

Result: The percentage MnO_2 in given sample of pyrolusite ore is…….

OBSERVATIONS

REACTIONS:

$AgNO_3 + NaCl \rightarrow AgCl + NaNO_3$

$2AgNO_3 + K_2CrO_4 \rightarrow Ag_2CrO_4 + KNO_3$

Part I: Estimation of Chloride Burette:

Standard 0.01N $AgNO_3$ solution

Conical flask: 25 cm^3 of sodium chloride solution

Indicator: Potassium chromate

End point: Reddish brown

Trial no.	1	2	3
Final burette reading			
Initial burette reading			
Vol. of $AgNO_3$ run down in cm^3			

X =..... cm^3

Part II: Blank Titration

Burette: Standard 0.01N $AgNO_3$ solution

Conical flask: 50 cm^3 of distilled water

Indicator: Potassium chromate

End point: Reddish brown

Trial no.	1	2	3
Final burette reading			
Initial burette reading			
Vol. of $AgNO_3$ run down in cm^3			

Y=. ..... cm^3

1 cm^3 of 0.01N $AgNO_3$ = 0.0003546g of chloride(Cl^-)

(X-Y) cm^3 of 0.01N $AgNO_3$ = 0.0003546 g of chloride

( ) cm^3 of 0.01N $AgNO_3$ = 0.0003546 g of chloride Hence

Amount of chloride = 0.0003546/( ) = ... g

EXPERIMENT NO. 09

AIM: ESTIMATION OF CHLORIDE BY MOHR'S METHOD USING POTASSIUM CHROMATE AN ADSORPTION INDICATOR

Principle: On gradual addition of silver nitrate to a solution of NaCl, AgCl precipitates. After the complete precipitation of it as AgCl, excess of Ag^+ ions added combines with chromate ions present as indicator to give a light brown coloured precipitate. As silver nitrate is not a primary standard, the solution is standardized by titration against standard solution of sodium chloride.

Procedure:

1.Estimation of chloride: 25 ml of sodium chloride solution is pipetted out into a 250ml conical flask, 1ml of potassium chromate indicator is added. The mixture is titrated against a white back ground against 0.01N silver nitrate soln. with constant stirring so that the red colour produced by the addition of each drop gradually disappears. When red colour begins to disappear very slowly the addition is continued drop wise until a pale brown colour persists after swirling the liquid. The titration is repeated for concordant values.

2. Blank titration: To avoid the error in the titration, the titer reading may be corrected by performing a blank experiment. 1ml of the indicator is added to 50ml of distilled water and the mixture is titrated against the same standard 0.01N silver nitrate solution until the colour matches with that of the solution titrated previously. This volume of silver nitrate(Y) is subtracted from the volume of silver nitrate required in the earlier titration(X).

Result: The amount of chloride ions in the given sample is.....g.

OBSERVATIONS

REACTIONS: $AgNO_3 + NaCl \rightarrow AgCl + NaNO_3$

$AgNO_3 + NH_4CNS \rightarrow AgCNS + KNO_3$

Part I: Standardization of NH_4CNS (Ammonium thiocyanide) Burette:
NH_4CNS solution
Conical flask: 25 cm^3 of 0.01N $AgNO_3$ + 5ml of 6N HNO_3
Indicator: Ferric alum
End point: Faint brown

Trial no.	1	2	3
Final burette reading			
Initial burette reading			
Vol. of **NH_4CNS** run down in cm^3			

X =. ..... cm^3

Normality of NH_4CNS = 0.01 x 25/ X = 0.01 x 25/..... =.....N

Part II: Estimation of chloride:
Burette: NH_4CNSsolution
Conical flask: 25 cm^3 of 0.01N chloride soln. + 50ml of $AgNO_3$ + 5ml of 6N HNO_3
Indicator: Ferric alum
End point: Faint brown

Trial no.	1	2	3
Final burette reading			
Initial burette reading			
Vol. of **NH_4CNS** run down in cm^3			

Y=. .....cm^3

Normality of chloride solution = Y x Normality of NH_4CNS**/**25
= .....N
Mass of chloride in one liter of its solution = Normality of chloride x 35.5
=..... g

EXPERIMENT NO. 10

AIM: ESTIMATION OF CHLORIDE BY VOLHARD'S METHOD

Principle: Chloride can be estimated in 0.5 to 1.5N HNO_3 medium by treating with measured volume of excess of 0.01N $AgNO_3$ and then back titrating the excess of $AgNO_3$ with standard NH_4CNS at 25^0C in presence of ferric alum as indicator, reddish brown coloration due to the formation of ferric thiocyanate complex indicates the end point. Little amount of nitrobenzene is added to prevent the loss of silver as AgCNS.

Procedure:

PART- I: **Standardization of ammonium thiocyanate**

25 ml of 0.01N silver nitrate solution is pipetted out into a clean conical flask followed by adding 5ml of 6N nitric acid and 1ml of ferric alum indicator. The mixture is slowly titrated with drop wise addition of ammonium thiocyanate from a burette.A milky liquid is produced and a reddish brown colour quickly produced disappears on shaking. At the end point, the precipitate becomes flo cm^3 ulent, readily settles and a faint brown colour is obtained. The titration is repeated for concordant values.

PART-II: **Estimation of Chloride**

25 ml of 0.01N chloride solution is pipetted out into a clean conical flask followed by adding 5ml of 6N HNO_3. A measured volume of 50ml of standard excess of silver-nitrate is added along with 3ml of nitrobenzene and 1ml of ferric alum as indicator. The mixture is slowly titrated with dropwise addition of thiocyanate from a burette. A milky liquid is produced and a reddish brown colour quickly produced disappears on shaking. At the end point, the precipitate becomes flo cm^3 ulent, readily settles and a faint brown colour is obtained. The titration is repeated for concordant values.

Result: The amount of chloride ions in the given sample is.....g.

OBSERVATIONS
REACTIONS:

$K_2Cr_2O_7 + 7H_2SO_4 + 6FeSO_4 \rightarrow K_2SO_4 + Cr_2(SO_4)_3 + 3Fe_2(SO_4)_3 + 7H_2O$

$2K_3Fe(CN)_6 + 3FeSO_4 \rightarrow Fe_3[Fe(CN)_6]_2 + 3K_2SO_4$

Part I: Estimation of ferrous iron

Burette: 0.1N $K_2Cr_2O_7$ solution

Conical flask: 25 cm^3 of Ferrous-ferric mixture + 2test tubes of dil.H_2SO_4 +1t.t of water,

Indicator: Di-phenyl amine

End point: Appearance of violet colour

Trial no.	1	2	3
Final burette reading			
Initial burette reading			
Vol. of $K_2Cr_2O_7$ run down in cm^3			

$(V_2N_2)_{\text{Ferrous iron}} = (V_1N_1)_{\text{pot.dich.}}$

Normality of Fe^{2+} iron(N_2) = …….x 0.1/ 25

Amount of ferrous iron present in 250 cm^3 of its solution=N_2 x g.eq.wt of /4

= .....x 27.925/4

=. .....g

Part I: Estimation of ferric iron Burette:

0.1N $K_2Cr_2O_7$ solution

Conical flask: 25 cm^3 of Ferrous-ferric mixture+2test tubes of dil.H_2SO_4 +1t.t of water

Indicator: Di-phenyl amine

End point: Appearance of violet colour

Trial no.	1	2	3
Final burette reading			
Initial burette reading			
Vol. of $K_2Cr_2O_7$ run down in cm^3			

$(V_2N_2)_{\text{Ferric iron}} = (V_1N_1)_{\text{pot.dich.}}$; Normality of Fe^{3+} iron(N_2) = …….x 0.1/ 25 =

Amount of total ferric iron present in 250 cm^3 of its solution=N_2 x g.eq.wt/4

= …….x 55.85/4 =.....g

EXPERIMENT NO. 11

AIM:ESTIMATION OF FERROUS AND FERRIC IRON IN A GIVEN USING STANDARD POTASSIUM DICHROMATE SOLUTION

PROCEDURE:

1. ESTIMATION OF FERROUS IRON

25 cm^3 of the given ferrous ferric mixture is pipetted out into a clean conical flask. Two test tubes of dil.sulphuric acid, ½ test tube of phosphoric acid, one test tube of distilled water and 2-3 drops of diphenyl amine indicator are added. The resulting mixture is titrated versus the standard potassium dichromate solution till the appearance of deep violet colour. The titration is repeated for concordant values.

2.ESTIMATION OF TOTAL IRON:

25 cm^3 of the given ferrous ferric mixture is pipetted out into a clean conical flask followed by ½ a test tube of 2N con.HCl. The solution is heated, to the hot solution stannous chloride solution is added drop wise from a burette until it becomes just colourless, 2-3 drops are added in excess and the conical flask is cooled externally by running cold water from a tap. To the cold solution 5 ml of mercuric chloride is added at a stretch when a silky white precipitate is got. This solution is titrated against standard potassium dichromate solution taken in a burette using diphenyl amine as indicator till violet colour is obtained as the end point. The experiment is repeated for concordant values.

RESULT: Amount of ferrous iron present in the given solution is ……g and the amount of ferric iron present in the given solution is ..... g.

OBSERVATIONS

REACTIONS:

1. $(NH_4)_2SO_4 + 2\,NaOH \rightarrow Na_2SO_4 + H_2O + 2NH_3$
2. $NH_4NO_3 + 2\,NaOH \rightarrow NaNO_3 + H_2O + NH_3$
3. $(NH_4)_2CO_3 + 2NaOH \rightarrow Na_2CO_3 + H_2O + 2NH_3$

Burette: 0.1N HCl solution
Conical flask: Ammonium salt solution + 50ml of 0,1N NaOH
Indicator: Methyl orange
End point: Yellow to orange red

Trial no.	1	2	3
Final burette reading			
Initial burette reading			
Vol. of HCl run down in cm^3			

X =. .....cm^3

1 cm^3 of 1N NaOH = 17g of ammonia
(50 – x) cm^3 of 0.1N NaOH = 17 x (50-x) mg of ammonia
a = 17 x (50-x) mg of ammonia

If w is the weight of the ammonium salt taken, then the percentage of ammonia nitrogen in the given ammonium salt sample = a x 100/w

EXPERIMENT NO. 12

AIM: ESTIMATION OF NITROGEN IN AN AMMONIUM SALT USING SODIUM HYDROXIDE SOLUTION AND STANDARD OXALIC ACID

Principle: A known mass of the ammonium salt is treated with an excess of standard alkali and boiled. The ammonium salt undergoes decomposition utilizing some amount of the alkali and liberating ammonia which is expelled by boiling. The remaining alkali is titrated against standard acid. From the titre values the amount of nitrogen sample of ammonium salt is calculated.

PROCEDURE : About 2g of ammonium salt sample is weighed a cm^3 accurately into a clean conical flask. One test tube of distilled water is added followed by 50 cm^3 of 0.1N sodium hydroxide through a pipette. The solution is boiled until a red litmus paper fails to turn blue when exposed to the vapor. The cold solution is titrated against 0.1N HCl using methyl orange as indicator. The experiment is repeated with one more sample of the ammonium salt. Let the volume consumed be x cm^3 .

RESULT:
The percentage of nitrogen in the given sample of the ammonium salt is .....

OBSERVATIONS

REACTIONS: **1.CO_3^{2-} + H_3O^+ → HCO_3^- + H_2O**
2. HCO_3^- + H_3O^+ → H_2CO_3 + H_2O

Burette: 0.1N HCl solution
Conical flask: W g of sodium carbonate and sodium bicarbonate mixture
Indicator: a) Phenolpthalein b) Methyl orange
End point: a) Appearance of permanent pale pink b) Yellow to pale pink

a) Phenolphthalein end point:

Trial no.	1	2	3
Final burette reading			
Initial burette reading			
Vol. of HCl run down in cm^3			

x =.....cm^3

b) Methyl orange end point:

Trial no.	1	2	3
Final burette reading			
Initial burette reading			
Vol. of HCl run down in cm^3			

y =.....cm^3

Percentage of sodium carbonate = y x 0.1 x 106 x 100/ w

Percentage of sodium bicarbonate = (x-y) x 0.1 x 84 x 100/w

EXPERIMENT NO. 13

AIM: **ESTIMATION OF CARBONATE AND BI-CARBONATE INA GIVEN MIXTURE**

Principle: Carbonic acid is a diprotic acid, which furnishes two protons per molecule.The estimation of carbonate and bi-carbonate in a mixture of the two is based on equilibria of diprotic acid and its salt.The first pK_a is 6.34 and the second is 10.36, Phenolphthalein is a suitable indicator for the first end point and methyl orange for the second. Mixtures of carbonates and bicarbonates are analyzed using the double indicator method.

PROCEDURE : A known mass(wg) of a mixture of sodium carbonate and sodium bicarbonate is transferred into a clean conical flask followed by one test tube of distilled water and two drops of phenolphthalein indicator and the solution is titrated against standard 0.1N HCl until a permanent pale pink color is got. Let the average value got is x cm^3 , At this stage two drops of methyl orange is added and the titration is continued till the orange color changes to permanent pale pink. Let the average titre value be y cm^3 .

RESULT: The percentage of carbonate and bicarbonate in the given salt is…….