

INDEX

SI No.	Date	Name of the Experiment	Page No.	Initial	Remarks
01	02-11-2021	Introduction to chemistry laboratory; use of equipments: Molarity, Normality, primary, secondary Standard Solutions, Volumetric titration, Quantitative analysis, Error.	01-15	JKR 23/11/2021	AT
02	09-11-2021	Determination of the Strength and amount of Hydrochloric acid.	16-19	XK 09/11/2021	AT
03	16-11-21	Preparation of Standard sodium oxalate solution & standardization of potassium permanganate solution.	20-22	XK 23/11/2021	AT
04	23-11-21	Determination of ferrousion (Fe^{2+}) with standard potassium permanganate solution.	23-25		
05	30.11.21	Preparation of standard potassium dichromate solution and standardization of sodium thiosulphate solution.	25-28	XK 30/11/2021	AT
				XK 30/11/2021	
				XK 30/11/2021	

DATE 02-11-2021
EXP. NO. 01
PAGE NO. 01

Name of the Experiment: Introduction to chemistry laboratory
use of equipments: Molarity, Normality, primary, secondary standard solutions, volumetric titration, Quantitative analysis, Error

Chemical analysis is carried out to understand the composition of the materials. This will enable to access the properties of materials and if necessary, modify the properties for technical and scientific applications. Chemical analysis is the resolution of a chemical compound into proximate or ultimate parts. Traditional manual chemical analysis is divided into two types.

(a) Qualitative analysis: It deals with the identification and confirmation of the nature of the substance or impurities present in a given sample.

(b) Quantitative analysis: It deals with the estimation of how much for each component or of specified components are present in a given sample, including in trace quantities. The substance determined is analyte and the minor or the trace quantities are impurities. The complete quantitative analysis consists of five steps Sampling, dissolution of the sample, conversion of the analyte into a form suitable for measurement, calculation and interpretation of data.

Quantitative chemical analysis is further divided into two types:

(a) Volumetric analysis: It is based on a chemical reaction and the calculations are on the sample stoichiometric relations of chemical reactions. It is a quantitative chemical analysis by measure, which consists essentially in determining the volume of solution of accurately known concentration required to react quantitatively with solution of substance being determined.

DATE 02-11-2021
EXP. NO. 01
PAGE NO. 02

Name of the Experiment.....
continued...

The weight of the substance to be determined is then calculated from the volume of the substance solutions and the known laws of chemical equivalence. The standard solution is usually added from a graduated glass vessel called a burette. The process of adding the standard solution until the reaction is just complete is termed as a titrimetric analysis. The point at which the titration is completed is called the equivalence point or endpoint. Volumetric methods need simpler apparatus and the process are quickly performed. They required a balance for weighing, calibrated measuring vessels like burettes, pipettes and volumetric flasks and substance for known purity for the preparation of standard solutions.

(b) Gravimetric analysis: Unlike volumetric analysis where measurements of volumes are involved, gravimetric method involves separation of the analyte into a solid form as precipitate. The measurement step in gravimetry is weighting the element or a definite compound of the element in as pure form as possible, is isolated and weighed. The weight of the element or compound may then be trap readily calculated from a knowledge of the formula of the compound and the atomic weights of the constituent elements. The isolation of the required species is achieved by precipitation methods, volatilization, evolution methods or electroanalytical methods. The advantage of gravimetric method over volumetric analysis is that the constituent may be seen and examined of the presence of impurities of the two methods, gravimetric analysis is accurate; but volumetric analysis is much

DATE 02-11-2021

EXP. NO. 01

PAGE NO. 03

Name of the Experiment.....
continued...

more readily and quickly carried out. Then error allowed the volumetric analysis is 0.2%.

Essential conditions for accurate titrimetry:

- (1) Clean glass apparatus must only be used in titrimetric analysis. Glass apparatus must be free from grease and thoroughly rinsed with distilled water and dried in an oven before use.
- (2) The stoichiometric equation governing the reaction must be known.
- (3) The reaction should be practically instantaneous or proceed with sufficient speed. Addition of suitable catalyst may help to increase the speed of the reaction.
- (4) There must be some marked change in some physical or chemical property of the titrator to assess the completion of the reaction.
- (5) An indicator should be available, which should sharply define the endpoint of the reaction.
- (6) The standard solution must not alter in its strength during the period of experimentation. The solution must be stable to light, atmosphere and must not react with the solvent or containers.

DATE 02-11-2021
EXP. NO. 01
PAGE NO. 04

Name of the Experiment
continued.....

(f) The balance must be accurate; stable and sensitive and give the same result in successive weighings.

Terms used in volumetric analysis :

(a) Titration : It is a process of adding one solution from the burette to another known volume of solution in a conical flask, in order to complete the chemical reaction.

(b) Titrate : The substance being titrated in the reaction vessel is termed as titrate. However, these terms titrant and titrate are relative.

(c) Equivalence point or stoichiometric end point : It is the point at which the amount of reagent and substance being determined are stoichiometrically equivalent according to the equation representing the chemical reaction or it the exact stage at which the chemical reaction involved in the titration is just complete.

(d) Titrant : The reagent being added through a burette is called as titrant.

(e) Indicator : The substance which helps in the visual detection of the completion of the reaction in the titration is known as indicator.

(f) Pipette : A glass apparatus with which a fixed volume

DATE 02-11-2021

EXP. NO. 01

PAGE NO. 05

Name of the Experiment.....
continued...

of solution as marked on the pipette can be delivered. It should be previously rinsed with the solution of titrate. Solution is drawn by suction above the mark and the end is closed with pointing tightly with three fingers on one side and thumb on the other side. Care must be taken to see that air bubbles aren't present while drawing solution and the solution drop in the nozzle is never released by blow off.

(g) Burette: Burettes are long cylindrical tubes of uniform bore throughout the graduated length with a closed bottom with stop cock and nozzle at the end. Titrant is filled into the burette including the nozzle and clamped to the stand vertically for five minutes before use, to eliminate air bubbles, if any. The stop cock can release solution as a continuous flow, but gentle operation will enable drop wise addition. Burette releases sufficient measured volume of the solution into the titration vessel till the reaction is complete as indicated by change in colour of indicator.

(h) Conical flask: It is the reaction vessel containing titrate and indicator solution in distilled water. It should be rinsed with distilled water only before and after each titration.

(i) Weighting bottle: This is used to weigh weight a required of specified solid for preparing standard solutions. The difference in weights before and after use gives the weight of the substance transferred.

DATE 02-11-2021
EXP. NO. 01
PAGE NO. 06

Name of the Experiment.....
continued...

(j) Analytical balance: It is a two pan balance with graduated beam holding aluminium rider.

(k) Measuring cylinder or jar: This is a graduated cylindrical glass vessel to draw approximate volumes of solution.

(l) Volumetric flask: This is a stoppered glass vessel with flat bottom and pear shape with a long narrow neck containing a thin line mark etched on the neck. This is used to prepare solutions with definite concentrations.

(m) Porcelain tile: A ceramic plate 6" x 6" size glazed white on one side and placed under the conical flask or any titration vessel during titration, which helps for better detection of colour change at the equivalence point.

(n) End-point: The point at which the colour change which of the indicator is visible is called end point.

(o) Titration error: Ideally, the visible end-point and the equivalence point should coincide, but in practice there is always a difference between the two, this difference being called the titration error.

Most probable value: The average value of these results is taken as most probable value. Hence, it is not a true value.

Absolute error: The difference between observed and measured

DATE 02-11-2021
EXP. NO. 01
PAGE NO. 07

Name of the Experiment
continued . . .

value to a true or most probable value is known as absolute error. It is measure of accuracy.

Accuracy: It is the concordance between measured value and most probable value or true value.

Relative error: It is the absolute error divided by true or most probable value. It is usually expressed in terms of % ppm.

$$\therefore \text{Relative error} = \frac{\text{absolute error}}{\text{most probable value}} \times 100 = x\%$$

$$= \frac{\text{absolute error}}{\text{most probable value}} \times 100 = x \text{ ppm}$$

Mean deviation: It is an agreement between a series of results. It can be evaluated by determining arithmetic means of the results, then calculating the deviation of each individual from the mean, finally divided the sum of the deviations by the number of measurements.

Table 8- 1.1 (Mean deviation calculation.)

Results		Deviations
48.32		0.04
48.36		0.08
48.23	Mean = $\frac{241.40}{5}$	0.05
48.11	= 48.28	0.17
48.38		0.10
241.40		0.44

DATE 02-11-2021
EXP. NO. 01
PAGE NO. 08

Name of the Experiment.....
continued...

Relative mean deviation: It is the mean deviation divided by the mean. It is expressed in % or ppm.

$$\begin{aligned}\text{Relative mean deviation} &= \frac{0.09}{44.28} \times 100 \\ &= 0.19\% \\ &= \frac{0.09}{44.28} \times 1000 \\ &= 1.9 \text{ ppm}\end{aligned}$$

It is a measure of precision.

Precision: Precision is a measure of reproducibility of measurements.

Standard deviation: It measures the closeness of the results with the mean. Smaller the standard deviation, more closely are the results to the mean value.

$$\text{Standard deviation } (s) = \sqrt{\frac{\sum_{i=1}^n (x_i - \bar{x})^2}{n-1}}$$

Where, x_i = Individual measurements.

\bar{x} = Average of the results.

n = Number of measurements.

Relative standard deviation (RSD): It is a measure of the quality of the sample. Large the RSD, shows the poor the quality of the sample.

$$\therefore RSD = \frac{s}{\bar{x}}$$

Standard solution: A solution of known strength is called as standard solution. It is prepared by dissolving a definite weighed

DATE 02-11-2021
EXP. NO. 01
PAGE NO. 09

Name of the Experiment.....
continued.....

amount of the substance in a small volume of solvent in a volumetric flask of prescribed volume.

Primary standard substance: A primary standard substance should satisfy the following requirements.

- (a) The substance used as a primary standard should be available in a state of high purity and preserved in a pure state. It is known as analar or AR grade.
- (b) The substance should maintain its composition during storage and unaltered in air during weighting.
- (c) It should not be hygroscopic, deliquescent or efflorescent.
- (d) The total amount of impurities should not in general exceed to 0.01 to 0.02 %
- (e) It is preferred to have high equivalent weight so that the errors in weighting are minimum.
- (f) The substance should readily be under the conditions in which it is employed.

Primary standard substances commonly employed: In acidometry or alkalimetry: Sodium carbonate (Na_2CO_3), Borax ($\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$) and potassium hydrogen phthalate ($\text{KHC}_8\text{H}_4\text{O}_4$)

In precipitation titrations: Potassium Dichromate, sodium oxalate, Arsenious Oxide etc.

Secondary standard substances: Those substance which don't loose water of crystallization such as sodium bicarbonate, ferric

DATE 02-11-2021
EXP. NO. 01
PAGE NO. 10

Name of the Experiment.....
continued ..

ammonium sulphate, copper sulphate, silver nitrate etc.

Classification of reaction in volumetric analysis: For convenience sake, reactions in volumetric analysis are classified into four types. However, the classification is not strictly followed and overlap may occur.

(1) Neutralization reactions (acidimetry and alkalimetry): These reactions are based on the principle of neutralization of a free acid or free base. The free bases are those formed from salts of weak acids by hydrolysis are titrated with a standard acid or vice versa. The basic common reaction involves the interaction of hydrogen and hydroxyl ions to form water molecule. Selective acid base indicators are only employed to mark mark the sudden change in pH during these titrations.

(2) Oxidation reduction reactions (redox-reactions): These reactions involve a change in the oxidation number or transfer of electrons amongst the reacting substances.

(3) complex formation reactions: They involve the combination of ions to form a soluble, slightly dissociated ion or compound.

(4) precipitation reactions: They involve the combination of ions to form a simple precipitate. No change in valency occurs. Concentration of a standard solution is generally expressed in following chemical units.

DATE 02-11-2021
EXP. NO. 01
PAGE NO. 11

Name of the Experiment
continued . . .

Molarity: Molarity of a solution is defined as the number of gram moles of solute present in one liter of the total solution.

$$\therefore \text{Molarity} = \frac{\text{Number of gram moles of solute}}{\text{Volume of solution in Liters}}$$

$$\text{Molarity} = \frac{\text{Weight of the substance}}{\text{Gram Mol. weight of the substance}} \times \frac{1000}{V \text{ in mL}}$$

Normality: The normality of a solution is defined as the number of gram-equivalents of solute present in one liter of the total solution.

$$\text{Normality} = \frac{\text{No. of Grm. Eqw. weights of solute}}{\text{Volume of solution in L}}$$

$$\text{Normality} = \frac{\text{Weight of the substance}}{\text{Gram Eq. wt of the substance}} \times \frac{1000}{V \text{ in mL}}$$

ppm: Parts per million (ppm) is a better notation for expressing concentrations of trace quantities of species present. The system is convenient for expressing the concentrations of very dilute solutions. It specifies the numbers of parts of solute in one million parts of solution and is expressed mathematically as —

$$\text{ppm} = w \times 10^6 \text{ or}$$

Where, w is the number of grams of solute.

$$w = \text{No of grams of solvent.}$$

DATE 02-11-2021
EXP. NO. 01
PAGE NO. 12

Name of the Experiment.....
continued.....

One liter of water at room temperature weighs approximately 10^6 mg, hence a convenient relationship to remember is that one milligram of solute in one liter of water is a concentration of 1 ppm. For even more dilute solution the system parts per billion (ppb) is employed. and $\text{ppb} = w \times 10^9 / w$

General safety precautions:

- ① Every students entering the lab shall wear a white apron or a protective coat and shoe.
- ② While leaving the lab ensure that all gas tap connections, electrical switches shall be kept in off mode. All instruments must be properly shut down as per instructions of the teacher in charge. Water taps, gas connections and fans, lights etc.
- ③ Students are advised not to tamper electrical or gas connections and they should not touch electrical main switches.
- ④ All water tap connections shall be used as and when needed or be turned into off mode mode when not in use.
- ⑤ All waste materials broken glass, filter papers etc, shall be dumped into dustbin provided. Any spilling of materials should be cleaned immediately.
- ⑥ The bottles and other apparatus should not be moved from one place to another unless introduced by teacher. Otherwise it becomes difficult for another user to trace it.
- ⑦ All doors and windows must be kept open while using the laboratory.
- ⑧ Solutions containing strong acids should never be poured

directly into the sink.

- ⑨ A fuming cup board must be used for handing reactions involving corrosive chemicals like concentrated acids, ammonia etc.
- ⑩ Dry alkalis and concentrated acids should never be touched with hands or spilled over on the skin.
- ⑪ The test tubes and open vessels are to be heated with caution. The face of the vessels being heated should not be kept towards your neighbouring colleague in lab.
- ⑫ Heating must be done gradually and carefully.
- ⑬ The nozzles of burettes and pipettes must be protected against casual handling resulting in breakage, as the entire apparatus become unusable if its nozzle is broken.
- ⑭ The glass corks of the volumetric flask should be tied through a small thread to prevent mixing of stoppers.
- ⑮ All electrical devices should be plugged in with a dry hand only, preferably with dry shoes.
- ⑯ Students should not sit on the work tables or keep hands often rest on tables for their own safety.
- ⑰ The balance pan must be clean and dry. A watch glass or paper may be used and necessary tare may be made.
- ⑱ Never return unused solutions or solids to the stock containers as they can contaminate the stock chemicals.
- ⑲ The floor space in the laboratory shall be clean and free from spillage, broken glass or pieces, straw, cloth pieces or paper to avoid accidents.
- ⑳ Fire buckets containing sand and water for fire extinguishers should always be available in the lab in easily

DATE 02-11-2021
EXP. NO. 01
PAGE NO. 14

Name of the Experiment.....
continued..

accessible places.

- (21) When fire originate all gas taps must be cut off immediately.
- (22) In case of emergency; don't get panicky and be brave enough to handle the solution. Medical attention should be requested immediately. If skin comes contact with acid solutions, it should be washed with water and sodium bicarbonate solution alternately and continuously till skin is smooth & smoothened. Similarly exposure to caustic alkalis may be treated with dilute acidic or basic acid solutions and water. If eyes come into contact with chemicals, wash them with plenty of water and clean them with a dry fresh cloth.

Introduction to students: The following instructions are offered to the students for acquiring curriculum based skills in chemistry laboratory. Since chemical analysis is a basic component in all branches of engineering & technology, which will be useful in later courses of study, research the technical job requirements.

- (1) The students should make himself completely familiar with the experiment she is about to perform, its theory, procedure and precautions, before the beginning of the experiment.
- (2) This lab manual is intended to help in this aspect. Each apparatus is designed to be used in a specific manner to give expected results.
- (3) All students should carry an exclusive observation note book

DATE 02-11-2021
EXP. NO. 01
PAGE NO. 15

Name of the Experiment.....
continued...

and make a record of every observation in it during experiment immediately.

- (4) The student should read the procedure of the experiment and conduct it accordingly.
- (5) Students should not use excess amount of reagents or chemicals.
- (6) After completion of the assigned experiment & proper record of all necessary observations on the note book, the apparatus may be returned to the in charge. Calculations shall be completed in the observation note books to arrive at the results, records will not be certified or evaluated unless signed observations are submitted to the teachers.
- (7) Students are advised to record observations on the spots of experimentation and after approval of the concerned teacher prepare the record in due proforma and submit for grading in the subsequent week.
- (8) If there is breakage of supplied equipment or abuse of any instrument the concerned student shall be charged as per present price list. Hence, students are advised to verify equipment before they obtain and give satisfactory report to lab in charge.

X/02/11/2021

DATE 09-11-2021

EXP. NO. 02

PAGE NO. 16

Name of the Experiment. Acidimetric - Alkalimetric determination of the strength and amount of Hydrochloric acid.

Objective: The objective of this experiment is -

- To determine the strength of Hydrochloric acid.
- To determine the amount of hydrochloric acid in given volume of solution.

Apparatus and chemical required: Burette, pipette, volumetric flask, funnel, conical flasks, wash bottle, with distilled water, burette stand with suitable burette clamp, a digital balance, anhydrous sodium carbonate AR (solid), watch glass, methyl orange indicator solution.

Theory:



According to the principle of neutralization reactions, a free acid reacts with a base to form salt and water, in which the hydrogen ions of the acid combine with the hydroxyl ions of the free base in stoichiometric proportions, this being common for all acid base reactions. For example, in the reaction two mole of hydrochloric acid reacts with one mole of sodium carbonate. Sodium carbonate, being a salt of strong base sodium hydroxide and weak carbonic acid, shows residual alkaline properties and hence can be considered as a weak base, since it was available in pure state and satisfies all other required conditions, anhydrous sodium carbonate was a primary standard. Thus, the concentration of a solution of an acid/base can be estimated by titrating it was a standardized base/acid solution. One of the above solutions shall be of known concentration. On mixing the two solutions in the process of titrating, the

DATE 09-11-2021
EXP. NO. 02
PAGE NO. 17

Name of the Experiment

continued...

reaction goes to completion instantaneously and for estimation of the unknown the end point should be detected with a suitable indicator, methyl orange solution here. On the addition of hydrochloric acid from burette to sodium carbonate in conical flask the pH of the solution slowly decreases and crosses the value of 7 of the end point. Methyl orange solution being yellow in its colour above pH 7 shows a pale pink colour at the equivalence point which indicates the completion of the reaction. During titration a fixed volume of a standard sodium carbonate solution taken in the titration vessel reacts with a measure volume hydrochloric acid at the end point indicated by the initial addition of a drop or two of methyl orange solution giving a due colour change at the end point. Measurement of these volumes and substitutions in the stoichiometric formula gives the concentration of the unknown species i.e., hydrochloric acid here.

Procedure:

- Preparation of standard solution carbonate solution (Primary Standard) :- About 1.37 g of AR grade anhydrous sodium carbonate solid sample was weighed accurately and was transferred into a 250 mL volumetric flask through a glass funnel. The substance is dissolved completely in a minimum amount of distilled water and the solution is made up to the mark. The solution is made homogenous by shaking in the stopped volumetric flask.

DATE 09-11-2021

EXP. NO. 02

PAGE NO. 18

Name of the Experiment.....
continued....Determination of the concentration of the given hydrochloric acid:

The given hot HCl solution was made up to the mark with distilled water and was shaken well to make it homogenous in concentration. Now the burette is rinsed with a small volume of given hydrochloric acid solution and the rinsed solution was discharged in to the sink. The burette including its nozzle portion was filled with Hydrochloric solution applied without air bubbles up to the zero mark and allowed to stand vertically for few minutes. 10 mL of the standard sodium carbonate solution is drawn through a pipette into a 250 mL clean conical flask. To this, 10 mL of distilled water 2-3 drops of methyl orange indicator solution are added to give a yellow colour. This mixture is then titrated with the hydrochloric acid solution run down from the burette. The contents of the conical flask are swirled throughout the titration, till the end point was reached. The end point was determined by a change in colour of the solution from yellow pale pink. The slight est pale pink colour obtained at the end point in the ~~white~~ back drop of the porcelain tile and nothing.

The initial and final burette readings marks the end of the titration. The difference in the initial and final readings of the burette gives the volume of HCl reacted with 10 mL of the sodium carbonate solution taken. The same procedure of titration was repeated until concurrent readings are obtained and the observations are tabulated.

Figure No.....

Calculation:

Weight of Na_2CO_3 taken = 1.37 g
 $\text{Strength of } \text{Na}_2\text{CO}_3, S_2 = \frac{\text{Weight taken}}{\text{Weight to be taken}} \times 0.05 \text{ M}$

Strength of $\text{Na}_2\text{CO}_3, S_2 = \frac{1.37}{1.325} \times 0.05 \text{ M}$

$S_2 = \frac{1.37}{1.325} \times 0.05 \text{ M}$

Now, Volume of HCl consumed, $V_1 = 5.8 \text{ mL}$

Volume of Na_2CO_3 taken, $V_2 = 10 \text{ mL}$

Molarity / Strength of $\text{Na}_2\text{CO}_3, S_2 = 0.05 \text{ M}$

Molarity / Strength of HCl, $S_1 = ?$

We know that,

$$V_1 \times S_1 = V_2 \times S_2$$

$$S_1 = \frac{V_2 S_2}{V_1} = \frac{10 \times 0.05}{5.8} = 0.0862 \text{ M}$$

Concentration of supplied HCl solution = 0.0862 M

The amount of HCl present in supplied solution in

the 100 mL volumetric flask = Molarity of HCl $\times \frac{36.5}{10}$

$$= 0.0862 \times \frac{36.5}{10} \text{ g}$$

$$= 0.31463 \text{ g}$$

DATE 09-11-2021 Name of the Experiment.....
 EXP. NO. 02 Continued....
 PAGE NO. 19

Experiment Data :

No. of Obs	Volume of Na_2CO_3 (mL)	Burette reading (mL) Initial burette reading (IBR)	Final Burette reading (FBR)	Diff (FBR-IBR)	Mean Volume of HCl (mL)	Strength of HCl (M)
1	10.0	0	5.8	5.8		
2	10.0	9.8	11.6	5.8	5.8	0.0862
3	10.0	11.6	17.6	6		

Result : The strength of HCl solution was 0.0862 M and the amount of Hydrochloric acid present in a given sample of 100 mL = 0.31463 g

Report :

- We have to measure the volume of hydrochloric acid.
- Also we measure the volume of sodium carbonate (Na_2CO_3) properly.
- In the end, we have to calculate the strength of Hydrochloric acid.

Discussion : While doing the experiment we were needed to follow some important notes such as—

- (1) We were advised to clean the apparatus properly.
- (2) We weighted Na_2CO_3 & HCl properly as per as teacher's instruction.
- (3) We were not lower to temper with other chemicals on the lab.
- (4) We were advised to be cautious while taking the readings of burette.
- (5) The slightest pale pink colour obtained we were advised to show the solution to teacher the calculated the required result.
- (6) We returned all the apparatus after doing the lab.
- (7) While leaving the lab, we needed to ensure that all gas connections electrical switches were kept in off mode.

X/09/11

DATE 16.11.21
EXP. NO. 03
PAGE NO. 20

Name of the Experiment Preparation of standard sodium oxalate solution & standardization of potassium permanganate solution.

Objective : The Objective of the experiment are -

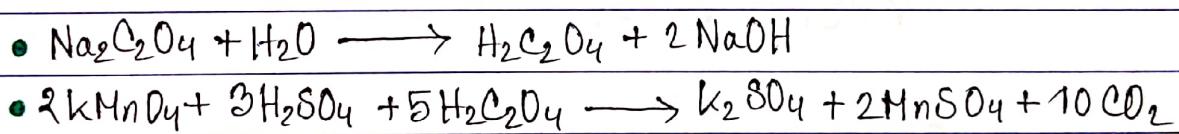
- (1) To prepare standard solution
- (2) To standardize the unknown solution.

Apparatus and chemicals required :

- (1) Burette and pipette.
- (2) conical flask and volumetric flask.
- (3) 2 N H_2SO_4
- (4) $Na_2C_2O_4$
- (5) $KMnO_4$
- (6) Distilled water.

Theory : The acidified solution of $KMnO_4$ being an oxidizing agent which can be standardized by titration against a compared solution of reducing agent like sodium oxalate. Thus $KMnO_4$ is presence of dilute H_2SO_4 oxidizes the to CO_2 & H_2O in the following ways

ways :-



The electronic interpretation of above eqⁿ are as follows:

- Oxidation reaction :- $5C_2O_4^{2-} - 10e^- \longrightarrow 10 CO_2$
- Reduction reaction :- $2MnO_4^- + 16H^+ + 10e^- \longrightarrow 2Mn^{2+} + 8H_2O$
- Final reaction :- $2MnO_4^- + 5C_2O_4^{2-} + 16H^+ \longrightarrow 2Mn^{2+} + 8H_2O + 10CO_2$

Potassium permanganate is a versatile and strong oxidizing agent. The advantage of $KMnO_4$ is that it serves as its own indicator. The pink colour being distinguishable even

Figure No.

Calculation:

Weight of $\text{Na}_2\text{C}_2\text{O}_4$ taken = 2.6 gm

$$\text{Strength of } \text{Na}_2\text{C}_2\text{O}_4, S_2 = \frac{\text{Weight taken}}{\text{Weight to be taken}} \times 0.1 \text{ N}$$

$$= \frac{0.68}{0.67} \times 0.1 \text{ N}$$

$$= 0.102 \text{ N}$$

Now,

Volume of KMnO_4 consumed, $V_1 = 12.5 \text{ mL}$

Volume of $\text{Na}_2\text{C}_2\text{O}_4$ taken, $V_2 = 10 \text{ mL}$

Normality / Strength of $\text{Na}_2\text{C}_2\text{O}_4, S_2 = 0.702 \text{ N}$

Normality / Strength of $\text{KMnO}_4, S_1 = ?$

We know, $V_1 \times S_1 = V_2 \times S_2$

$$V_1 \times S_1 = V_2 \times S_2$$

$$0.125 \times S_1 = \frac{V_2 \times S_2}{V_1}$$

$$S_1 =$$

$$= \frac{10 \times 0.102}{12.5}$$

$$= 0.0816 \text{ N}$$

$$= 0.0816 \text{ N}$$

DATE 16.11.21
EXP. NO. 03
PAGE NO. 21

Name of the Experiment.....
continued ...

If the solution is very dilute. The equivalent weight of KMnO_4 is therefore $1/5$ mole of molecular weight i.e. 31.61 gm.

Procedure: Prepare 100mL of 0.1N sodium oxalate (0.67g) solution by shaking well through proper mixing and make up to the mark of volumetric flask. Take 10mL of sodium oxalate solution in a conical flask and add 40mL of 2N H_2SO_4 . Heat the solution up to $(60-70)^\circ\text{C}$. We have to take KMnO_4 solution in a burette and 2-3 drops of the solution into sodium oxalate solution until the colour disappears. Adding the KMnO_4 solution at moderate rate while stirring the clear solution continuously and we make sure that the temperature is more than 60°C . Now continue the adding of KMnO_4 solution dropwise until one drop is observed to give a definite pink colour to the solution. This is the end point of titration.

Experimental Data :- Preparation of 0.2N 100mL $\text{Na}_2\text{C}_2\text{O}_4$ solution required :-

No of observation	Volume of $\text{Na}_2\text{C}_2\text{O}_4$ solution (mL)	Burette reading			Mean Volume of KMnO_4 (mL)
		Initial Burette reading (IBR)	Final Burette reading (FBR)	Difference (FBR - IBR)	
1	10.0	0	12.5	12.5	
2	10.0	12.5	24.5	12	12.5
3	10.0	24.5	37	12.5	

Result: The strength of KMnO_4 solution is = 0.0816N

DATE 16.11.21
EXP. NO. 03
PAGE NO. 22

Name of the Experiment continued ...

Report :

- We have to measure the volume of oxalic acid.
- Also we measure the volume of potassium permanganate pro perty.
- In the end, we have to calculate the strength of potassium permanganate .

Discussion : The main purpose of this experiment is to preparation of standard sodium oxalate & standardization of potassium permanganate solution . During this experiment we were needed to follow some important instruction such as :-

- We were instructed to clean the apparatus properly with distilled Water.
- We weighted $\text{Na}_2\text{C}_2\text{O}_4$ & H_2SO_4 properly as per as teacher instruction.
- We were advised to be coutions while taking the reading of burette .
- The pink colour obtained we were advised to show the solution to teacher the calculate the required result .
- After doing the experiment, we returned all the apparatus to the lab attended after clearing with clean water .

~~Ans 03/11/21~~

DATE 23.11.21
EXP. NO. 04
PAGE NO. 23

Name of the Experiment Determination of Ferrous Ion (Fe^{2+}) with standard potassium permanganate solution

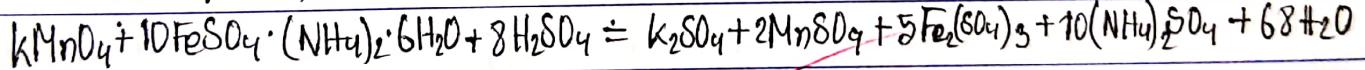
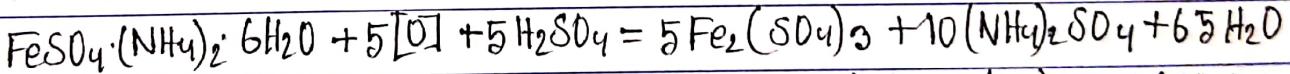
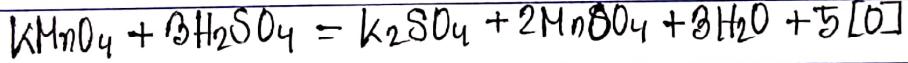
Objective : The objective of this experiment is -

- (i) To calculate the amount of ferrous ion (Fe^{2+}) in supplied sample.
- (ii) The percentage of iron in any alloy can also be determined by this experiment.

Apparatus & chemical required :

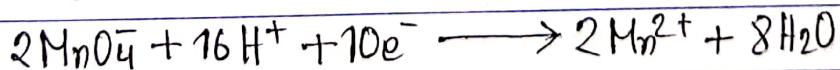
- (i) Burette and pipette.
- (ii) Conical flask and volumetric flask.
- (iii) 1N H_2SO_4
- (iv) Mohr's salt.
- (v) KMnO_4
- (vi) Distilled water.

Theory : The acidified solution of KMnO_4 being an oxidizing agent which converts ferrous ions to ferric ions according to the following equation :-

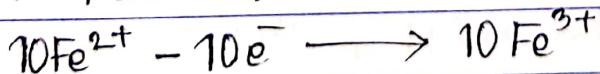


The electronic interpretation of above equation is as follow:-

• Reduction Reaction :



• Oxidation Reaction :



• Final Reaction :



Since the oxidation of ions from ferrous to ferric states involves

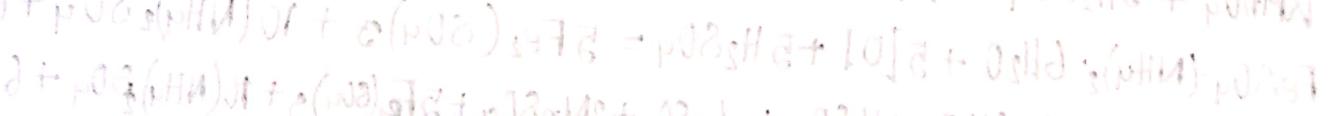
Figure No.

Calculation:

1000 mL 1N KMnO_4 = $\frac{55.85 \text{ gm}}{1000 \text{ ml}}$ gm of iron

$$\therefore 1 \text{ mL } 1\text{N } \text{KMnO}_4 = \frac{55.85 \times 10^{-3}}{1000} \text{ gm of iron}$$

$$= 0.56400 \text{ gm of iron}$$



so overall reaction leads to regeneration of catalyst



so overall reaction

DATE 28.11.21
EXP. NO. 04
PAGE NO. 24

Name of the Experiment : Reduction of Iron by Potassium Permanganate
continued ...

loss of one electron. So, the equivalent weight of the ferrous sulphate or the Mohr's salt is equal to its molecular weight. One litre of normal solution of ferrous sulphate of Mohr's salt contain one gram mole atom of iron.

Procedure : Dilute the supplied mohr's salt (0.92g) sample solution with about 70 ml 1N sulphuric acid. Make up to the mark with distilled water and shaking well through mixing. Pipette out 10ml of the solution into conical flask and add about 40ml 1N sulphuric acid in it. Titrate the mohr's salt solution with standard KMnO_4 (0.79)g solution.

Experiment Data :- $1000 \text{ mL } 1\text{M } \text{KMnO}_4 = 5 \text{ mol } \text{Fe}^{2+}$ or,

or,

$1000 \text{ mL } 1\text{N } \text{KMnO}_4 = 55.85 \text{ gm of iron}$

Titration Table :

No. of observation	Volume of Mohr's salt solution (ml)	Burette Reading IR	Burette Reading FR	Difference	Mean Reading of volume of KMnO_4 solution (ml)
1.	10.0	0	10.1	10.1	
2.	10.0	10.1	22	11.9	10.1
3.	10.0	22	32.1	10.1	

Result :- Amount of Fe^{2+} in Mohr's salt is = 0.5040g Gm

Report :- • We have measure the volume of mohr's salt solution.
• Also we measure the volume of potassium permanganate properly.

DATE 23.11.21
EXP. NO. 04
PAGE NO. 25

Name of the Experiment
continued....

- In the end, we have to calculate the amount of Fe^{2+} in Mohr's salt.

Discussion: The main purpose of the experiment is to determination of ferrous ion (Fe^{2+}) with standard potassium permanganate solution. During this experiment we were needed to follow some important instruction such as :-

- We were advised to clean the apparatus properly with distilled water.
- We weighted Mohr's salt and H_2SO_4 properly as per as teacher's instruction.
- We were not allowed to lower tempere with other chemical on the lab.
- We were advised to be cautious while taking the readings of burette.
- The slightest pale pink colour obtained we were advised to show the solution to teacher then calculated the required result.
- After doing the experiment, we returned all the apparatus to the lab assistant after cleaning with clean water.
- While leaving the lab, we needed to ensure that all gas connections, electrical switches were kept in off mode.

~~* 22/11/2021~~

DATE 30-11-2021
EXP. NO. 05
PAGE NO. 26

Name of the Experiment Preparation of standard potassium dichromate solution and standardization of sodium Thiosulphate solution.

Objective: The objective of this experiment is -

- (i) To prepare standard solution.
- (ii) To standardize the unknown solution.

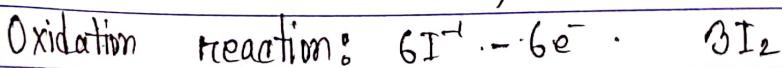
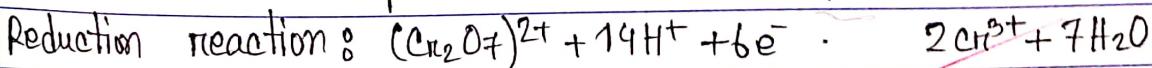
Apparatus and chemicals required:

- (1) Burette and pipette
- (2) Conical flask and volumetric flask.
- (3) Sodium Carbonate.
- (4) $K_2Cr_2O_7$ solution.
- (5) $Na_2S_2O_3$ solution.
- (6) 10% KI solution.
- (7) Distilled water.

Theory: In Iodometric titration some oxidizing agent liberate iodine and then liberated iodine is titration against standard solution of a reducing agent. This principle is employed to determine the strength of solution thiosulphate solution. When standard solution of $K_2Cr_2O_7$ reacts with KI in presence of acid, the dichromate is reduced to green chromic salt and liberated an equivalent amount of iodine which is titrated against sodium thiosulphate solution using strong solution as indicator when the end point reached the blue colour of starch iodo complex suddenly disappears and a pure green solution due to chromic salt is left. The reaction is -

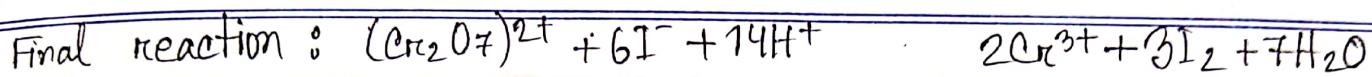


The electronic interpretation of above equation is follows:-



DATE 00-11-2021
EXP. NO. 05
PAGE NO. 27

Name of the Experiment continued....



Procedure: Prepare 100ml 0.1N $\text{K}_2\text{Cr}_2\text{O}_7$ solution in volumetric flask. Pipette out 100mL of standard $\text{K}_2\text{Cr}_2\text{O}_7$ solution. Dissolve about 10mL of 10% sodium carbonate solution into the conical flask. Add also 10mL of 10% KI solution and shake well for mixing and pour 10mL of 10% HCl slowly. Add it into the conical flask. Allow the flask in dark for about 5 minutes. Make up to the mark of supplied sodium thiosulphate solution and fill up the burette with it. We have to take out the conical flask from dark after 5 minutes. Reverse the watch glass. Dilute the solution about 50 ml with distilled water through inner wall of the conical flask. Titrate the liberated iodine with sodium thiosulphate solution. Add $\text{Na}_2\text{S}_2\text{O}_3$ dropwise until the brown colour becomes light yellow. Add 2ml starch solution and gently shake the flask and colour become deep blue. Wash the inside of conical flask and complete the titration by adding $\text{Na}_2\text{S}_2\text{O}_3$ solution. At the end point, the blue colour of the starch into complex will disappear and the colour of the solution will become chromic green. The end point is sharp.

Experiment Data: Preparation of 0.1N 250 ml $\text{K}_2\text{Cr}_2\text{O}_7$ Solution

Titration Table:

Figure No. 7

Calculation

Volume of $K_2Cr_2O_7$; $V_1 = 10 \text{ mL}$

Strength of $K_2Cr_2O_7$; $S_1 = \frac{\text{Weight taken}}{\text{Weight to be taken}} \times 0.01\text{N}$

Strength of $Na_2S_2O_3$; $S_2 = \frac{\text{Weight taken}}{\text{Weight to be taken}} \times 0.01\text{N}$

Strength of $Na_2S_2O_3$; $S_2 = \frac{0.0104 \times 10}{2.9} \times 0.01\text{N}$

Strength of $Na_2S_2O_3$; $S_2 = \frac{0.0104 \times 10}{2.9} \times 0.01\text{N}$

Strength of $Na_2S_2O_3$; $S_2 = \frac{0.0104 \times 10}{2.9} \times 0.01\text{N}$

Strength of $Na_2S_2O_3$; $S_2 = \frac{0.0104 \times 10}{2.9} \times 0.01\text{N}$

Strength of $Na_2S_2O_3$; $S_2 = \frac{0.0104 \times 10}{2.9} \times 0.01\text{N}$

Strength of $Na_2S_2O_3$; $S_2 = \frac{0.0104 \times 10}{2.9} \times 0.01\text{N}$

Strength of $Na_2S_2O_3$; $S_2 = \frac{0.0104 \times 10}{2.9} \times 0.01\text{N}$

Strength of $Na_2S_2O_3$; $S_2 = \frac{0.0104 \times 10}{2.9} \times 0.01\text{N}$

DATE ...

EXP. NO.

PAGE N

Titration

No. 03

observed

2

2

1

Result

X

USP
Udayan

DATE 30-11-2021
EXP. NO. 05
PAGE NO. 28

Name of the Experiment
continued....

Titration Table :

No. of observation	Volume of $K_2Cr_2O_7$ (ml)	Burette Reading IR	Burette Reading FR	Difference	Mean reading of volume of $Na_2S_2O_3$ solution in ml
1.	10.0	17.6	20.6	3	
2.	10.0	20.9	23.8	2.9	2.9
3.	10.0	24	26.9	2.9	

Result : The strength of $Na_2S_2O_3$ solution is 0.0358 N

Report : • We have measure the volume $K_2Cr_2O_7$

• Also we measure the volume of $Na_2S_2O_3$ properly.

• In the end we have to calculate the strength of $Na_2S_2O_3$

Discussion : The main purpose of the experiment is to preparation of standard potassium dichromate solution and standarization of sodium thiosulphate solution. During this experiment we were needed to follow some important instruction as :-

• We were advised to clean the apparatus properly with distilled water.

• We had to careful with the chemicals as they are strong.

• We need to be careful with the solution that we made into the conical flask.

• While taking the B.R. we need to be careful so that the calculation won't mess up.

• After 5 minutes when conical flask was taked out we needed to be titrated it very carefully with the mixing of stack untill the chromic green didn't show up.

• While leaving the lab we needed to check if the gas lines, electrical switches were open or not so that no accidents occur.