CHEMISTRY sessional

Course Code	**: CHE 0531-1202**	**Credits**	**03**
Exam Hours	**: 02**	**SEE Marks**	**50**

1. Course Learning Outcomes: at the end of the Course, the Student will be able to-

CLO1	Able to understand various apparatus and chemicals
CLO2	Understand the the quality of water
CLO3	Make student be more patitent
CLO4	Helps to work with hazardous environment

ASSESSMENT PATTERN

SEE- Semester End Examination (60 Marks)

ASSESSMENT STRATEGY		
Cos	Assessment Method	(100%)
Class Assessment		
1-4	Conduct of Lab Test /Class Performance	25%
1-4	Report Writing/Programming	15%
1-4	Mid-Term Evaluation (Exam/Project/assignment)	20%
1-4	Final Evaluation (Exam/Project/assignment)	30%
1-4	Viva Voce / Presentation	10%
	Total	100%

Instructions for Laboratory

- The objective of the laboratory is learning. You should understand this laboratory manual clearly and how to use it effectively. It is his duty to learn it carefully. Conduct the experiments with interest and an attitude of learning.
- You need to come well prepared for the experiment.
- Work quietly and carefully (the whole purpose of experimentation is to make reliable measurements!) and equally share the work with your partners.
- You must try to understand both the purpose and the principle behind each of the experiment you will do.

- You will organize your time effectively in advance of each experiment. As in the chemistry laboratory a student has to work with various hazardous chemicals, you should wear an apron during your seasonal work.
- Do no put inflamed stick of matches here and there. Smocking.
- All chemicals are toxic to some extent, so you should never eat or drink in the laboratory
- Handle instruments with care. Report any breakage to the Instructor. Return all the equipment you have signed out for the purpose of your experiment.

Week: 01
Topic: Flame test
Page no:

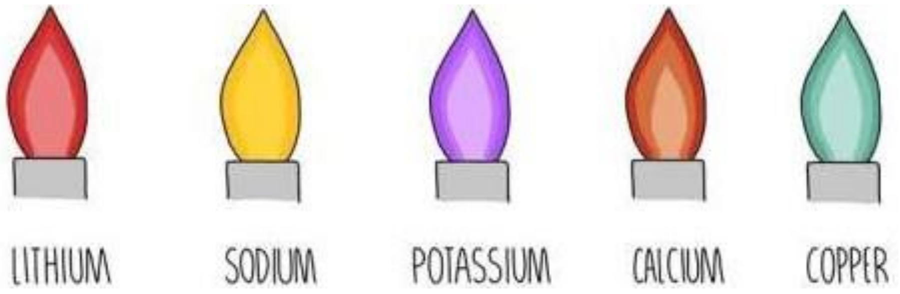
Identifying Ions

Some solid metal ions produce coloured flames when you heat them in a Bunsen burner. Many solutions containing metal ions undergo reactions where a precipitate is produced. We can use this knowledge to identify unknown metal ions in the lab.

Flame tests

A flame test is carried out by cleaning a **platinum** or **nichrome wire** by placing it in concentrated **hydrochloric acid**. We use platinum or nichrome because they have a **high melting point** (so it won‘t melt in the flame) and are **unreactive**. We place the acid-soaked wire into the centre of a hot Bunsen flame then place it into a sample of the metal we want to test. We then place it back into the **non-luminous flame** - it needs to be non-luminous (not very orange) so we can clearly see the colour.

- **Lithium**, Li^+ burns with a **red/crimson** flame
- **Sodium**, Na^+ burns with a **yellow-orange** flame
- **Potassium**, K^+ burns with a **lilac** flame
- **Calcium**, Ca^{2+} burns with an **orange-red** flame
- **Copper**, Cu^{2+} burns with a **green** flame

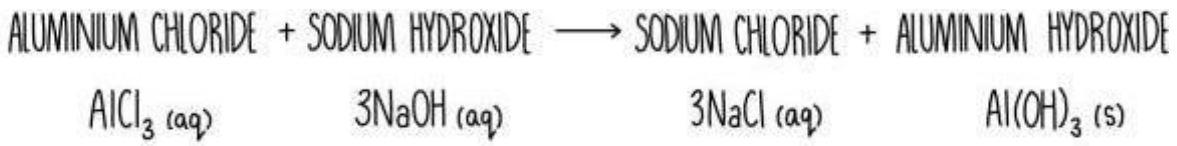


Week: 02 and 03
Topic: Cation and Anion test
Page no: 04-07

Cation and Anion test

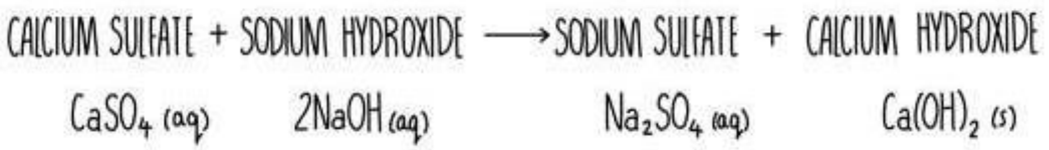
Metal hydroxides

We can also use **sodium hydroxide** solution to identify metal ions (cations). When you add sodium hydroxide to solutions containing **aluminium ions** (Al^{3+}), **calcium ions** (Ca>sup>2+) or **magnesium ions** (Mg^{2+}), **white precipitates** are formed. This is because aluminium hydroxide, calcium hydroxide and magnesium are all insoluble white solids. These reactions are examples of **displacement reactions**.



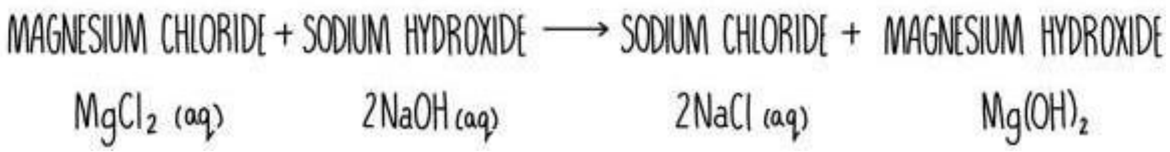
ALUMINIUM CHLORIDE + SODIUM HYDROXIDE ⟶ SODIUM CHLORIDE + ALUMINIUM HYDROXIDE

$AlCl_3\ (aq)$ $3NaOH\ (aq)$ $3NaCl\ (aq)$ $Al(OH)_3\ (s)$



CALCIUM SULFATE + SODIUM HYDROXIDE ⟶ SODIUM SULFATE + CALCIUM HYDROXIDE

$CaSO_4\ (aq)$ $2NaOH\ (aq)$ $Na_2SO_4\ (aq)$ $Ca(OH)_2\ (s)$



MAGNESIUM CHLORIDE + SODIUM HYDROXIDE ⟶ SODIUM CHLORIDE + MAGNESIUM HYDROXIDE

$MgCl_2\ (aq)$ $2NaOH\ (aq)$ $2NaCl\ (aq)$ $Mg(OH)_2$

Of the three hydroxides, only **aluminium hydroxide** precipitate **dissolves** in **excess sodium hydroxide** solution. This means that if you have a solution containing an unknown cation, you could test it by adding a small amount of sodium hydroxide. If a white precipitate is formed which is able to dissolve in excess sodium hydroxide solution, then you know that the solution contained aluminium ions.

Solutions containing **copper (II) ions** (Cu^{2+}), **iron (II) ions** (Fe^{2+}) and **iron (III) ions** (Fe^{3+}) form **coloured precipitates** when **sodium hydroxide** solution is added. Copper (II) forms a **blue** precipitate of **copper (II) hydroxide**, iron (II) forms a **green** precipitate of **iron (II) hydroxide** and iron (III) forms a **brown** precipitate of **iron (III) hydroxide**.

$$Cu^{2+} + 2OH^- \longrightarrow Cu(OH)_2$$

$$Fe^{2+} + 2OH^- \longrightarrow Fe(OH)_2$$

$$Fe^{3+} + 3OH^- \longrightarrow Fe(OH)_3$$

From left to right: iron (II) hydroxide, iron (III) hydroxide and copper hydroxide. Image: Kingston Grammar School

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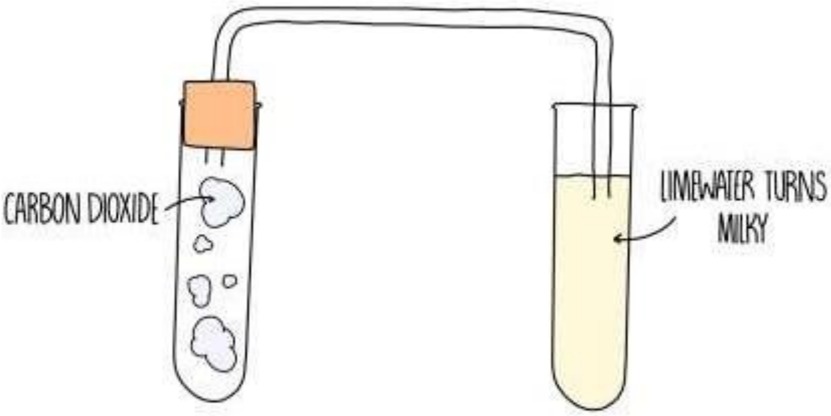
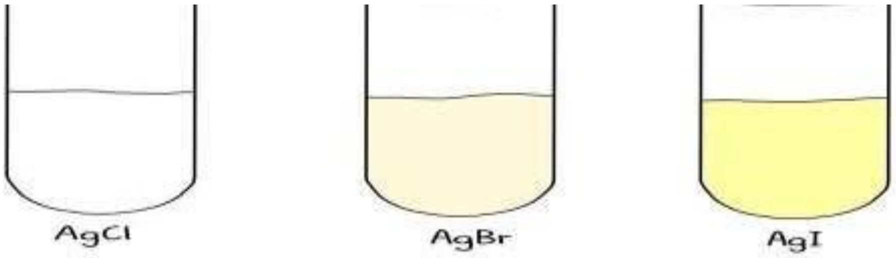


2. Carbonates

You can test to see if a compound is a carbonate by reacting it with a **dilute acid**.

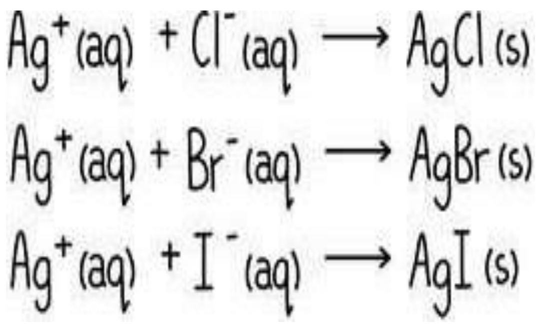
If the substance is a carbonate, **carbon dioxide gas** will be formed which can be identified by **bubbling through limewater**. If the limewater turns **milky** then the gas is carbon dioxide.

ACID + CARBONATE ⟶ SALT + WATER + CARBON DIOXIDE



3. Halides

We can test for the **halide ions** (the ions formed from the group 7 elements) using **dilute nitric acid** followed by **silver nitrate.** We add dilute nitric acid to remove any carbonate or sulfate ions which would give a false positive result. Silver ions react with the halide ions to form precipitates of silver chloride, silver bromide or silver iodide which deepen in colour from **white**, **cream** then **yellow**. The equations for these reactions are:



$$Ag^+(aq) + Cl^-(aq) \longrightarrow AgCl(s)$$
$$Ag^+(aq) + Br^-(aq) \longrightarrow AgBr(s)$$
$$Ag^+(aq) + I^-(aq) \longrightarrow AgI(s)$$

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4. Sulfates

Sulfate ions are tested by adding **dilute hydrochloric acid** followed by **barium chloride**. The purpose of the acid is to remove any carbonate ions that may give a false positive result. The reaction between sulfate ions and barium chloride forms **barium sulfate** which is an insoluble **white precipitate.**

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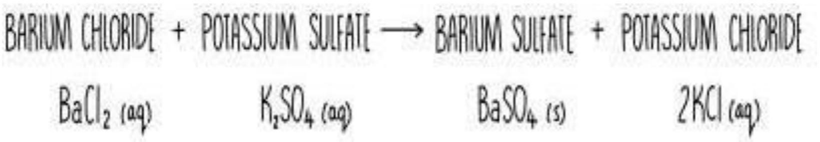


Week: 04-16

Topic: Titration

Page: 07-28

Standardization of Sodium Hydroxide solution with standard Oxalic Acid solution.

Objectives:

i. To study the neutralization reaction
ii. To observe the quantity of acid or base needed for neutralization.

Theory: In this experiment we shall determine the strength of NaOH solution by a standard solution of Oxalic Acid. This is done by means of ―Titration‖. The important matters that are related with the experiment are stated below:

Titration: In presence of a suitable indicator, the volumetric analysis in which a standard solution is added in another solution (whose strength is not known) to reach its end point to determine the strength of that solution is called _titration‘.

Standard Solution: A solution of known concentration is called a _standard solution.

Indicator: In our acid-base titration there is an important use of indicator. An _indicator‘ is a chemical substance that detects the equivalent point (i.e. the end point) of reaction by changing its color.

Equivalent Point: The _equivalent point‘ is the point in a titration when a stoichiometric amount of reactant has been added.

Normality (N) = gm equivalent of solute /liters per solution.

In this experiment the reaction we shall use is as follows:

HOOC-COOH + 2NaOH ——> NaOOC-COONa + $2H_2O$

The formula required to determine the strength of NaOH solution is:-

$V_{base} \times S_{base} = V_{acid} \times S_{acid}$(For 1:1 mole ratio)

Modified formula for Oxalic acid and Sodium hydroxide will be:

$V_{base} \times S_{base} = 2V_{acid} \times S_{acid}$

M_{acid} = Molarity of the acid, M_{base} = Molarity of the base

V_{acid} = Volume of the acid, V_{base} = Volume of the base

The volume of Oxalic acid is measured by watching the Equivalent point. The point at which acid-base neutralizes each other is called ―Equilibrium point‖. This point is determined with the help of an indicator.

Why Phenolphthalein is used as indicator: In this experiment, ‗Phenolphthalein' is selected as indicator whose working pH range is 8.3-10.0 i.e. it is works when the environment is base. This indicator gives pink color in basic solution and becomes colorless when the base is neutralized.

Indicator name	**pH range**	**Color in Alkaline solution**	**Color in Acid solution**
Phenolphthalein	8.3-10.0	Pink	Colorless

In this experiment we are using NaOH and Oxalic acid. NaOH is a strong base but Oxalic acid is a weak acid. So the solution at equilibrium point consists of a salt whose basic part is strong. As a result there will be more OH- in the solution than H+ as the salt will be dissociated in the aquas solution. So, the solution would be basic which provides phenolphthalein to work properly. So Phenolphthalein becomes the perfect indicator to determine the end point of this reaction.

Procedure: Take 10 ml of supplied NaOH solution in a conical flask by means of a pipette and dilute it to about 50 ml. Add one drop of phenolphthalein indicator to the solution. Then add standard oxalic acid solution drop by drop from a burette. Shake the flask frequently while adding the acid solution. Stop the addition of oxalic acid solution as soon as the pink color of the solution just disappears. Note the burette reading. The burette reading should be taken carefully at the lower meniscus of the liquid. Difference of the initial and final burette reading gives the volume of the acid added. The process should be repeated at least twice. These should agree within ±0.1 ml. calculate the normality of the supplied NaOH solution. Using the following relation.

Data Table:-

Standardization of NaOH solution with standard Oxalic Acid Solution

No. of Obs.	**Volume of NaOH(ml)**	**Burette reading in ml**			**Average Reading (ml)**	**Strength of NaOH (M)**
		Initial Reading	**Final Reading**	**Differ.**		
1	10.00	6.88	16.45	9.57	9.63	
2	10.00	16.45	26.10	9.65		
3	10.00	30.20	39.87	9.67		

Calculation:—

We know

$V_{\text{Oxalic-acid}} \times S_{\text{Oxalic-acid}} = V_{\text{NaOH}} \times S_{\text{NaOH}}$

Result:—

Strength of NaOH solution is: S_{NaOH} = M

Students should know:

a. What is normality of supplied solution?
b. What are the molecular structure of phenolphthalein and oxalic acid?

c. How does the indicator (phenolphthalein) work?
d. At what pH does the indicator changes it color?
e. Do you know why we see different color of different solutions?

Standardization of Commercial Hydrochloric Acid (HCl) solution with standard Sodium Hydroxide (NaOH) solution.

Objectives:

1. To study the strength of commercial hydrochloric acid
2. To observe the quantity of acid or base needed for neutralization.
3. To study secondary standard substance for titration

Theory: The strength determination of both commercial Hydrochloric acid and NaOH is done by means of titration. In presence of a suitable indicator, a chemical substance that detects the end point of a reaction by changing its color, the volumetric analysis in which a standard solution is added in another solution to reach its end point and to determine the strength of that solution is called titration. The reaction between NaOH and Oxalic acid is –

$$2NaOH + HOOC\text{-} COOH \longrightarrow NaOOC\text{-} COONa + 2H_2O$$

Indicator – Phenolphthalein, and the reaction between HCl and NaOH is -

$$NaOH + HCl \longrightarrow NaCl + H_2O$$

Indicator – Methyl Orange

Procedure: Take 10 ml standard NaOH solution in a conical flask and dilute it to about 50 ml. Add 2 or 3 drops of methyl orange indicator to the solution. Then add previously prepared (approx. M/10) HCl acid solution drop wise from a burette. Shake the flask frequently during addition of HCl acid. Stop addition of HCl acid solution as soon as the yellow color of the solution just changes to orange. Note the burette reading. Repeat the process at least two times and these should agree within ± 0.1 ml. Calculate the normality of the dilute HCl and from there calculate the strength of commercial HCl.

Experimental Data: Table-1

Standardization of NaOH solution with standard Oxalic Acid solution

No. of Obs.	**Volume of NaOH (ml)**	**Burette reading in ml**			**Average Reading (ml)**	**Strength of NaOH (M)**
		Initial Reading	**Final Reading**	**Differ.**		
1						
2						
3						

Table-2: Standardization of HCl solution with standard NaOH solution

No. of Obs.	**Volume of NaOH (ml)**	**Burette reading in ml**			**Average Reading(ml)**	**Strength of HCl (M)**
		Initial Reading	**Final Reading**	**Differ.**		
1	10.00	32.10	38.50	6.40	6.34	
2	10.00	38.50	44.62	6.12		
3	10.00	0.00	6.50	6.50		

Calculation: -

We know that, a) $V_{acid} \times S_{acid} = V_{base} \times S_{base}$

We also know, b) $V_{\text{concentrated acid}} \times S_{\text{concentrated acid}} = V_{\text{dilute acid}} \times S_{\text{dilute acid}}$,

Result: The strength of HCl (dilute) is found: M

The strength of the supplied HCl (concentrated) is: M

Percentage of error:

$$\text{Percentage of error} = \frac{(Known\ Value - Observed\ Value)}{Known\ Value} \times 100\%$$

Discussion: The following causes can be assumed for the possible cause of error.

1) It is very difficult to determine the end point accurately. So the measured value is deviated from the actual value due to either over titration or under titration. This can also be one of the reasons of the error. This is the main cause of error.

2) Another cause of error may rise from faulty burette reading.

If these causes could be avoided, we could have got more accurate result of the strength of Commercial Hydrochloric Acid.

Student should know

a. What is the normality of the supplied HCl solution?
b. What is the molecular structure of methyl orange indicator?
c. Can we use phenolphthalein instead of methyl orange here? Justify your answer.
d. At what pH does the indicator changes its color?
e. Is it necessary to use buffer solution here? If not why?

Standardization of Hydrochloric Acid with Standard Sodium Carbonate Solution

Objectives:

1. To observe the molar ratio of acid or base needed for neutralization.
2. To study effect of different indicators and end point.

Theory: In this experiment we shall determine the strength of commercial Hydrochloric Acid solution by a primary standard solution of Na_2CO_3.

This is done by means of _Titration‘. The important matters that are related with the experiment are stated below:

Titration: In presence of a suitable indicator, the volumetric analysis in which a standard solution is added in another solution (whose strength is not known) to reach its end point to determine the strength of that solution is called _titration‘.

Standard Solution: A solution of known concentration is called a _standard solution‘.

Indicator: -In our acid-base titration there is an important use of indicator. An _indicator‘ is a chemical substance that detects the equivalent point (i.e. the end point) of reaction by changing its color. Indicators have different structures in acidic and in basic solution.

Equivalent Point: - The _equivalent point‘ is the point in a titration when a stoichiometric amount of reactant has been added.

Normality: The number of gram equivalent weight of a solute per liter of solution is called normality.

Normality (N) = gram equivalent of solute /liter solution.

It is known to us that both alkalimetry and acidimetry are based on neutralization reaction.

Neutralization reaction between Na_2CO_3 and HCl acid takes place into two steps-

$Na_2CO_3 + HCl \Rightarrow NaHCO_3 + NaCl$

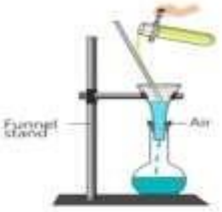
$NaHCO_3 + HCl \Rightarrow NaCl + H_2CO_3$

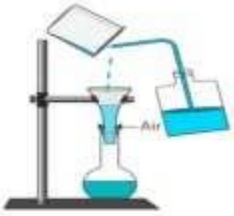
The ultimate reaction,

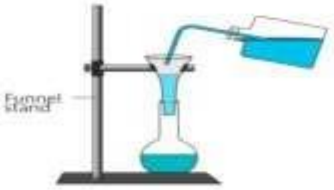
$Na_2CO_3 + 2HCl \Rightarrow 2NaCl + H_2CO_3$

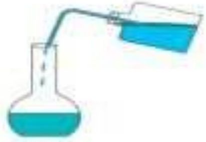
In the first step, the solution is basic due to the formation of a salt where the basic part is stronger than the acidic part ($NaHCO_3$). So, in order to determine the equivalent point of this reaction Phenolphthalein is used. As the salt that forms due to the neutralization reaction, produces more OH-, so the solution becomes a basic one and thus it has a pH range above 7. We know that the working environment needed for phenolphthalein is basic; thus, phenolphthalein becomes the perfect indicator for determining the end point of the first step of the reaction. In the second reaction, NaCl and Carbonic Acid is formed. Because of the presence of Carbonic Acid in the solution, it becomes acidic. So, _Methyl Orange‘ (pH range 2.9-4.6) is used as indicator to determine the equivalent point.

Indicator name	**pH Range**	**Color in Alkaline solution**	**Color in Acid solution**
Phenolphthalein	8.3 - 10.0	Pink	Colorless
Methyl Orange	2.9 - 4.6	Yellow	Red







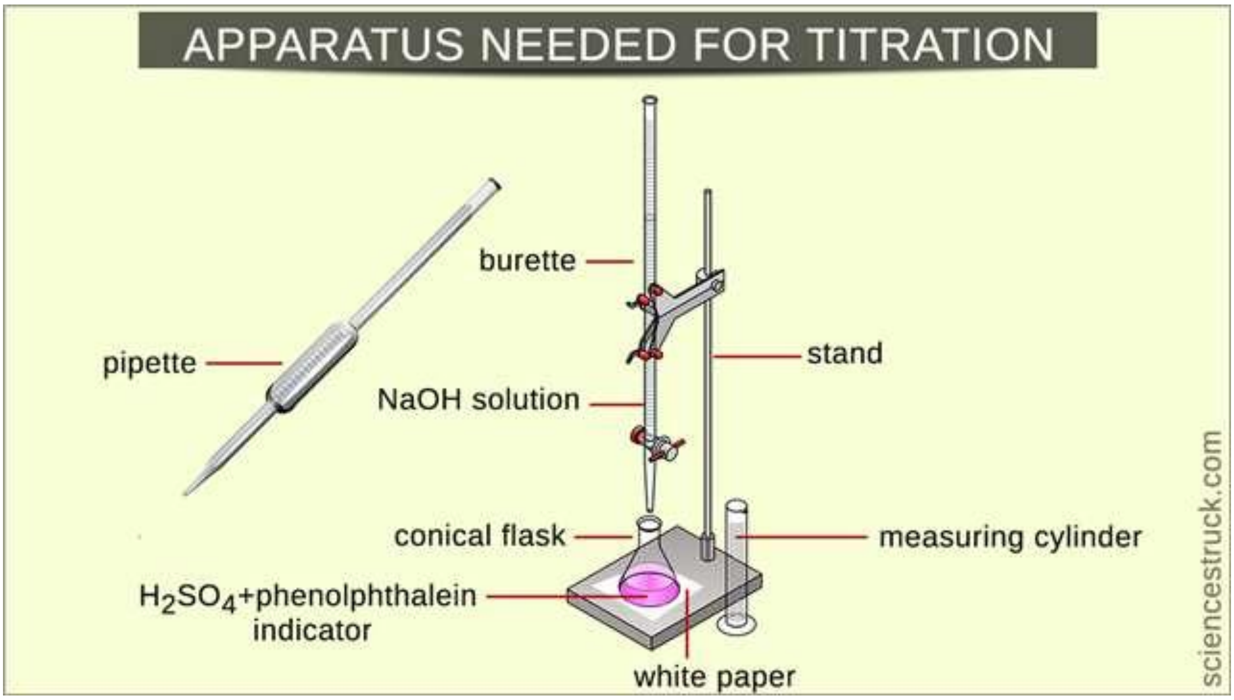


Materials Required

1. Chemical balance
2. Watch glass
3. Weight box
4. 250ml beaker
5. Glass rod
6. 250ml measuring flask
7. Wash bottle
8. Weighing tube
9. Sodium Carbonate
10. Funnel
11. Funnel stand
12. Distilled water

Procedure:

1. Take 10 ml of standard Na_2CO_3 solution in a conical flask and dilute it to about 50 ml.
2. Add one drop of phenolphthalein
3. Titrate against HCl contained in a burette.
4. Now note the burette reading when just one drop of HCl discharges the pink color of the solution. This is the first end point. (no need take reading)
5. Then add 3-4 drops of methyl orange inside the same conical flask and continue titration against the same HCl acid.
6. The end point reached when the yellow color of the solution just changes to faint pink (orange). Note the burette reading. This is the second end point.(Final reading)
7. The difference of the burette reading from initial to final end point will be the volume of the acid required in the titration.
8. Repeat the whole experiment twice and these should agree within ± 0.1 ml initial to final end point.
9. Calculate the strength of dil. HCl and then find out the normality of commercial conc. HCl acid.



Experimental Data: Standardization of HCl acid with standard Na_2CO_3 solution

No. of Obs.	Volume of Na_2CO_3 (ml)	Burette Reading (ml)			Average (ml)	Strength of HCl (M)
		Initial reading	Final reading	Differ.		
1	10.00	0.00	12.25	12.25	12.23	0.082
2	10.00	12.25	24.56	12.31		
3.	10.00	24.56	36.68	12.12		

We know that, $V_{acid} \times S_{acid} = 2.V_{base} \times S_{base}$

Result: The strength of the supplied HCl ………. M

Percentage of error:

$$\text{Percentage of error} = \frac{(Known\ Value - Observed\ Value)}{Known\ Value} \times 100\%$$

Discussion: -The following causes can be assumed for the possible cause of error:
While the solution of Na_2CO_3 was prepared, a little amount of extra water might have been added into the volumetric flask, this can be one of the reasons.
If these causes could be avoided, we could have got a perfect result of the concentration of Commercial Hydrochloric Acid.

Students should know:

a. Is Na_2CO_3 a primary standard substance?
b. Why do you need to use two indicators here?
c. Can you use methyl orange first instead of phenolphthalein? Justify your answer.
d. Can you calculate the normality or molarity from the half reaction?

Standardization of Sodium Thiosulphate solution with standard Potassium Dichromate solution.

Objectives:

1. To study the strength of thiosulphate.
2. To study oxidation reduction titration.
3. To study the liberation and its titration.

Theory: -Standardization is the process by which the strength of a solution is determined with the help of a standard solution. A solution of known concentration is called a standard solution. This experiment is done by means of titration. In presence of a suitable indicator, a chemical substance that detects the end point of reaction by changing its color, the volumetric analysis in which a standard solution is added in another solution (whose strength is unknown) to reach its end point to determine the strength of that solution is called titration. Titration involving iodine or dealing with liberated iodine in chemical reaction is called iodimetry and iodometry respectively.

This reaction is iodometric because iodine is obtained from KI.
The reactions of this experiment are:

$$6KI+14HCl+ K_2Cr_2O_7 =2CrCl_3+3I_2 +7H_2O+8KCl$$
$$(6\ I^- + 14\ H^+ + Cr_2O_7^{-2} + 6e^- = 2\ Cr^{+3} + 3\ I_2 + 7\ H_2O)$$

$$2Na_2S_2O_3 + I_2 = Na_2S_4O_6 + 2NaI$$
$$(S_2O_3^{2-} - e^- = S_4O_6^{2-} + I^-)$$

Here $K_2Cr_2O_7$ is an oxidizing agent and I^- is a reducing agent. Again in the second reaction I_2 is an oxidizing agent and $S_2O_3^{--}$ is a reducing agent. In the 2nd Step of the reaction a specific indicator is used that is ―Starch‖- which has a significant effect on iodine.

$$Starch+I_2=Starch\text{-}Iodine\ (blue\ color)$$

Starch-Iodine+$6S_2O_3$--=Starch +$6I^-$ + $3S_4 O_6^{--}$

If to a solution containing a little iodine, some starch solution is added and $Na_2S_2O_3$ is run in from the burette, the blue color of the starch-iodine complex will disappear from the solution as soon as all the iodine has been reduced to iodide ion.

Procedure:

Prepare 0.05 M $K_2Cr_2O_7$ solution. (MW=294)

1. Take 4 ml of 12%potassium iodide (KI) solution in a conical flask and
2. Dilute it to about 50 ml.
3. Add about 1 g. of $NaHCO_3$ and shake the flask until the salt dissolves.
4. Add about 4 ml. conc. HCl acid and
5. Then add 10 ml. standard $K_2Cr_2O_7$ solutions by means of a pipette in the same flask and cover it with a watch glass, allow the solution to stand for about five minutes in the dark (inside the desk). Rinse the watch glass and dilute the solution about 100 ml. titrate the liberated iodine with sodium thiosulphate solution from burette until the brown color fades (light yellow). Add about 1 ml starch solution and continue titration by adding sodium thiosulphate solution from the burette until one drop of sodium thiosulphate solution changes the color of the solution from deep blue to light blue. This is the end point. Calculate the strength of sodium thiosulphate solution using the following equation: $V_1 \times S_1 = V_2 \times S_2$

Data:-Standardization of $Na_2S_2O_3$ solution with standard $K_2Cr_2O_7$solution

No. of obs.	**Volume Of $K_2Cr_2O_7$(ml)**	**Burette Reading (ml)**			**Average (ml)**	**Strength of $Na_2S_2O_3$ (M)**
		Initial Reading	**Final Reading**	**Differ.**		
1	10	0.00	10.20	10.20	10.16	0.0082 M
2	10	10.20	20.10	9.90		
3	10	20.10	30.50	10.40		

Calculation:-

We know, $V_{Na2S2O3} \times S_{Na2S2O3} = 6V_{K2Cr2O7} \times S_{K2Cr2O7}$

$V_{Na2S2O3}$ = 10.16 ml
$S_{Na2S2O3}$ = ? 0.0082 M
$V_{K2Cr2O7}$ = 10 ml
$S_{K2Cr2O7}$ = 0.05 M

Result: - Determined strength of $Na_2S_2O_3$ solution is = 0.295 M (Real value: 0.275M)

Error:

Percentage of error $= \frac{(Known\ Value - Observed\ Value)}{Known\ Value} \times 100\ \%$

Discussion: -As the color change of the titration of $Na_2S_2O_3$ with $K_2Cr_2O_7$ is very confusing, the end point of the titration may not have been properly determined. This may be the cause of error.

Student should know:

a. How does a redox indicator work?
b. What is the difference between acid-base and redox indicator?
c. Can you identify whether KI, $K_2Cr_2O_7$, $NaHCO_3$ are primary or secondary substance?
d. Can you use any other chemicals instead of $NaHCO_3$?
e. Why you should cover the conical flask after adding $NaHCO_3$?
f. Why it is necessary to keep your experimental solution in the dark?
g. Why you observe brown color for liberation of iodine instead of deep violet color?
h. How do you say which oxidizing / reducing agent stronger or weaker than other?
i. What is the function of starch and why is the necessary to add starch just before the end point of the titration?

Estimation of Copper contained in a supplied solution by Iodometric method.

Objectives:

1. To study the amount of copper present in copper salt solution.
2. To study oxidation reduction titration.
3. To study the percentage of copper present.

Theory: In this experiment, the amount of copper in a copper salt solution is determined. The reaction that occurs here is oxidation and reduction reaction as well as iodometric reaction. Titration involving with iodine or dealing with iodine liberated in chemical reaction is called Iodimetric and Iodometric titration respectively. This reaction is iodometric because iodine is got from KI. The reaction takes place in two steps:

a. $Cu^{2+} + KI + H^+ = I_2 + \ldots\ldots$

b. $Na_2S_2O_3 + I_2 = Na_2S_4O_6 + \ldots\ldots$

In the 2nd Step of the reaction a specific indicator is used that is ―Starch‖- which has a significant characteristic on iodine.

Here, for the first part of the experiment, the basis of the volumetric measurement is that one equivalent weight of an oxidizing agent will completely react with one equivalent weight of a reducing agent. To perform the second part of the experiment, we can follow the process discussed below:

Since one atom of iodine is set free for each atom of copper 1000 ml (1M) $Na_2S_2O_3$ solution should be equivalent to one-gram molecular weight that is 63.54 gram of copper.

Therefore

1 ml of (1M)$Na_2S_2O_3 \equiv$ 0.06354 gram of Copper ($Cu+^2$)

V ml (SM) $Na_2S_2O_3$ estimated = (VS/1000)* 63.54 g Cu^{+2}

So, 10 ml supplied solution contained ………………… g Cu^{+2} ion.

1000 ml supplied solution contained …………….. g Cu^{+2} ion

So, the weight of copper in the supplied solution may then be easily calculated using the above relation.

Procedure:

Standardization of Sodium Thiosulphate solution as experiment no. 04

0.0082 M

Pipette out 10 ml of supplied copper salt solution into a conical flask.

Add a few drop of dil. NaOH or $NaHCO_3$ or Na_2CO_3.

A pale greenish precipitate should appear.

Dissolve the precipitate by adding

few drop of acetic acid (CH_3COOH).

Add about 10 ml of 10% potassium iodide (KI) solution

and titrate the liberated iodine against the standard thiosulphate solution (standardized previously) until the brown color of iodine changes to light yellow. Add 1 ml of starch solution and continue titration till the blue color begins to fade. Now add few drops of 10% NH_4SCN solution and continue titration until the blue color is just discharged. Calculate the amount of copper present in one liter of the supplied solution.

Data: Table 1

Standardization of Sodium Thiosulphate solution with standard Potassium Dichromate Solution

No. of obs.	**Volume of $K_2Cr_2O_7$ (ml)**	**Burette Reading (ml)**			**Average (ml)**	**Strength of $Na_2S_2O_3$(M)**
		Initial	**Final**	**Difference**		
1						

2						
3						

Calculation:

TABLE 2: Estimation of Cu^{2+} solution with standard $Na_2S_2O_3$ solution.

No of obs.	**Volume of Cu^{2+} Soln(ml)**	**Burette Reading (ml)**			**Average (ml)**	**Amount of Cu^{2+} Soln. (g/L)**
		Initial Reading	**Final Reading**	**Differ.**		
1	10	0.00	10.20	10.20	10.16	**38.00**
2.	10	10.20	20.10	9.90		
3	10	20.10	30.50	10.40		

Calculation:—

from $V_{red} \times S_{red} = V_{ox} \times S_{ox}$

$S_{Na2S2O3} \times V_{Na2S2O3} = S_{Cu2+} \times V_{Cu2+}$

$S_{Na2S2O3}$ = 0.0082 M

$V_{Na2S2O3}$ = 10.23 ml

S_{Cu2+} = ?

V_{Cu2+} = 10 ml

S_{Cu2+} = **0.0083 M**

= (0.0083 × 63.57**) g**

= 0.533 g Cu2+

10 ml Cu2+ solution contain = 0.53 g Cu2+

1000 ml ………………………… = 53.30 g/L

Result: The amount of Cu^+ (in g) present in the supplied $CuSO_4$ solution (per liter) is: W_{cu^+} = …. G

Error: Percentage of error $= \frac{(Known\ Value - Observed\ Value)}{Known\ Value} \times 100\%$

Discussion: The following may be the possible cause of error:

There might have been a little mistake in determining the end point of titration.

Students should know:

a. What is the purpose of adding the Na_2CO_3 and CH_3COOH solution?
b. What is desirable pH for the reaction between Cu^{2+} and KI?
c. What is the purpose of during NH_4SCN solution?
d. Do you know why most of the copper salts have blue color?

Determination of Ferrous Iron (Mohr's Salt) by Standard Potassium Dichromate Solution

Mohr's salt: $FeSO_4.(NH_4)_2SO_4.6H_2O$
Fe^{2+}

Objectives:

1. To study the amount of Ferrous Iron present in salt solution.
2. To study oxidation reduction titration.
3. To study the percentage of Iron present.

Theory: In this experiment, the amount of iron in a supplied solution is determined with the help of standard potassium dichromate.

The reaction that occurs here is oxidation and reduction reaction. Oxidation-reduction reactions are those in which there is a net change in the oxidation number. Or in other word an oxidation-reduction reaction (redox) is a reaction in which electrons are transferred between species or in which atoms change oxidation numbers. Oxidation is the half reaction in which there is loss of electrons by a species (or increase of oxidation number of an atom). Reduction is the half reaction in which there is a gain of electrons by a species (or decrease of oxidation number of an atom). Reaction involved in this reaction is:

$$K_2Cr_2O_7 + 14H^+ + 6Fe^{2+} \equiv 2K^+ + 2Cr^{3+} + 7\ H_2O + 6Fe^{3+}$$

In this reaction, Fe^{+2} is oxidized into Fe^{+3}. And $Cr_2O_7^{-2}$ is reduced to Cr^{+3}.

From the above reaction we get:
1 equivalent weight of $K_2Cr_2O_7$≡ 1 equivalent weight of Fe^{+2}
1000 ml (1 M) $K_2Cr_2O_7$solution ≡1000 ml (1M) ferrous iron solution
≡ 1 atomic weight of iron
So, 1000 ml (1M) $K_2Cr_2O_7$ solution should be equivalent to

Fe = 55.85 g

one gram atomic weight that is 55.85 gram of iron.

Therefore, 1 ml of (1M) $K_2Cr_2O_7$ ≡ 0.05585 gram of iron
So, the weight of iron in the supplied solution may then be easily calculated using the above relation.

INDICATOR AND WHY WE USED: In this experiment **di-phenyl amine** is used. A serious limitation to the use of $K_2Cr_2O_7$ as a titrant is that the end point in the resultant green chromic ion solution is not easily detected. This limitation has been overcome by the use of an internal oxidation reduction indicator and of the potentiometric titration method for detecting the end point. This indicator may be used as internal indicator to determine the end point in titration with dichromate. It is oxidized less readily than ferrous ion. So for proper detection of the end point di-phenyl amine is used here as an indicator.
With this indicator, phosphoric acid must be added in the solution. Because it lowers the reduction potential of the ferrous-ferric system. The Reduction Potential of di-phenyl benzidine is 0.76 volt and the Reduction Potential of ferrous ion is 0.77 volt. Although this potential difference is quite low, but this low difference can cause significant error in determining the weight of ferrous ion. To reduce this potential difference for getting proper result, phosphoric acid is used. So that the two potential can almost coincide. Phosphoric acid lowers the difference between two reduction potential by forming a complex with the ferric ion which is $[Fe(HPO_3)]^{+}$.

Procedure:

Step-1: (Preparation of 100 ml 0.02M $K_2Cr_2O_7$ solution)

1000 ml 1M $K_2Cr_2O_7$ solution contains = 294 g

$$W = \frac{C \times M \times V}{1000}$$

C = concentration = 0.02M
M = Molecular weight = 294
V = 100 ml

Therefore, 100 ml 0.02M solution contains = $\frac{294\times100\times0.02}{1000}$ = 0.588g

So, the strength of prepared $K_2Cr_2O_7$ = $\frac{weight\ Take(g)}{weight\ to\ be\ take(0.588)}\times$ 0.02M

Step-2: Titration of the supplied Iron Solution against $K_2Cr_2O_7$ Solution:

1. Take 10 ml of the supplied iron (Mohr‘s salt) solution in a conical flask.
2. Add 50 ml 1M sulfuric acid and 5 ml conc. of phosphoric acid.
3. Then add 4-5 drops of diphenyl amine indicator and titrate slowly against standard potassium dichromate solution drop wise maintaining an interval of few second between each drop until the addition of one drop causes the formation of intense purple or violet blue coloration which remains permanent.

4. Record the burette reading in the table below.
5. Repeat the steps 1-4 at least twice
6. Calculate the amount of Iron content in the solution.
7. Calculate the amount of iron per liter of solution.

Data: Determination of Ferrous Iron by Standard Potassium Dichromate Solution

No. of Obs.	Volume Of Fe^{2+} Soln (ml)	Burette Reading (ml)			Average (ml)	Amount of Fe^{2+}(g/L)
		Initial	Final	Difference		
1.	**10**	**0.00**	**10.20**	**10.20**	**10.30**	**690.30**
2.	**10**	**10.20**	**21.50**	**11.30**		
3	**10**	**21.50**	**30.90**	**9.40**		

Calculation:

$S_1 V_1 = S_2 6V_2$ S1 = concentration of Fe = ?
V1 = Volume of Fe solution = 10 ml
S2 = Conc. Of $K_2Cr_2O_7$ = 0.02 M
V2 = Volume of $K_2Cr_2O_7$ = 10.30 ml

S1 = 0.1236 M Fe^{2+}
= 0.1236 × 55.85 g
= 6.903 g

10 ML solution contain = 6.903 g Fe^{2+}
1000……….. = 6.903× 1000/ 10
= 690.30 g/L

Volume of $K_2Cr_2O_7$ required to titrate 10 ml of Sample= V ml
1 mole $K_2Cr_2O_7$= 6 mole of Fe(II)
1000 ml of 1 (M) $K_2Cr_2O_7$ = 1 mole of F (II)
V ml of S (M) $K_2Cr_2O_7$ = (V×S/1000) mole Fe^+ (II)
= (VS/1000) ×55.84 g
= X g Fe^+ (II)
10 ml of sample contains = X g Fe^+ (II)
1000 ml of sample contains =1000 × X /10gFe (II)
= y gFe (II)

Result: The amount of iron (in g) present in the supplied Fe^{+2}solutn (per liter) is: W $_{\overset{+}{Fe}}$ = 114.5g/L

Error:

$$\text{Percentage of error} = \frac{(Known\ Value - 0bserved\ Value)}{Known\ Value} \times 100\ \%$$

Precautions: At first the burette was cleaned with distilled water carefully; then it was rinsed with rinse solution to avoid the density change of the given solution. In the same way the pipette was also washed with distilled water and rinsed with the rinse solution.

a. All the apparatus was handled carefully and according to the rules.
b. The pipette was kept on a clean sheet of paper.
c. The key of the burette was operated carefully to make sure that one-drop of. Acid falls into the conical flask at a time.
d. The conical flask was kept on a white paper to trace the color change of the solution.
e. At first the key of the burette was fully opened to let the solution fall free to make the bubbles go out.
f. If any air bubble enters the burette while pouring acid, air bubble should be removed by dropping out the solution forcibly until the air bubble is out of the burette. Or else, the presence of air bubble will hamper the reading of the volume.
g. Reading might be wrong due to poor eye sight and non-vertical readings. The readings must be taken vertically considering the lower meniscus of the concave surface of the

liquid.

Discussion: This experiment is performed in order to determine the ferrous iron by standard potassium dichromate solution. On the basis of the experiment it was found that $K_2Cr_2O_7$ is a primary standard substance. We observe that there is slight deviation of the obtained result from the original one. The following factors may be mentioned for this deviation:

1. On the basis of the result it was found that the balance is very much sensitive. So for this reason the result can be varied.
2. While measuring the lower meniscus of the burette an error may be happened for the parallax error. So, concentration should be needed here.
3. The phosphoric acid and the sulphuric acid should not be mixed in the solution more than it is needed. If this happened then an error can be taken place. The end point of the titration of the experiment will come later.
4. As the color change is very confusing, the end point of titration may not have been properly determined.
5. Concentrated phosphoric acid must be added in order to lower the difference between two substances‘ standard reduction potential. If it is not done then it will hamper to determine the weight of ferrous ion correctly.
6. After pouring any substance in the conical flask with the help of the pipette, if there is any liquid at the tip of the pipette, it should be ignored. But by no means should it be blown out.
7. If the solution is not properly mixed with water then error can occur in determining the weight of ferrous ion. This may be attributed as a reason of error in our experiment

Students should know: -

a. Why it is necessary to use both the sulfuric acid as well as phosphoric acid in the experiment?
b. Do you know the equation corresponding to the color changes of diphenyl amine indicator?
c. What causes the intense purple or violet coloration of the experimental solution?
d. Could you use $KMnO_4$ instead of $K_2Cr_2O_7$ and what advantage does $K_2Cr_2O_7$ has over $KMnO_4$?
e. Why the solution shows light bottle green color after addition of $K_2Cr_2O_7$ and also after the end point is achieved?

Standardization of Potassium Permanganate solution with Standard Sodium Oxalate Solution.

Objectives:

1. To study the strength of Potassium Permanganate with standard Sodium Oxalate solution.
2. To study oxidation reduction titration.

Theory: In this experiment, potassium permanganate solution is standardizing with the help of standard sodium oxalate solution. The reaction that occurs here is oxidation and reduction reaction. An oxidation-reduction reaction (redox) is a reaction in which electrons are transferred between species or in which atoms change oxidation numbers. Oxidation is the half reaction in which there is loss of electrons by a species (or increase of oxidation number of an atom). Reduction is the half reaction in which there is a gain of electrons by a species (or decrease of oxidation number of an atom).

Reaction involved in this reaction is:

MW of $C_2O_4Na_2 = 134$

$$MnO_4^{-} + 8\,H^{+} + 5\,e^{-} \rightarrow Mn^{+2} + 4\,H_2O$$

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

In this reaction, MnO^{4-} is reduced to Mn^{+2} and $Na_2C_2O_4$ is oxidized to CO_2.

The following equation is used to calculate the strength of Potassium Permanganate:

$$V_A \times S_A = V_B \times S_B$$

Here,

V_A = Volume of Potassium Permanganate

S_A = Strength of Potassium Permanganate

VB = Volume of Sodium Oxalate

SB = Strength of Sodium Oxalate

INDICATOR AND WHY USED: The direct reaction is slow as one can see in a titration. The first few drops of permanganate added to the acidified oxalate solution are not decolorized immediately.

Mn^{+2} ions produced in the reaction acts as a catalyst.

They react with permanganate to form intermediate oxidation states of manganese. These states, in turn, react rapidly with oxalate to give the products. So, $KMnO_4$ acts as an auto catalyst in this reaction. This is the advantage of $KMnO_4$ is that it serves as its own indicator, the pink color being distinguishable even if the solution is very dilute. Therefore, no indicator is used in this reaction.

Procedure:

Preparation of 0.05M oxalate solution.

1. Pipette out 10 ml 0.05M standard oxalate solution in a conical flask.
2. Add about 50 ml 1M sulfuric acid solutions to it.
3. Heat the solution up to 60-70ºC and titrate against the Potassium Permanganate solution until the color changes to pink.
4. Calculate the strength of potassium permanganate from the data.

Data: TABLE1

Standardization of Potassium Permanganate solution with Standard Sodium Oxalate solution

No. of Obs.	**Volume Of $Na_2C_2O_4$ (ml)**	**Burette Reading (ml)**			**Average (ml)**	**Strength Of $KMnO_4$ (M)**
		Initial reading	**Final Reading**	**Difference**		
1	10	0.00	8.20	8.20	8.40	0.059
2	10	8.20	17.30	9.10		
3	10	20.20	28.10	7.90		

Calculation: Preparation of 0.05 M sodium oxalate solution.

In this case weight taken $Na_2C_2O_4$ g =?

Weight to be taken $Na_2C_2O_4$g =?

So the strength of $Na_2C_2O_4$ $= \frac{Weight\ to\ be\ taken}{Weight\ taken} \times 0.1\ M$

From $V_{Na2C2O4} \times S_{Na2C2O4} = V_{KMnO4} \times S_{KMnO4}$

$V_{Na2C2O4}$ = 10 ml

$S_{Na2C2O4}$ = 0.05 M

V_{KMnO4} = 8.40 ml

S_{KMnO4} = ?

0.059 M

Result: The Determined Strength of Potassium Permanganate Solution is: S_{KMnO4} = 0.059 M

Error: Percentage of error $= \frac{(Known\ Value - Observed\ Value)}{Known\ Value} \times 100\%$

Precautions: At first the burette was cleaned with distilled water carefully; then it was rinsed with rinse solution to avoid the density change of the given solution. In the same way the pipette was also washed with distilled water and rinsed with the rinse solution.

a. All the apparatus were handled carefully and according to the rules.

b. The pipette was kept on a clean sheet of paper.

c. The key of the burette was operated carefully to make sure that one-drop of Permanganate falls into the conical flask at a time.
e. The conical flask was kept on a white paper to trace the color change of the solution.
f. At first the key of the burette was fully opened to let the solution fall free to make the bubbles go out.
g. On the basis of the result it was found that the balance is very much sensitive. So for this reason the result can be varied. While measuring the lower meniscus of the burette an error may be happened for the parallax error. So concentration should be needed here.

Students should know: -

a. Why do you don‘t have to use any indicator in this experiment?
b. Why you have to heat the experiment solution?
c. Do you know about the effect produced by the change in temperature of a reaction mixture?
d. What happens if you do not shake solution properly?

Determination of Ferrous Ion in a Solution by Standard Potassium Permanganate ($KMnO_4$) Solution.

Objectives:

1. To study oxidation reduction titration.
2. To study the percentage of Iron present.

Theory:

In this experiment, the amount of iron in a supplied solution is determined. The reaction that occurs here is oxidation and reduction reaction.
An oxidation-reduction reaction (redox) is a reaction in which electrons are transferred between species or in which atoms change oxidation numbers.
Oxidation is the half reaction in which there is loss of electrons by a species.
And reduction is the half reaction in which there is a gain of electrons by a species.

Reaction involved in this reaction is:
$KMnO_4 + 5Fe^{2+} \equiv K^+ + Mn^{2+} + 4H_2O + 5Fe^{3+}$

In this reaction, Fe^{+2} is oxidized into Fe^{+3}. And
MnO_4^{-2} is reduced to Mn^{+2}.

From the above reaction we get:

1equivalent weight of $KMnO_4 \equiv$ 1 equivalent weight of Fe^{+2}

1000 ml (N) $KMnO_4$ solution ≡ 1000 ml (N) ferrous iron solution
≡ 1 atomic weight of iron

So, 1000 ml (N) $KMnO_4$ solution should be equivalent to one-gram atomic weight that is 55.855 gram of iron.

Therefore,
1 ml of (N) $KMnO_4 \equiv$ 0.5585 gram of iron, so, the weight of iron in the supplied solution may then be easily calculated using the above relation.

Procedure: N.B. standardizes $KMnO_4$ with sodium oxalate solution as Expt. No. 7.

1. Pipette out 10 ml of iron solution (Mohr‘s salt) in a conical flask.
2. Add about 6 ml of 1M sulfuric acid solutions. Dilute it to about 50 ml distill water.
3. Titrate the resulting solution with standard potassium permanganate solution.
4. The end point is indicated by the first appearance of permanent light pink color as in Expt. No.7.
5. Repeat the titration at least thrice. Calculate the amount of iron of the supplied solution.

DATA TABLE 1: Standardization of Potassium Permanganate solution with Standard Sodium Oxalate solution

No. of obs.	**Volume Of $Na_2C_2O_4$ (ml)**	**Burette Reading (ml)**			**Average (ml)**	**Strength Of $KMnO_4$ (M)**
		Initial	**Final**	**Difference**		
1						
2						
3						

Calculation:

TABLE2 : (Determination of Ferrous Iron by Standard Potassium Permanganate Solution)

No. of obs.	**Volume of Mohr’s salt (ml)**	**Burette Reading (ml)**			**Average (mL)**	**Amount of Fe^{+2}(g/L)**
		Initial	**Final**	**Difference**		
1	10	5.60	15.30	9.70	9.36	307.10
2	10	15.30	24.50	9.20		
3	10	29.30	38.50	9.20		

Calculation:—

From $V_{Fe2+} \times S_{Fe2+} = V_{KMnO4} \times S_{KMnO4}$

$V_{Fe2+} = 10$ ml
$S_{Fe2+} = ?$

V_{KMnO4} = 9.36 ml
S_{KMnO4} = 0.059 M

S_{Fe2+} = 0.055 M
0.055 × 55.85 g
= 3.071 g
10 ml Iron solution contain 3.071 g Fe^{2+}
1000 ml..307.10 g/L

Again, 1000 ml (1.0N) $KMnO_4$= 55.855 g. Fe^{+2}(10 ml)
=> ml N $KMnO_4$ = …. g. Fe^{+2} (10 ml)
Thus the amount of iron present per liter= (…×100) g.

Result: The amount of iron (in g.) present in the supplied Fe^{+2}solution (per liter) is: W_{Fe} = 307.10 g/L

Error:

Percentage of error $= \frac{(Known\ Value - Observed\ Value)}{Known\ Value} \times 100\%$

Discussion:

The following causes may be mentioned for the slight deviation of the result from the original one:

1) As the color change of the titration of $KMnO_4$ with $Na_2C_2O_4$ is very confusing, the end point of the titration may not have been properly determined.
2) As the color change is very confusing, the end point of the titration may not have been properly determined. During titration one or two extra drop of $KMnO_4$ might have been added to the $Na_2C_2O_4$ solution after the end point.

Student should know:

a. What would happen if you use HCl or HNO_3 acid instead of H_2SO_4?
b. What are the disadvantages of using $KMnO_4$?