Engineering Chemistry Laboratory Manual
(Common for All B. Tech Courses)

HEAS Department
University Department

Rajasthan Technical University, Kota

Vision of HEAS Department

To provide a sound foundation to the undergraduate students by giving quality education in basic sciences along with moral ethics and effective communication skills to become competent engineers of tomorrow's to serve the society.

Mission of HEAS Department

The Department is committed to train the young graduates in basic sciences and inculcate ethical values and social responsibilities through an innovative teaching and learning methodology.

CO's and CO's - PO's Mapping

Credit: 1   **Max. Marks: 75**
0L+0T+2P   **(IA: 50, ETE: 25)**

Course Outcome for Engineering Chemistry Laboratory Classes
The students should be able to:

CO1	Estimate the concentration of copper sulphate, ferrous ammonium sulphate and alkali mixture.
CO2	Analyse different properties of lubricating oils.
CO3	Estimate quality parameters of water like hardness, dissolved oxygen and residual chlorine in water.
CO4	Analyse quality of coal by proximate analysis.
CO5	Understand about Synthesis of drugs Aspirin and Paracetamol.

CO-PO Mapping for Engineering Chemistry Laboratory Classes
(3-Strong, 2- Moderate and 1- Weak)

CO	PO1	PO2	PO3	PO4	PO5	PO6	PO7	PO8	PO9	PO10	PO11	PO12
CO1	3	3	-	1	-	-	-	-	-	-	-	-
CO2	3	2	-	-	-	-	1	-	-	-	-	1
CO3	3	-	1	-	-	1	-	-	-	-	-	-
CO4	3	-	1	-	-	1	-	-	-	-	-	1
CO5	3	-	1	-	-	1	-	-	-	-	-	1

Program Outcomes (POs) (Common to all Branches):

PO1	**Engineering knowledge**: Apply the knowledge of mathematics, science, engineering fundamentals, and an engineering specialization to the solution of complex engineering problems.
PO2	**Problem analysis**: Identify, formulate, review research literature, and analyze complex engineering problems reaching substantiated conclusions using first principles of mathematics, natural sciences, and engineering sciences.
PO3	**Design/development of solutions**: Design solutions for complex engineering problems and design system components or processes that meet the specified needs with appropriate consideration for the public health and safety, and the cultural, societal, and environmental considerations.
PO4	**Conduct investigations of complex problems**: Use research-based knowledge and research methods including design of experiments, analysis and interpretation of data, and synthesis of the information to provide valid conclusions.
PO5	**Modern tool usage**: Create, select, and apply appropriate techniques, resources, and modern engineering and IT tools including prediction and modelling to complex engineering activities with an understanding of the limitations.
PO6	**The engineer and society**: Apply reasoning informed by the contextual knowledge to assess societal, health, safety, legal and cultural issues and the consequent responsibilities relevant to the professional engineering practice.
PO7	**Environment and sustainability**: Understand the impact of the professional engineering solutions in societal and environmental contexts, and demonstrate the knowledge of, and need for sustainable development.
PO8	**Ethics**: Apply ethical principles and commit to professional ethics and responsibilities and norms of the engineering practice.
PO9	**Individual and team work**: Function effectively as an individual, and as a member or leader in diverse teams, and in multidisciplinary settings.
PO10	**Communication**: Communicate effectively on complex engineering activities with the engineering community and with society at large, such as, being able to comprehend and write effective reports and design documentation, make effective presentations, and give and receive clear instructions.
PO11	**Project management and finance**: Demonstrate knowledge and Understanding of the engineering and management principles and apply these to one's own work, as a member and leader in a team, to manage projects and in multidisciplinary environments.
PO12	**Life-long learning**: Recognize the need for, and have the preparation and ability to engage in independent and life-long learning in the broadest context of technological change.

Laboratory Accident and First Aid

EYE ACCIDENTS

Flood your eyes immediately with water. For on acid, use dilute sodium bicarbonate solution, for an alkali, use dilutes boric acid solution. If glass has entered the remove the loose glass pieces by washing the eye gently, without rubbing, with lot of water. If glass has penetrated the eye, do not rub the eye, keep the eye open as far as possible and consult a doctor immediately.

BURNS

If burn is caused by flame or touching of some hot object pour plenty of cold water on the burnt area and apply burnol or furacin but if the burn is of serious type involving broken skin, apply 1% solution of sodium bicarbonate. If the burn is caused by some chemicals then proceed as follows.

ACID BURN

WASH immediately with large quantities of water, then with dilute 8% sodium bicarbonate solution. If burn is severe wash again with and apply the acriflavine or picric acid solution.

CUTS

For minor cuts allow bleeding to occur for few seconds, remove any glass piece, wash with water and apply dettol or savlon and bandage. In case, the cut is serious, try to check the bleeding by pressing above the cut till a doctor arrives, also wash with dettol or savlon and do not apply pressure continuously for more than five minutes. In case of minor cut, first wash it thoroughly with Iodine solution (2%). Remove glass pieces etc., if any with sterile forceps and apply sterile dressing (cotton wool soaked in rectified spirit or tincture benzoin may be used) and apply a bandage for serious cuts, try to stop bleeding and call a doctor immediately.

POISONS

Reagents in Mouth: If the reagent is in the mouth and not swallowed then spit out at once and wash the mouth thoroughly with water.

If swallowed: If the substance is acid or alkali, dilute by drinking much water. In case of acid, drink sufficient amount of lime water also after drinking water. Do not drink any emetic; however milk may be taken. For arsenic or mercury compounds take an emetic, e.g. table salt solution without delay. For salts of heavy metals, take an emetic or a tea spoon full of mustard oil or zinc sulphate with warm water and vomit.

Fire

In case the fire is small and contained to liquids in a beaker or flask then cover the opening of the vessel with a damp cloth. Fire is extinguished due to lack of air. Bench fires may be extinguished either by fire extinguishers or by sand. In every case, if possible remove any container of inflammable solvents and turn off burners in the affected area.

In case a person's clothes catch fire, do not allow to run, use force to make him lie down and smother the flame either with laboratory coat or fire blanket. If the fire is too extensive, ring up the nearest fire station immediately.

LIST OF EXPERIMENTS

1. Determination of the hardness of water by EDTA method.**[K3 Level; CO3]**

2. Determination of residual chlorine in water. **[K3 Level; CO3]**

3. Determination of dissolved oxygen in water. **[K3 Level; CO3]**

4. Determination of the strength of ferrous ammonium sulphate solution with the help of $K_2Cr_2O_7$ solution by using diphenyl amine indicator. **[K3 Level; CO1]**

5. Determination of the strength of $CuSO_4$ solution idometrically by using hypo solution. **[K3 Level; CO1]**

6. Determination of the strength of NaOH and Na_2CO_3 in a given alkali mixture. **[K3 Level; CO1]**

7. Proximate analysis of Coal. **[K4 Level; CO4]**

8. Determination of the flash & fire point and cloud & pour point of lubricating oil. **[K3 Level; CO2]**

9. Determination of the kinematic viscosity of lubricating oil by Redwood viscometer no.1 at different temperature**[K3 Level; CO2]**
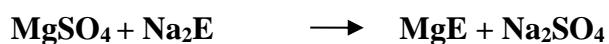
10. Synthesis of Aspirin/ Paracetamol. **[K5 Level; CO5]**
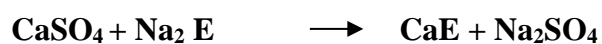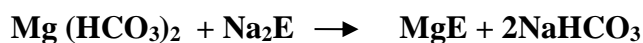
EXPERIMENT-1

1. **Object:** Determination of the hardness of water by EDTA method.

2. Materials required:

1) Bottle A_1 = Tap water
2) Bottle A_2 = Boiled water
3) Bottle B = Standard EDTA solution
4) Eriochrome Black-T (EBT) Indicator
5) Ammonia buffer solution

3. Chemical reactions:

$$CaCl_2 + Na_2E \longrightarrow CaE + 2NaCl$$

$$Mg(HCO_3)_2 + Na_2E \longrightarrow MgE + 2NaHCO_3$$

$$CaSO_4 + Na_2E \longrightarrow CaE + Na_2SO_4$$
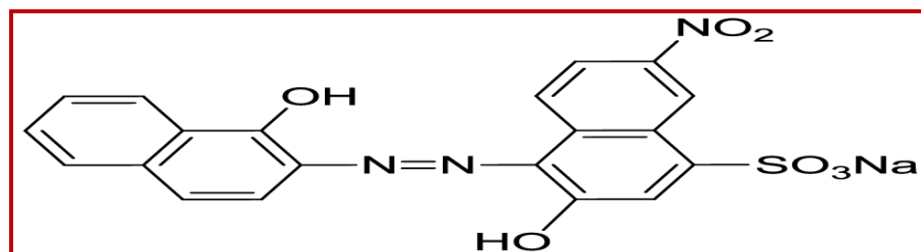
$$MgSO_4 + Na_2E \longrightarrow MgE + Na_2SO_4$$

(Na_2E = disodium salt of EDTA)

4. **Theory:** Ethylene Diamine Tetra Acetic Acid (**EDTA**) is a well-known powerful complexing agent.
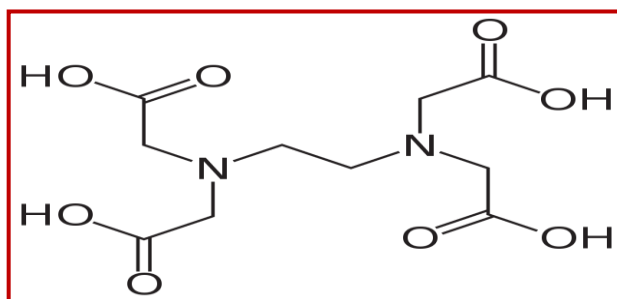
Structure of EDTA
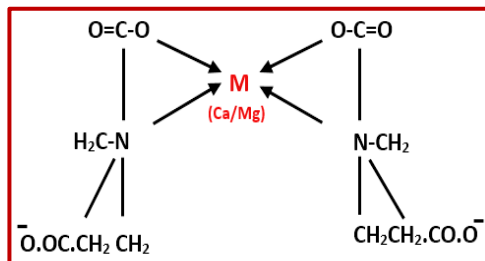

Structure of EBT Indicator

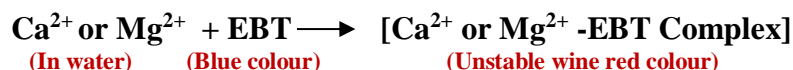EDTA form complexes with Ca^{2+} and Mg^{2+} ions as well as with many other metal cations. These complexes have the general formula as (Ca-EDTA) or (Mg-EDTA).

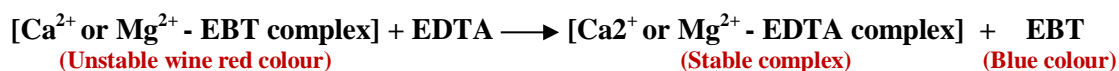**EDTA-Metal (Ca/Mg) Complex**

When indicator is added to hard water which is buffered at pH 9.7, it combines with Ca^{2+}, Mg^{2+} ions to form weak complex of wine red colour, when an excess of EDTA is added, colour change to blue due to free EBT. Thus change of wine red colour to distinct blue marks the end point of titration. Which is describes as blue.

$$Ca^{2+} \text{ or } Mg^{2+} + EBT \longrightarrow [Ca^{2+} \text{ or } Mg^{2+} \text{-EBT Complex}]$$
(In water)   **(Blue colour)**   **(Unstable wine red colour)**

When hard water is titrated against EDTA solution, EDTA or its sodium salt forms stable complex with Ca^{2+} or Mg^{2+} ions in water.

$$[Ca^{2+} \text{ or } Mg^{2+} \text{ - EBT complex}] + EDTA \longrightarrow [Ca2^+ \text{ or } Mg^{2+} \text{ - EDTA complex}] + EBT$$
(Unstable wine red colour)   **(Stable complex)**   **(Blue colour)**

5. Procedure:

1) Fill the burette with standard EDTA solution.
2) Take 25 or 50ml of tap water in a conical flask and add half a test tube of buffer solution and 1-2 drops of Eriochrome black T indicator, solution turns wine red.
3) Titrate this with EDTA solution until the wine red colour change to sky blue. Note the reading as X_1.
4) Repeat the above procedure with boiled water and note the burette reading as X_2.

6. Observations:

Table A (for Tap Water)

Sr.No.	Volume of Tap water (ml)	Burette reading (ml)		Volume of EDTA consumed (ml)	
		Initial (I)	Final (II)	(II-I)	Concordant Reading X_1

Table B (for Boiled Water)

Sr.No.	Volume of Boiled water (ml)	Burette reading (ml)		Volume of EDTA consumed (ml)	
		Initial (I)	Final (II)	(II-I)	Concordant Reading X_2

7. Calculations:

(A) For Total Hardness

$N_1V_1 = N_2V_2$

Where N_1= Normality of given **EDTA**

N_2= Normality of tap water as hardness

V_1= Volume of **EDTA** consumed (X_1)

V_2= Volume of tap water (25 or 50 ml)

Normality of tap water as hardness $N_2= (N_1 \times V_1)/V_2$

Total hardness of given water sample, Equivalent as $CaCO_3$

= **Normality of Tap water as hardness x Eq. Wt. of CaCO3**

= **N_2 x 50 gm /litre**

= **$(N_1 \times V_1)/V_2$ x 50 x 1000 mg/litre (ppm)**

(B) For Permanent Hardness

$N_3V_3 = N_4 V_4$

Where N_3 = Normality of given **EDTA**

N_4 = Normality of Boiled water as hardness

V_3 = Volume of **EDTA** consumed (X_2)

V_4= Volume of Boiled water (25 or 50 ml)

Normality of boiled water as hardness $N_4= (N_3 \times V_3)/V_4$

Permanent hardness of given water sample, Equivalent as $CaCO_3$

= **Normality of Boiled water as hardness x Eq. Wt. of CaCO$_3$**

= **N_2 x 50 gm /litre**

= **$(N_3 \times V_3)/V_4$ x 50 x 1000 mg/litre (ppm)**

(C) For Temporary hardness

Temporary hardness = Total hardness - Permanent hardness

8. Result:

1) Total hardness of given water sample, equivalent as $CaCO_3$ = ---------- ppm.
2) Permanent hardness of given water sample, equivalent as $CaCO_3$ = ---------- ppm.
3) Temporary hardness of given water sample, equivalent as $CaCO_3$ = ---------- ppm.

9. Precautions:

1) All the glass wares should be properly cleaned and dried.
2) Addition of reagents from the burette should be drop wise.
3) Be careful about colour change at the time of end point.
4) There should be no air bubbles inside the burette.
5) Note the reading of lower meniscus inside the burette in case of colourless solution and upper meniscus in case of coloured solution.
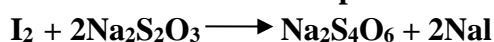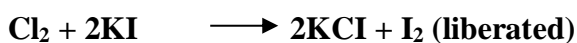
EXPERIMENT-2

1. **Object:** Determination of residual chorine in water.

2. **Materials required:**

   1) Bottle A_1 = water sample
   2) Bottle **B** = Standard sodium thiosulphate (Hypo) solution
   3) Starch indicator
   4) Potassium Iodide, Acetic Acid (glacial)

3. **Chemical reactions :**

   $$Cl_2 + 2KI \longrightarrow 2KCI + I_2 \text{ (liberated)}$$
   $$I_2 + Starch \longrightarrow Deep\ Blue\ colour$$
 $$I_2 + 2Na_2S_2O_3 \longrightarrow Na_2S_4O_6 + 2NaI$$
   Colourless solution

4. **Theory:**

   The principle of the residual chlorination is to add sufficient chlorine to oxidise all organic matter, iron, manganese, kill all the pathogens and other reducing substances present in the water being treated. It is also used to oxidise free ammonia in raw water. So that the remaining residual chlorine be presents free residual chlorine rather than the less active combined residual chlorine or chlorine or chloramines.

   Chlorine is strong oxidizing agent and liberates free iodine KI solutions at pH 3-4. The liberated iodine is titrated against standard sodium thiosulphate solution using starch as an indicator. The blue colour disappears, which shows the end point of titration.
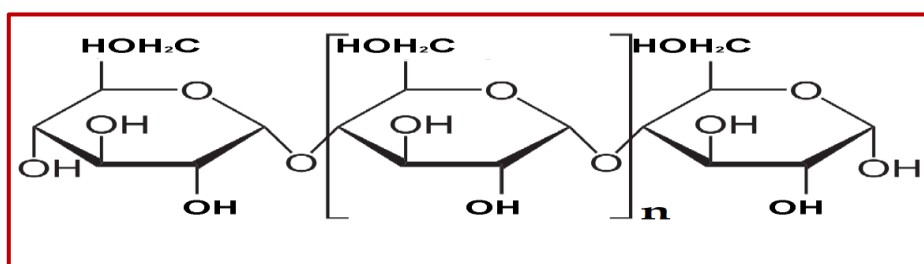
Structure of Starch Indicator

5. **Procedure:**

   1) Fill the burette with the sodium thiosulphate (Hypo) solution.
   2) Take 50 ml of water sample in conical flask and add 4-5 ml of acetic acid to maintain pH up to 3-4 and then add 10 ml of KI solution.
   3) To mix the solution, shake properly and gently to complete the reaction.
   4) Titrate it with hypo solution from burette till the solution becomes pale yellow.

Page No.: 12

5) Add 3-4 drops of freshly prepared starch indicator. The colour of solution turns to blue.

6) Continue titration with hypo till the blue colour disappears and note the volume of hypo consumed as X_1 ml.

6. Observations:

Titration of water sample with Hypo solution:

Sr. No.	Volume of water sample (ml)	Burette reading (ml)		Volume of Hypo consumed (ml)	
		Initial (I)	Final (II)	(II - I)	Concordant Reading X_1

7. Calculations:

$$N_1V_1 = N_2V_2$$
$$N_2 = N_1V_1/V_2$$

Where N_1= Normality of given **Hypo**
V_1= Volume of **Hypo consumed(X_1)**
N_2 = Normality of water sample
V_2= Volume of water Sample (50 ml)

Normality of water sample as residual chlorine; $N_2 = (N_1 \times V_1)/V_2$

Strength of Residual Chlorine = **Normality water sample as residual Chlorine x Eq. wt. of Chlorine**
= **N_2 x 35.5 gm/litre**
= **$(N_1 \times V_1)/V_2$ x 35.5 x 1000 mg/litre (ppm)**

8. Result:

The amount of residual chlorine in given water sample = ... mg/litre (ppm).

9. Precautions:

1) Chlorine vapours are harmful so the solution should not be sucked by pipette.
2) All the glass wares should be rinsed with distilled water.
3) Starch indicator should be freshly prepared.
4) The titration should be completed rapidly, to avoid atmospheric oxidation of iodine.
5) There should be no air bubbles inside the burette.
6) Note the reading of lower meniscus inside the burette in case of colourless solution and upper meniscus in case of coloured solution.
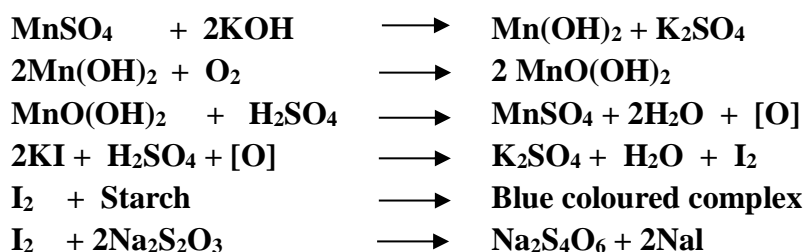
EXPERIMENT-3

1. **Object:** Determination of dissolved oxygen (DO) in water.

2. **Materials required:**

   1) Bottle A_1 = water sample
   2) Bottle B = Standard sodium thiosulphate (Hypo) solution
   3) Manganese sulphate solution
   4) Concentrated Sulphuric acid
   5) Alkali iodide azide (KOH/KI/NaN$_3$) solution
   6) Starch indicator

3. **Chemical reactions:**

$$MnSO_4 + 2KOH \longrightarrow Mn(OH)_2 + K_2SO_4$$
$$2Mn(OH)_2 + O_2 \longrightarrow 2\ MnO(OH)_2$$
$$MnO(OH)_2 + H_2SO_4 \longrightarrow MnSO_4 + 2H_2O + [O]$$
$$2KI + H_2SO_4 + [O] \longrightarrow K_2SO_4 + H_2O + I_2$$
$$I_2 + Starch \longrightarrow Blue\ coloured\ complex$$
$$I_2 + 2Na_2S_2O_3 \longrightarrow Na_2S_4O_6 + 2Nal$$

Colourless solution

4. **Theory:**

   Dissolved oxygen (DO) is the amount of oxygen present in water and is essential for maintaining its natural ecology. The DO of unpolluted natural waters is usually in the range of 4 to 8 mg/lit.

   DO is estimated using the Winkler or iodometric method. Manganese reacts with alkali iodide azide (during fixation) to form a white precipitate of $Mn(OH)_2$, which gets oxidized to brown precipitate of $MnO(OH)_2$. In acidic medium, Mn ions are reduced by I$^-$ (iodide) ions to form I_2 (iodine). The amount of I_2 liberated is equivalent to the amount of dissolved oxygen in the sample. This I_2 is titrated with standard $Na_2S_2O_3$ solution using starch indicator. At the end point of titration formation of blue colour by the addition of starch disappears. Fast quantification is ensured by addition of Mn (II) salts in strongly alkaline medium.
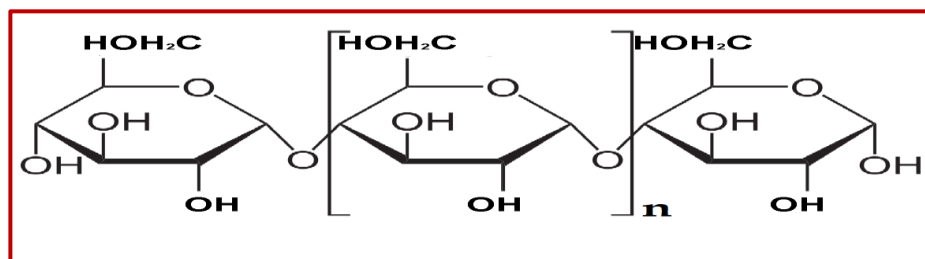
Structure of Starch Indicator

5. Procedure:

DO is measured by Winkler test. Before proceeding with the experiment, the water sample has to be fixed for DO immediately at the site itself.

A. DO Fixing:

1. The 250 ml DO bottle is filled completely with water sample.
2. The bottle is immediately corked up to ensure that no air bubble is trapped. To the sample water, add 2ml of $MnSO_4$ solution with a pipette.
3. The solution should be added well below the surface of the water. Mix the contents by inverting the bottle 3-4 times.
4. Next, add 2 ml of alkali iodide azide ($KOH/KI/NaN_3$) solution to the sample water. Mix well until a brown precipitate forms.
5. A brown precipitate is formed and will soon settle down. Place the stopper tightly and shake the bottle vigorously.
6. Allow the sample to stand for at least 20 minutes.

B. Measurement of DO:

1. To the above fixed water sample, add 2 ml of concentrated sulphuric acid.
2. Replace the stopper and mix the solution to dissolve the precipitate.
3. The colour of the solution becomes yellow due to formation of free iodine I_2.
4. Pour out 20/25/50 ml of this sample, with pipette, into the conical flask.
5. Add dropwise starch indicator (1 % starch solution, about 15-20 drops) with a dropper to the conical flask.
6. Shake the flask and observe the colour change to blue.
7. Now add the standard hypo solution drop wise until the blue colour disappears. This is the endpoint of the titration. Note the volume of hypo, sodium thiosulphate added.
8. Note the volume of standard hypo solution added.
9. Take three readings and finally volume (X_1) ml of hypo is considered as concordant volume for the calculation purpose.

7. Observation Table:

Sr. No.	Volume of water sample (ml)	Burette Reading (ml)		Volume of Hypo solution consumed (ml)	
		Initial (I)	Final (II)	(II)-(I)	Concordant Reading (X_1)
1.					
2.					
3.					

8. Calculation:

For dissolved oxygen (DO) in water:

$$N_1V_1 = N_2V_2$$

Where N_1 = Normality of water sample as dissolved oxygen
V_1 = Volume of water sample (50 ml)
N_2 = Normality of standard Hypo solution
V_2 = Volume of Hypo solution (X_1)

Normality of water sample as dissolved oxygen (DO); $N_1 = (N_2 \times V_2)/V_1$
Dissolved oxygen in a water sample = **Normality of water sample as DO x Eq. Wt. of Oxygen**
= **N_1 x 8 gm/litre**
= **$(N_2 \times V_2)/V_1$ x 8 x 1000 mg/litre (ppm)**

9. Result:

Dissolved oxygen (DO) in given water sample = ……... ppm.

10. Precautions:

1. The glass wares used should be thoroughly washed with distilled water and dried.
2. All the solutions should be freshly prepared.
3. Special care should be taken in sampling.
4. The reagents should be added inside the bottle with the help of pipette to avoid air contact with the sample.
5. There should not be any bubble after the addition of reagents in the water sample inside the bottle.
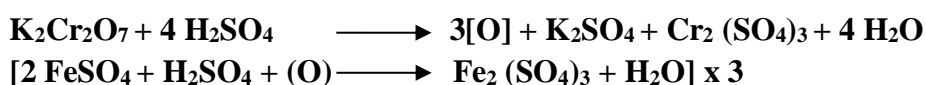6. Same volume of indicator should be used every time.

EXPERIMENT-4

1. Object: Determination of the strength of ferrous ammonium sulphate solution with the help of $K_2Cr_2O_7$ solution by using N-Phenyl anthranilic acid indicator.

2. Requirement:

1) Bottle A_1 = Known FAS
2) Bottle A_2 = Unknown FAS
3) Bottle B = $K_2Cr_2O_7$ solution
4) N-Phenyl anthranilic acid indicator
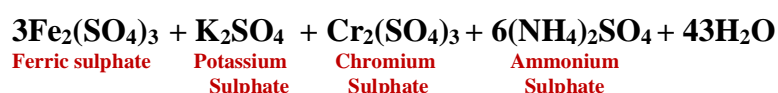5) Dilute H_2SO_4 acid

3. Chemical Reaction:

$$K_2Cr_2O_7 + 4\ H_2SO_4 \longrightarrow 3[O] + K_2SO_4 + Cr_2\ (SO_4)_3 + 4\ H_2O$$
$$[2\ FeSO_4 + H_2SO_4 + (O) \longrightarrow Fe_2\ (SO_4)_3 + H_2O]\ x\ 3$$

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

4. Theory:

$K_2Cr_2O_7$ is a good oxidizing agent, and in the presence of dilute H_2SO_4 it liberates three atoms of nascent oxygen.

$$K_2Cr_2O_7 + H2SO4 \longrightarrow K_2SO_4 + Cr_2\ (SO_4)_3 + 4H_2O + 3[O]$$
Nascent oxygen
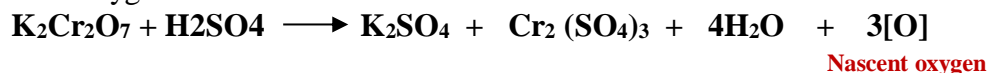
This liberated nascent oxygen oxidizes Fe^{+2} ferrous ions of FAS into Fe^{+3} ferric ions, per the following reaction:

$$6[FeSO_4 \cdot (NH_4)_2SO_4\ 6H_2O] + K_2Cr_2O_7 + 7H_2SO_4 \longrightarrow$$
Ferrous ammonium sulphate (FAS)   **Potassium dichromate**

$$3Fe_2(SO_4)_3 + K_2SO_4 + Cr_2(SO_4)_3 + 6(NH_4)_2SO_4 + 43H_2O$$
Ferric sulphate   **Potassium Sulphate**   **Chromium Sulphate**   **Ammonium Sulphate**

N-phenyl anthranilic acid is used as an indicator. Indicator is not oxidized as long as Fe^{+2} ions are there in the solution. The slight excess amount of dichromate will oxidize the indicator when all of the Fe^{+2} ferrous ions have been converted to Fe^{+3} ferric ions resulting in colour change of the solution from greenish (due to Cr^{+3}) to red violet.
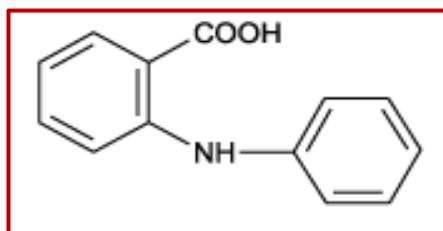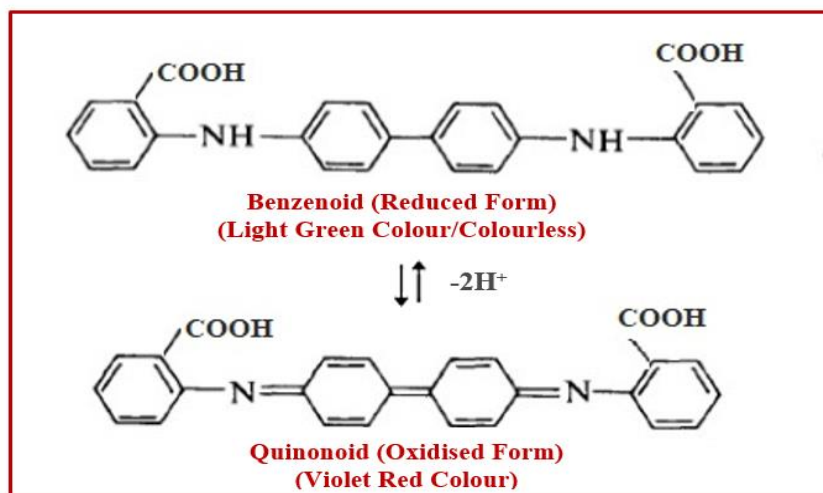

Structure of Indicator N-Phenyl anthranilic acid

Structure of N-Phenyl anthranilic acid in Benzenoid/Quinonoid Forms

5. Procedure:

1) Fill the burette with Potassium dichromate solution.
2) Take 20 or 25 ml of known FAS in conical flask and add half test tube of dilute H_2SO_4 and add 5-6 drops of indicator.
3) Colour of the solution become light green or colourless.
4) Run $K_2Cr_2O_7$ solution from burette in to the flask until the colour becomes violet and note the reading as X_1.
5) Follow the same procedure with unknown FAS solution and note the reading as X_2.

6. Observation table:

Table A for Known FAS

Sr.No.	Volume of Known FAS (ml)	Burette Reading (ml)		Volume of $K_2Cr_2O_7$ consumed (ml)	
		Initial (I)	Final (II)	(II-I)	Concordant Reading X_1
1					
2					
3					

Table B for Unknown FAS

Sr.No.	Volume of Unknown FAS (ml)	Burette Reading (ml)		Volume of $K_2Cr_2O_7$ consumed (ml)	
		Initial (I)	Final (II)	(II-I)	Concordant Reading X_2
1					
2					
3					

7. Calculation:

(A) For strength of $K_2Cr_2O_7$ with known FAS solution:

$$N_1V_1 = N_2V_2$$

Where N_1 = Normality of known FAS

V_1 = Volume of known FAS (20 or 25 ml)
N_2 = Normality of $K_2Cr_2O_7$
V_2 = Volume of $K_2Cr_2O_7$ **(X_1)**

Normality of $K_2Cr_2O_7$ solution $N_2 = (N_1 \times V_1)/V_2$

(B) For strength of unknown FAS solution:

$$N_3V_3 = N_4V_4$$

Where N_3 = Normality of unknown **FAS**

V_3 = Volume of unknown **FAS** (20 or 25 ml)
N_4 = Normality of $K_2Cr_2O_7$ **(N_2)**
V_4 = Volume of $K_2Cr_2O_7$ **(X_2)**

Normality of unknown FAS solution; $N_3 = (N_4 \times V_4)/V_3$

Strength of unknown FAS in gm/litre = **Normality of unknown FAS x Eq. Wt. of FAS**
= N_3 x 392 gm/litre
= $(N_4 \times V_4)/V_3$ x 392 gm /litre

8. Result:

Strength of FAS in given unknown solution is -------- gm/litre.

9. Precautions:

1) All the glass wares should be properly cleaned and dried.
2) Addition of reagent from the burette should be drop wise.
3) Be careful about change of colour at the time of end point.
4) There should be no air bubble inside the burette.
5) Note the reading of lower meniscus inside the burette in case of colourless solution and upper meniscus in case of coloured solution.
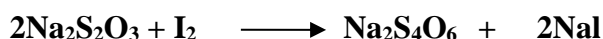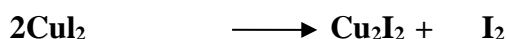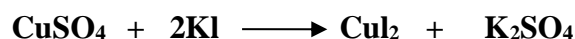
EXPERIMENT-5

1. Object: Determination of the strength of $CuSO_4$ solution idometrically by using hypo solution.

2. Material required:

1) Bottle A_1 = Known $CuSO_4$ `
2) Bottle A_2 = Unknown $CuSO_4$
3) Bottle B = Sodium thiosulphate (Hypo) solution)
4) Starch Indicator $\{(C_6H_{10}O_5)\ n\}$
5) 10% KSCN solution & 10% KI solution

3. Chemical reaction:

$$CuSO_4\ +\ 2Kl\ \longrightarrow\ Cul_2\ +\ K_2SO_4$$

$$2Cul_2\ \longrightarrow\ Cu_2I_2\ +\ I_2$$

$$2Na_2S_2O_3\ +\ I_2\ \longrightarrow\ Na_2S_4O_6\ +\ 2Nal$$

4. Theory:

For the determination of strength of copper sulphate ($CuSO_4$), 10 % solution of potassium iodide (KI) is added to given $CuSO_4$ solution. It gives white precipitate of cuprous iodide (Cu_2l_2) and equivalent amount of free iodine (I_2) is liberated.

$$2CuSO_4\ +\ 4KI\ \longrightarrow\ Cu_2I_2\ +\ 2K_2SO_4\ +\ I_2$$
Copper Sulphate   Potassium Iodide   Cuprous Iodide (White ppt.)   Potassium Sulphate   Free Iodine

This free iodine is titrated against standard solution of sodium thiosulphate ($Na_2S_2O_3$) hypo solution, using starch as indicator. This iodine is reduced to sodium iodide (NaI) and hypo converts into sodium tetrathionate.

$$2Na_2S_2O_3\ +\ I_2\ \longrightarrow\ Na_2S_4O_6\ +\ 2NaI$$
Sodium Thiosulphate   Iodine   Sodium Tetrathionate   Sodium Iodide
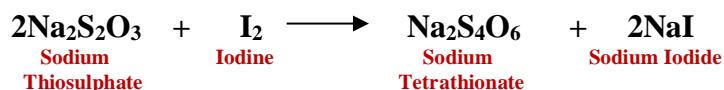
The Iodine also reacts with starch and form blue coloured iodo-starch complex.

$$Starch\ +\ I_2\ \longrightarrow\ Iodo–Starch\ Complex$$
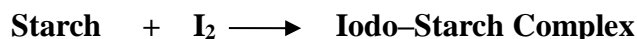
Due to this, starch is added to reaction mixture just before the completion of reaction between sodium thiosulphate and iodine, so that, the minimum amount of iodine is available for starch to indicate completion of reaction. At the end point and completion of reaction iodine is reduced to sodium iodide and the blue colour of iodo-starch complex gets disappeared due to formation of white colour of the cuprous iodide.
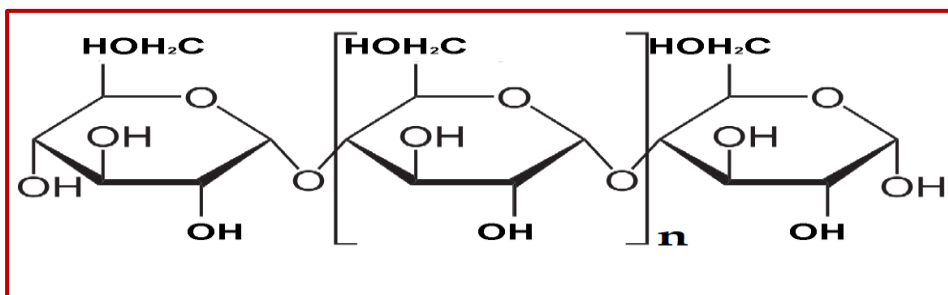
Structure of Starch Indicator

5. Procedure:

1) Take Hypo solution and fill it into the burette.
2) Take 20 or 25 ml of $CuSO_4$ of known strength (From bottle A_1), add 2 ml of 10% KI solution & shake it, the colour becomes dark yellow.
3) Start adding hypo solution from burette till the colour changes to light yellow.
4) Add 5 ml of 10% KSCN solution & add 3-4 drops of starch indicator in above solution yellow colour change to dark blue.
5) Again titrate it, with hypo solution till the blue colour becomes colourless. Note the reading (say X_1)
6) Repeat the above procedure with unknown solution $CuSO_4$ and note the reading (say X_2)

6. Observation:

(A) For known $CuSO_4$ solution

Sr.No.	Volume of known $CuSO_4$ (ml)	Burette Reading (ml)			Volume of $Na_2S_2O_3$ Hypo Solution (ml)
		Initial (I)	Final (II)	(II - I)	Concordant Reading X_1
1.					
2.					
3.					

(B) For unknown $CuSO_4$ solution

Sr.No.	Volume of unknown $CuSO_4$ (ml)	Burette Reading (ml)			Volume of $Na_2S_2O_3$ Hypo Solution (ml)
		Initial (I)	Final (II)	(II - I)	Concordant Reading X_2
1.					
2.					
3.					

7. Calculation:

(A) For strength of Hypo solution with known $CuSO_4$ Solution:

$$N_1V_1 = N_2V_2$$

Where N_1 = Normality of known $CuSO_4$ (Given)

V_1 = Volume of known $CuSO_4$ (20 or 25 ml)

N_2 = Normality of Hypo solution

V_2 = Volume of Hypo solution (X_1)

Normality of Hypo solution $N_2 = (N_1 x V_1)/V_2$

(B) For strength of unknown $CuSO_4$ Solution:

$$N_3V_3 = N_4V_4$$

Where N_3 = Normality of unknown $CuSO_4$

V_3 = Volume of unknown $CuSO_4$ (20 or 25ml)

N_4 = Normality of Hypo solution (N_2)

V_4 = Volume of Hypo solution (X_2)

Normality of unknown $CuSO_4$ solution $N_3 = (N_4 x V_4)/V_3$

Strength of unknown $CuSO_4$ in gm/litre = **Normality of unknown $CuSO_4$ x Eq. Wt. of $CuSO_4$**

= N_3 **x 249.69 gm/litre**

= $(N_4 x V_4)/V_3$ **x 249.69 gm/litre**

8. Result:

Strength of given unknown $CuSO_4$ solution is ------------------ gm/litre.

9. Precautions:
1) All the glass wares should be properly cleaned and dried.
2) Addition of reagent from the burette should be drop wise.
3) Starch indicator should be freshly prepared.
4) Be careful about the colour change at the time of end point.
5) There should be no air bubble inside the burette.
6) Note the reading of lower meniscus inside the burette in case of colourless solution and upper meniscus in case of coloured solution.
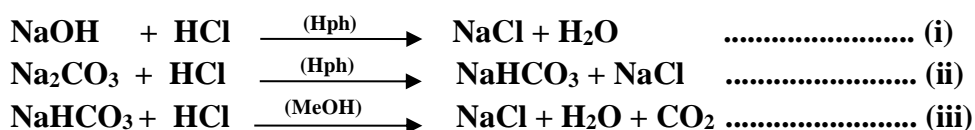
EXPERIMENT-6

1. Object: Determination of NaOH and Na_2CO_3 in a given alkali mixture.

2. Materials required:
1) Bottle A_1 = Alkali mixture
2) Bottle B = Standard HCl
3) Phenolphthalein (Hph) Indicator
4) Methyl Orange (MeOH) Indicator

3. Chemical reactions:

$$NaOH + HCl \xrightarrow{\text{(Hph)}} NaCl + H_2O \quad \text{......................... (i)}$$
$$Na_2CO_3 + HCl \xrightarrow{\text{(Hph)}} NaHCO_3 + NaCl \quad \text{....................... (ii)}$$
$$NaHCO_3 + HCl \xrightarrow{\text{(MeOH)}} NaCl + H_2O + CO_2 \text{....................... (iii)}$$

4. Theory:

The estimation of strength of NaOH and Na_2CO_3 in a given alkali mixture is an acid-base titration and these can be determined separately by titrating against standard acid using phenolphthalein and methyl orange indicators.

Phenolphthalein indicator is used for completing reactions (i) and (ii) whereas methyl orange indicator is used for reaction (iii). Hence, the volume of HCl acid used for phenolphthalein indicator at the end point, where the colour of the medium changes from pink to colourless, corresponds to the neutralization of NaOH to NaCl and half of the Na_2CO_3 to $NaHCO_3$. Similarly volume of HCl acid used for methyl orange indicator at the end point, where the colour of the medium changes from yellow to red, corresponds to the neutralization of remaining half of the Na_2CO_3 i.e. $NaHCO_3$ to NaCl.

Reactions (i) and (ii) completed in the pH range of 8-10 therefore phenolphthalein is used for this purpose. Reaction (iii) completed in the pH range of 3-4 which can be easily determined using methyl orange indicator. Phenolphthalein and methyl orange indicators are being used as acid-base internal indicators. The resonating structures of these indicators have varying colours in different medium as per the following:
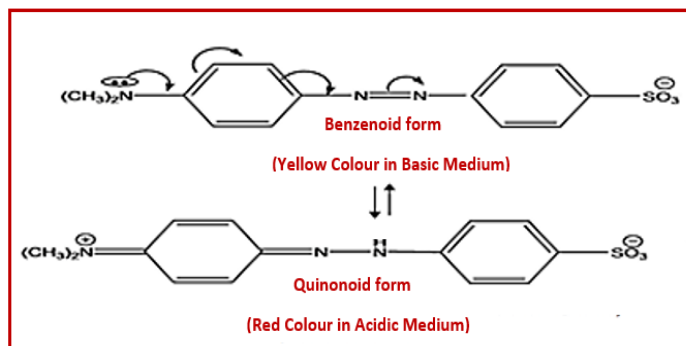

Benzenoid form
(Colourless in Acidic Medium)

Quinonoid form
(Pink Colour in Basic Medium)
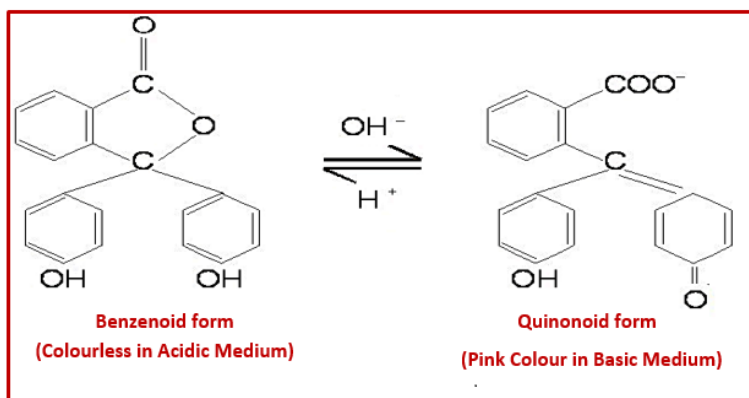
Structure of Phenolphthalein Indicator

Structure of Methyl Orange Indicator

In this titration at the end point colour changes from pink to colourless with phenolphthalein indicator and with methyl orange indicator at the end point the colour changes from yellow to red.

5. Procedure:

1) Take standard HCl from bottle B in the burette.
2) Take 25 or 20 ml alkali mixture in a conical flask from bottle A_1 and add 2-3 drops of Hph indicator. Pink colour is observed.
3) Titrate this solution with standard HCl till the pink colour disappears. (Note this reading of HCl consumed as X_1 ml.)
4) Now add 3-4 drops of methyl orange indicator in the same flask, the colour of the solution becomes yellow, continue titration with HCl until yellow colour changes to red. (Note this reading of HCl consumed as X_2 ml.)

6. Observations:

Sr. No.	Volume of alkali mixture(ml)	Volume of HCl with Hph (ml)				Volume of HCl with MeOH (ml)			
		Initial (I)	Final (II)	(II-I)	Concordant Reading X_1	Initial (II)	Final (III)	(III-II)	Concordant Reading X_2

7. Calculations:

A. For the strength of Sodium carbonate

$$N_1 V_1 = N_2 V_2$$

Where N_1 = Normality of sodium carbonate

V_1 = Volume of Alkali mixture (25 or 20 ml)

N_2 = Normality of standard **HCl** (given)

V_2 = Volume of **HCl** consumed for Na_2CO_3 $[2(X_2)]$

Normality of Na_2CO_3; $N_1 = (N_2 \times V_2)/V_1$

Strength of Na_2CO_3 in gm/litre = **Normality of Na_2CO_3 x Eq. Wt. of Na_2CO_3**

= N_1 x 53 gm/litre

= $(N_2 \times V_2)/V_1$ x 53 gm/litre

B. For the strength of Sodium hydroxide

$$N_3V_3 = N_4V_4$$

Where, N_3 = Normality of sodium hydroxide

V_3 = Volume of Alkali mixture (25or 20 ml)

N_4 = Normality of standard **HCl**

V_4 = Volume of **HCl** consumed for **NaOH [X_1- X_2]**

Normality of NaOH; $N_3 = (N_4 \times V_4)/V_3$

Strength of NaOH in gm/litre = **Normality of NaOH x Eq. wt. of NaOH**

= **N_3 x 40 gm /litre**

= **$(N_4 \times V_4)/V_3$ x 40 gm/litre**

8. Result:
1) Strength of Na_2CO_3 = --------- gm/litre.
2) Strength of NaOH   = --------- gm/litre.

9. Precautions:
1) All the glass wares should be properly cleaned and fried.
2) Addition of reagents from the burette should be drop wise
3) Be careful about the colour change at the time of end point
4) There should be no air bubble inside the burette.
5) Note the reading of lower meniscus inside the burette in case of colourless solution and upper meniscus in case of coloured solution.

EXPERIMENT-7

1. **Object:** Proximate analysis of coal.

2. **Materials required:**

   1) Coal sample
   2) Muffle furnace
   3) Electric hot oven
   4) Weighing machine
   5) Silica crucible

3. **Theory:**

   Analysis of coal is significant since the available coal may be having varying compositions of carbon, moisture and volatile matter which decide its calorific value. Proximate and ultimate are two important analyses are carried out to assess the quality of coal.

   Proximate analysis of coal includes the determination of moisture content, volatile matter, ash and fixed carbon. In this analysis, the data varies with the procedure adopted for study. The content of moisture, volatile matter and ash are experimentally determined, while that of fixed carbon is calculated.

4. **Procedure:**

   Procedures for the determination of content of moisture, volatile matter, ash and fixed carbon are as per the following:

   1. **Moisture content:**

      About 1 g of finely powdered, air-dried coal sample is weighed in a crucible, which is placed inside an electric hot oven, maintained at 105°C–110°C. The crucible is allowed to remain in oven for 1 hour and then taken out, cooled in desiccators and weighed. The loss in weight is reported as moisture.

      $$\text{Moisture Content (\%)} = \frac{\text{Loss in weight}}{\text{Weight of coal sample taken}} \times 100$$

      Moisture in coal evaporates during the burning of coal. Therefore, moisture lowers the effective calorific value of coal. Hence, lesser the moisture content, better the quality of coal as a fuel. However, presence of moisture, up to 10%, produces a more uniform fuel bed and less amount of fly-ash.

   2. **Volatile matter:**

      The dried sample of coal left in the crucible in analysis of moisture content is then covered with a lid and placed in an electric furnace (muffle furnace) maintained at 925°C. The crucible is taken out of the oven after 7 minutes of heating and cooled, first in air

and then inside a desiccators; it is again weighed. The loss in weight is reported as volatile matter in percentage terms.

$$\text{Volatile matter (\%)} = \frac{\text{Loss in weight due to removal of volatile matter}}{\text{Weight of coal sample taken}} \times 100$$

The volatile matter present in the coal may be combustible gases or non-combustible matter. The presence of non- combustible matter is always undesirable as they do not add to heat value. Therefore, higher volatile content in coal is undesirable. A high volatile matter containing coal burns with a long flame, large amount of smoke and has low calorific value. Hence, lesser the volatile matter, better is the rank of the coal.

3. Ash content:

The residual coal in the crucible in analysis of volatile matter is then heated without lid in a muffle furnace at around 750°C for 30 minutes. The crucible is then taken out, cooled first in air, then in a desiccator and weighed. Heating, cooling and weighing is repeated, till a constant weight is obtained. The residue is reported as ash in percentage terms.

$$\text{Ash Content (\%)} = \frac{\text{Weight of ash left}}{\text{Weight of coal sample taken}} \times 100$$

Ash is a useless non-combustible matter which reduces the calorific value of coal. Moreover, ash causes the hindrance to the flow of air and heat, thereby lowering the temperature and the combustion of coal becomes incomplete. Hence, lower the ash content, better is the quality of coal. The presence of ash also increases the transport, handling, storage and disposal costs.

4. Fixed carbon:

This refers to the amount of carbon left behind that can be burnt by a primary current of air from the hot bed of the fuel. It is calculated by deducting the percentage amount of ash, moisture and volatile matter from 100.

Fixed Carbon (%) = 100 – (Moisture + Volatile matter + Ash)

Higher the percentage of fixed carbon content, better is the quality of coal as higher is the calorific value of coal. Hence, high percentage of fixed carbon is desirable. It is useful for designing the furnace and firebox.

5. Result:

1) Moisture content of given coal sample = ……... %.
2) Volatile matters of given coal sample = ……... %.
3) Ash content of given coal sample = ……... %.
4) Fixed carbon content of given coal sample = ……... %.

6. Precautions:

1) Special care should be taken in sampling.
2) The crucible must be properly cleaned and dried before it is being used.
3) Crucible after ignition in the furnace must be cooled in desiccator before its weighing.
4) Handle the furnace very carefully.
5) Wear safety goggles at all times.

EXPERIMENT-8

1. Object: To determine the flash & fire point and cloud & pour point of a given lubricating oil.

2. Material Required:

1) Pensky Marten's apparatus
2) Cloud-Pour Point apparatus
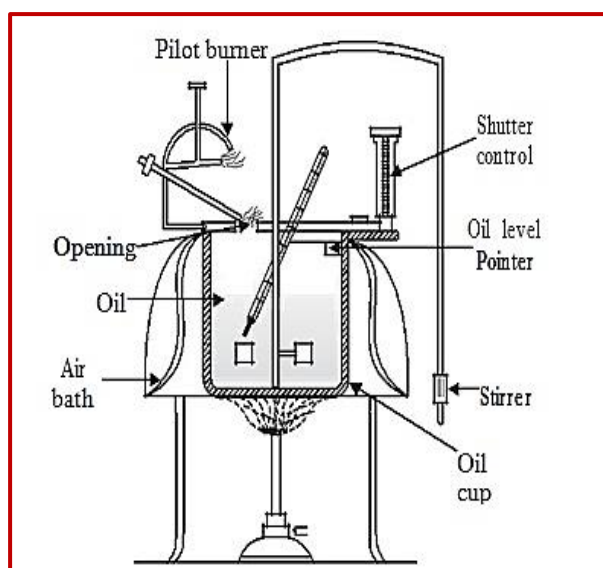3) Lubricating oil
4) Thermometer
5) Heater
6) Sprit lamp
7) Match sticks

3. Theory:

1) Flash & Fire Point

Good lubricating oil should not volatize under the working temperature and even if some volatization takes place, the vapours should not form inflammable mixture with air under the condition of lubrication. The flash and fire point is helpful in providing safeguard against fire hazard during their storage and their industrial use. Flash point of the oil should be above than the operating temperature of the machine.

Flash Point: Flash point is the lowest temperature at which the lubricating oil gives off enough vapours to ignite for a moment when a test flame is brought near to it.

Fire Point: Fire point is the lowest temperature at which the vapours of the lubricating oil burns continuously for at least five second when a test flame is brought near to it.

Pensky Marten's Flash & Fire Point Apparatus

The flash & fire point of a lubricating oil is determined by Pensky Marten's Apparatus. The oil cup is made of brass, fitted with a thermally insulated lifting handle. The cup lid, with insulated handle, is fitted with two opposite wedge cams, which engage under pillars secured to the cup flange. This feature facilitates easy removal of the lid by rotating the handle a few degrees to disengage the cams. The shutter opening mechanism is designed for smooth operation and is operated by a spring handle with a knurled knob. An exposure device is mounted over the lid. The manually operated stirrer has a flexible shaft. The cup assembly is positioned in a cast iron air bath, fitted with a chrome plated brass top. The air bath is mounted on an upright.
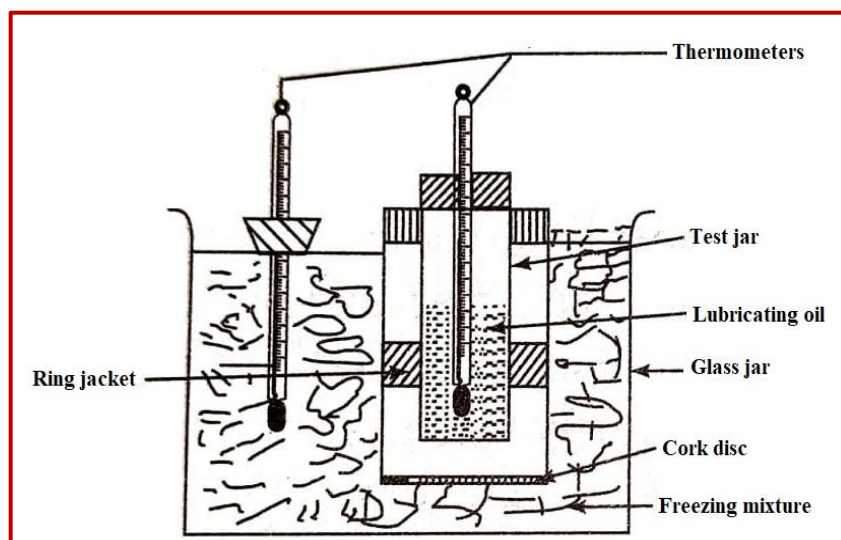
2) Cloud & Pour Point

Cloud and pour point indicates the suitability of lubricants in cold conditions. Lubricant used in a machine working at low temperatures should possesses low-pour point, otherwise solidification of lubricant will jam the machine.

The cloud and pour points are related to low temperature characteristics of lubricating oil and tells the behaviour of oil at low temperature. Cloudiness or haziness of lubricating oil is due to separation of waxes from the oil. Due to the separation of waxes viscosity of fuel oil increases and its fluidity decreases. The presence of solidified waxes increases the density or thickness of oil which may clogs oil filter and injector in engine and thereby affects the performance of engine. The pour point is the temperature below which oil cannot be used as lubricant. The cloud point indicates the tendency of oil to the filters and small orifices of machines at cold operating temperatures.

Cloud Point: Cloud point is the lowest temperature at which the lubricating oil gives cloudy appearance.

Pour Point: Pour point is the lowest temperature at which the lubricating oil tends to freeze or ceases to flow.

Cloud & Pour Point Apparatus

4. Procedure:

1) Flash & Fire Point

1) The oil sample under test is poured into the oil cup up to the mark. The cover incorporating the stirring device, thermometer and the flame exposure device is fixed at the top of the apparatus.
2) The Pensky Marten's apparatus is heated to increase the oil temperature. When temperature reaches about 100^0C the heating is slowed down and the vapours are tested with the help of an igniting material for the flash point.
3) After the determination of the flash point heating is continued rather slowly and the temperature is noted as the fire point when flame burns at least for five second.
4) Above procedure is repeated for different readings.

2) Cloud & Pour Point

1) Cloud and pour points of a lubricating oil is determined with the help of a cloud and pour point apparatus.
2) The apparatus is consist essentially of a flat bottom glass tube of about 3 cm in diameter and 12 cm in height and closed in air jacket. The glass tube is placed in a glass jar having suitable freezing mixture fitted with a thermometer.
3) The filtered lubricating oil is filled up to the mark inside the flat bottom glass tube and the cork is then fitted along with the thermometer. The thermometer should be dipped inside the oil.
4) The glass tube is then kept in the freezing mixture of the glass jar. The temperature of the oil will falls on cooling.
5) Note the temperature as cloud point of the lubricating oil when haziness or cloudiness appears in the glass tube. On further cooling of the oil, the temperature is noted as pour point at which flow of the lubricating oil seized due to its solidification.
6) The glass tube containing the lubricating oil is taken out of the freezing mixture after every one degree centigrade fall in temperature and then inspected for a moment to observe the formation of cloud and solidification of oil.
7) Above procedure is repeated for different readings.

5. Observation Table:

Sr. No.	Flash Point (ºC)	Fire Point (ºC)	Cloud Point (ºC)	Pour Point (ºC)
1.				
2.				
3.				

6. Result:

1. Flash point of the given lubricating oil   = ... ^{0}C.
2. Fire point of the given lubricating oil   = ... ^{0}C.
3. Cloud point of the given lubricating oil   = ... ^{0}C.
4. Pour point of the given lubricating oil   = ... ^{0}C.

7. Precautions:

1) The apparatus should be thoroughly cleaned and dried.
2) The bulb of the thermometer should be dipped inside the oil surface.
3) Rate of heating/rate of cooling should be slow.
4) Note the temperature very carefully.
5) Oil should be pure.

EXPERIMENT-9

1. Object: Determination of kinematic viscosity of lubricating oil by Redwood viscometer No.1 at different temperature.

**2. Requirements:**
    1) Redwood viscometer No.1
    2) Lubricating oil
    3) Stop watch
    4) Kohlrauch flask (50 ml)
    5) Thermometer.

3. Theory:

The viscosity of a fluid oil can be defined as the internal resistance offered by the fluid to the movement of the molecules of one layer over an adjacent layer of fluid due to the cohesion between the molecules of the fluid.

The Redwood Viscometer No.1, commonly used to measure the viscosity, is consist of vertical cylindrical oil cup with an orifice, also called agate jet, in the centre of its base. The orifice can be closed by a ball of a valve rod. A hook pointing upward serve as a guide mark for filling the oil. The cylindrical cup is surrounded by the water bath. The water bath maintain the temperature of the oil to be tested at constant temperature. The oil is heated by heating the water bath by means of an immersed electric heater in the water bath, the provision is made for stirring the water, to maintain the uniform temperature in the water bath and to place the thermometer to record the temperature of oil and water bath. The cylindrical shaped oil cup made up of brass which is silvered from inside, has the diameter of 46.5 mm and height of 90.00 mm. The orifice/agate jet at the centre, of the base of the oil cup, have diameter of 1.62 mm and 10 mm length, this viscometer is used to determine the kinematic viscosity of the oil.
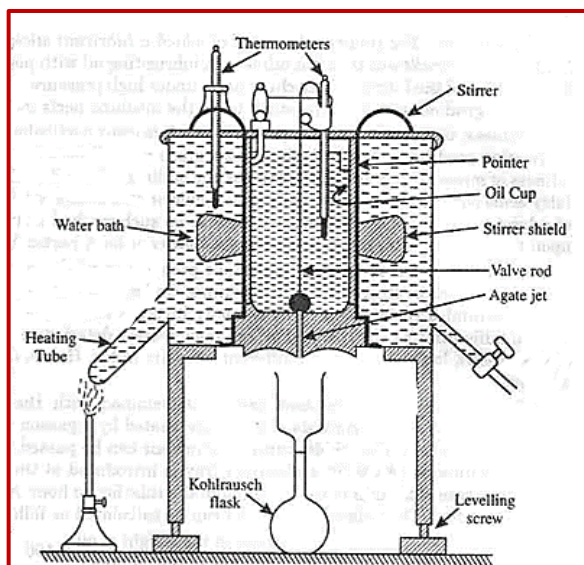
Diagram of Redwood viscometer No.1

Page No.: 33

Viscosity is the tangential force per unit area required to maintain a unit velocity gradient between two adjacent layers in the fluid which are unit distance apart. Theoretically viscosity is generally expressed in centipoise or centistoke. Viscosity of lubricating oil can be calculated by the following Formula:

$$v = ct - b/t$$
$$c = 0.00247 \text{ and } b = 0.50$$

Where c and b are constant and t is the time in second taken for the flow of 50ml of lubricating oil.

4. Procedure:

1) Fill the Redwood viscometer with the given lubricating oil and note the temperature.
2) Place clean and dried Kohlrauch flask just below the viscometer and remove the ball so that oil can flow from the viscometer into the flask. Start the stop watch immediately when ball is removed.
3) Stop the stop watch when oil is filled up to 50 ml mark of the Kohlrauch flask (observe the meniscus of the oil in the flask at 50 ml mark). Record the total time in seconds.
4) Repeat the experiment and note the time of flow of the oil.

5. Observations:

Temperature of lubricating oil at the time of experiment ....^{0}C.

Sr. No.	Volume of Lubricating oil (ml)	Time of flow (Seconds)	Average time (Seconds)
1.			
2.			
3.			

6. Calculations:
Viscosity = **ct - b/t** where c and b are constant and t is average time
$$c = 0.00247 \text{ and } b = 0.50$$

7. Result:

Viscosity of the given lubricating oil at ....^{0}C is...........Centipoise.

8. Precautions:
1) Clean the Kohlrauch flask thoroughly with petrol and dry it.
2) Viscometer should be thoroughly cleaned, there should be no dust in the oil cup.
3) Oil must be free from any dust or suspended particle.
4) Put the Kohlrauch flask just below the viscometer.
5) Note carefully the time for the flow of lubricating oil.

EXPERIMENT-10

1. Object: Synthesis of Aspirin.

2. Requirements:
   1) Erlenmeyer flask
   2) Measuring jar
   3) Beaker
   4) Stirring rod
   5) Buchner funnel
   6) Watch glass
   7) Salicylic acid
   8) Acetic anhydride
   9) Acetic acid
   10) Ethanol
   11) Sulphuric acid

3. Theory:

Aspirin is a trade name of acetylsalicylic acid, it has long been known as excellent analgesic antipyretic and anti-inflammatory drug.

Aspirin is the acetyl ester of a phenol called acetylsalicylic acid. Acetylation of an alcohol or phenol can be carried out with a variety of acid or base catalyst, and in this experiment we will use acetic acid as a catalyst. It is important that the apparatus should be scrupulously dry because a small amount of water will reverse the reaction, to reform the reactant. The product, a typical organic compound, is insoluble in water and is isolated by precipitation from water.

Salicylic Acid   Acetic Anhydride   Aspirin Acetylsalicylic Acid   Acetic acid
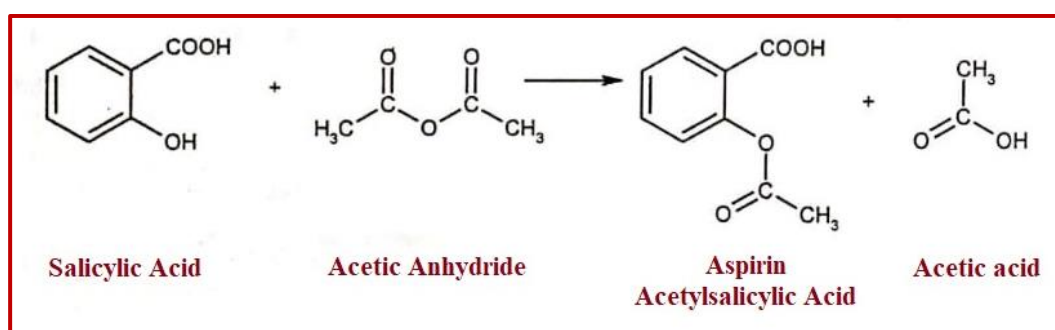
4. Procedure:

   1) Weigh out 2.0 gram of salicylic acid. Place it in a 125 ml Erlenmeyer flask.
   2) Add 5 ml of acetic anhydride. Swirl the flask to wet the salicylic acid crystals. Add 5 drops of concentrated sulphuric acid, H_2SO_4, to the mixture.
   3) Gently heat the flask in a boiling water bath for about 10 minutes.
   4) Remove the flask from the hot water bath and add 10 ml of deionized ice water to decompose any excess acetic anhydride.

5) Chill the solution in an ice bath until crystals of Aspirin no longer form, stirring occasionally to decompose residual acetic anhydride. If an oil appears instead of a solid, reheat the flask in the hot water bath until the oil disappears and again cool.

6) Set up a vacuum filtration apparatus. Wet the filter paper in the Buchner funnel with 1-2 ml of distilled water.

7) Turn on the water aspirator. Decant the liquid on to the filter paper, minimising any transfer of the solid aspirin. If some Aspirin is inadvertently transferred to the filter, that will not cause any difficulty.

8) Add 15 ml of cold water to the flask and chill again. Pour the liquid and the crystal of the aspirin onto the filter paper. Repeat until the transfer of the crystals to the vacuum filter is complete. Wash the aspirin crystals on the filter paper with 10 ml of ice water.

9) Maintain the vacuum to drive the crystal as best possible.

10) If Aspirin forms in the filtrate in the vacuum flask, transfer the filtrate and Aspirin to a beaker, chill in an ice bath, and vacuum filter as before, using a new piece of filter paper. Dispose of the filtrate in the sink.

11) The major impurity in Aspirin is salicylic acid it can be removed by recrystallization

12) Place the aspirin crystals in a 100 ml beaker. Add 8 ml of ethanol and 25 ml of water.

13) Warm the mixture in a 60^0C water bath (no flame, use a hot plate or a hot water bath). Warm the mixture until the aspirin dissolves.

14) Cover the beaker with a watch glass, remove it from the heat and set it aside to cool slowly. Set the beaker in an ice bath. Beautiful needle-like crystals of acetylsalicylic acid, aspirin, forms.

15) Collect the aspirin by vacuum filtration. Wash the crystals twice with 10 ml volumes of ice water. Maintain the vacuum to air dry the aspirin. If time does not permit, place the filter paper and aspirin sample on watch glass and allowed them to air-dry. The time for air-drying the sample may require that it be left with your instructor until the next laboratory period.

16) Transfer the dry Aspirin crystals to a pre-weighed sample container and determine the mass of the aspirin crystals.

5. Result:

Yield of aspirin obtained = ... gm.

6. Precautions:

1) Wear safety glasses, all the time, during the preparation of aspirin.

2) Acetic anhydride is corrosive and its vapor is irritating to the respiratory system and avoid its skin contact and inhalation. In the event of skin contact, rinse well with cold water. If its vapors are inhaled, move to an area where fresh air is available.

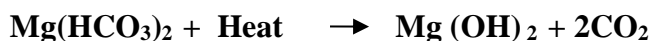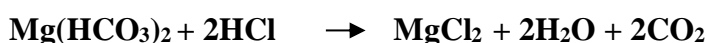3) Sulphuric acid is corrosive and avoid its skin contact. In the event of skin contact, rinse well with cold water.

EXPERIMENT-11

1. Object: To determine the total, temporary and permanent hardness of given water sample, equivalent as **CaCO₃**, in ppm by acid titration method.

2. Materials required :
1) Bottle A_1 = Tap water
2) Bottle A_2 = Boiled water
3) Bottle B = Standard HCl
4) Methyl Orange (MeOH) Indicator

3. Chemical reactions:

$$Ca(HCO_3)_2 + 2HCl \longrightarrow CaCl_2 + 2H_2O + 2CO_2$$

$$Mg(HCO_3)_2 + 2HCl \longrightarrow MgCl_2 + 2H_2O + 2CO_2$$

$$Ca(HCO_3)_2 + Heat \longrightarrow CaCO_3 + H_2O + 2CO_2$$

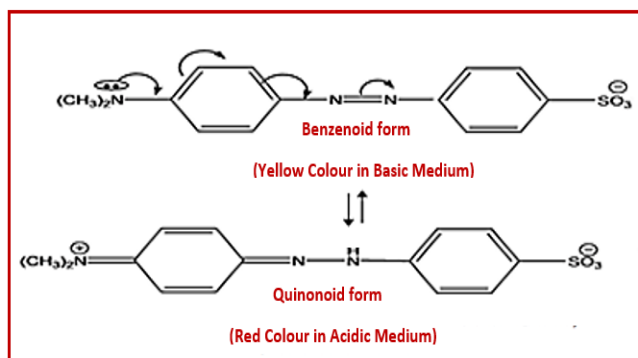$$Mg(HCO_3)_2 + Heat \longrightarrow Mg(OH)_2 + 2CO_2$$

4. Theory:

Hardness of water is the property of water which resists the formation of lather when soap is mixed. It is also defined as the soap consuming capacity of water. There are two types of hardness of water, temporary and permanent hardness. Temporary hardness is caused by the presence of bicarbonates of Ca and Mg which can be removed by prolonged boiling as it decomposes to carbonate of Ca and hydroxide of Mg with elimination of CO_2.

$$Ca(HCO_3)_2 \xrightarrow{\triangle} CaCO_3 + CO_2 + H_2O$$
$$Mg(HCO_3)_2 \xrightarrow{\triangle} Mg(OH)_2 + 2CO_2$$
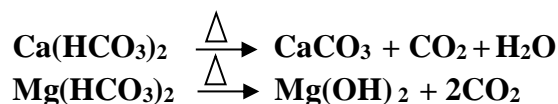
Temporary hardness in water arises due to bicarbonate salts of calcium and magnesium. These are alkaline in nature. Thus temporary hardness can also be determined by estimating the alkalinity of water sample before and after boiling it. For this purpose a standard solution of acid is used in the presence of indicator methyl orange. Before boiling the alkalinity is present and following chemical reaction occurs.

$$Ca(HCO_3)_2 + 2HCl \longrightarrow CaCl_2 + 2H_2O + 2CO_2$$
$$Mg(HCO_3)_2 + 2HCl \longrightarrow MgCl_2 + 2H_2O + 2CO_2$$

(CH₃)₂N — Benzenoid form (Yellow Colour in Basic Medium)

Quinonoid form (Red Colour in Acidic Medium)

Structure of Methyl Orange

5. Procedure:

1) Take burette and fill it with given standard HCl solution up to zero mark.
2) Take 25 or 50 ml of tap water in to a 250 ml conical flask and add a few drops of methyl orange, the solution turns yellow.
3) Start adding HCl solution from the burette into the conical flask drop by drop until the colour of the solution changes to red.
4) Note the volume of HCl consumed as **X_1 ml**.
5) Repeat the same procedure with boiled water and note the volume of HCl consumed as **X_2 ml**.

6. Observations:

Table A (for Tap Water)

Sr. No.	Volume of Tap water (ml)	Burette reading (ml)		Volume of HCl Consumed (ml)	
		Initial (I)	Final (II)	(II-1)	Concordant Reading X_1

Table B (for Boiled Water)

Sr. No.	Volume of Boiled water (ml)	Burette reading (ml)		Volume of HCl Consumed (ml)	
		Initial (I)	Final (II)	(II-1)	Concordant Reading X_2

7. Calculations:

(A) For Total Hardness of Water

$$N_1V_1 = N_2V_2$$

Where N_1 = Normality of given HCl
N_2 = Normality of tap water as hardness
V_1 = Volume of HCl consumed(X_1)
V_2 = Volume of Tap water (25 or 50 ml)

Normality of tap water as hardness, $N_2 = (N_1 \times V_1)/V_2$

Total hardness of given water sample, Equivalent as $CaCO_3$
 = **Normality of Tap water as hardness x Eq. Wt. of $CaCO_3$**
 = **N_2 x 50 gm/litre**
 = **$(N_1 \times V_1)/V_2$ x 50 x 1000 mg /litre (ppm)**

(B) For Permanent Hardness of Water

$$N_3 V_3 = N_4 V_4;$$

Where N_3 = Normality of given HCl

N_4 = Normality of Boiled water as hardness

V_3 = Volume of HCl consumed(X_2)

V_4 = Volume of Boiled water (25 or 50 ml)

Normality of boiled water as hardness, $N_4 = (N_3 \times V_3)/V_4$

Permanent hardness of given water sample, Equivalent as $CaCO_3$

= **Normality of boiled water as hardness x Eq. Wt. of $CaCO_3$**

= **N_2 x 50 gm / litre**

= **$(N_3 \times V_3)/V_4$ x 50 x 1000 mg/litre (ppm)**

(C) For Temporary Hardness of Water

Temporary hardness = Total hardness – Permanent hardness

8. Result:

1) Total hardness of given water sample, equivalent as $CaCO_3$ = ----------- ppm.
2) Permanent hardness of given water sample, equivalent as $CaCO_3$ = ----------- ppm.
3) Temporary hardness of given water sample, equivalent as $CaCO_3$ = ----------- ppm.

9. Precautions:

1) All the glass wares should be properly cleaned and dried.
2) Addition of reagents from the burette should be drop wise.
3) Be careful about colour change at the time of end point.
4) There should be no air bubbles inside the burette.
5) Note the reading of lower meniscus inside the burette in case of colourless solution and upper meniscus in case of coloured solution.