TEERTHANKER MAHAVEER UNIVERSITY

(Established Under Govt. of UP Act No. 30, 2008)

NH-24 Delhi Road, Moradabad-244001(U.P.)

Website: www.tmu.ac.in

Department of Chemistry

Lab Manual B.Tech

Engineering Chemistry Lab

Course Code EAS 163

Head
Dept. of Chemistry
College of Engineering
Teerthanker Mahaveer University
Moradabad

Semester I
Engineering Chemistry Lab

Course Code: EAS163/263

L T P
0 0 2

LIST OF EXPERIMENTS

Note: Select any ten experiments from the following list.

1. Determination of Total Hardness of a given water sample.
2. Determination of mixed alkalinity (a) Hydroxyl & Carbonate (b) Carbonate & Bicarbonate
3. To determine the pH of the given solution using pH meter and pH-metric titration.
4. Determination of dissolved oxygen content of given water sample.
5. To find chemical oxygen demand of waste water sample by potassium dichromate
6. Determination of free chlorine in a given water sample.
7. To determine the chloride content in the given water sample by Mohr's method.
8. To prepare the Bakelite resin polymer.
9. To determine the concentration of unknown sample of iron spectrophotometrically.
10. To determine the viscosity of a given sample of a lubricating oil using Redwood Viscomete
11. To determine the flash & fire point of a given lubricating oil.
12. Determination of calorific value of a solid or liquid fuel.
13. Determination of calorific value of a gaseous fuel.
14. Determination of % of O_2, CO_2 % CO in flue gas sample using Orsat apparatus.
15. Proximate analysis of coal sample.

Evaluation Scheme of Practical Examination:

Internal Evaluation (50 marks)

Each experiment would be evaluated by the faculty concerned on the date of the experiment on point scale which would include the practical conducted by the students and a Viva taken by faculty concerned. The marks shall be entered on the index sheet of the practical file.

Evaluation scheme:

PRACTICAL PERFORMANCE & VIVA DURING THE SEMESTER (30 MARKS)			ATTENDANCE (10 MARKS)	VIVA (10 MARKS)	TOTAL INTERNAL (50 MARKS)
EXPERIMENT (10 MARKS)	FILE WORK (10 MARKS)	VIVA (10 MARKS)			

External Evaluation (50 marks)

The external evaluation would also be done by the external Examiner based on the experiment conducted during the examination.

EXPERIMENT (20 MARKS)	FILE WORK (10 MARKS)	VIVA (20 MARKS)	TOTAL EXTERNAL (50 MARKS)

VOLUMETRIC ANALYSIS

Quantitative analysis can be done by two methods:

1. Gravimetric analysis

2. Volumetric analysis

Volumetric analysis is a technique of finding the volume of one solution which will exactly react with a definite volume of known concentration and then the strength of unknown solution can be calculated using the law of equivalence or normality equation.

According to law of equivalence, the substances react with each other in definite ratio by mass.

Number of gram equivalence of acid = Number of gram equivalence of acid.

$$N_1V_1 = N_2 V_2$$

Where N_1 and N_2 are Normality of acid and base and V_1 and V_2 are volume of acid and base respectively.

Common word used in volumetric analysis

1. Titration: The process of finding of volume of one of solution required to react completel with the definite volume of the solution is known as titration.

2. Titrant: The solution of known strength is called as titrate.

3. Titrate: The solution which contain a substance to estimated or whose strength has to be estimated.

4. End point and Equivalence point: The point at which completion of reaction occurs is called equivalence point where as end point is an observable physical change that occurs nea the equivalence point.

5. Indicator: The substance which indicate the end point of titration by observable color change in the reaction.

6. Strength: The amount of substance dissolved in on liters of solution when expressed in gram is termed as strength of solution. Strength of solution can also be expressed in number of ways given below.

(à) Normality: It is the number of gram equivalent of substance dissolved per liter of solution.

It is represented by N

(b) Molaritiy: It is the number of moles of solute present per liter of solution. It is denoted b M

(C). Molality: It is the number of mole of the substance in 1000 gm or 1 kg of solvent. It is denoted by m

(d) Formality: It is the number of gram formula weight of substance dissolve in one litre of solution. It is represented by F

7. Equivalent Weight: The equivalent weight of substance is defined as the number o part by weight of it combine or displace one part by weight of hydrogen or eight part by weight of oxygen or 35.5 part by weight of chlorine. Equivalent weight expressed in gram is called gram equivalent weight.

(a) Equivalent weight of base = Molecular weight of base/Number of replicable Hydroxyl grou present in one molecule of base(or Acidity).

(b) Equivalent weight of an oxidizing agent or Reducing agent=Molecular mass of oxidizing or reducing substance/Number of electron gain or lost by one molecule in Redox reaction.

(c) Equivalent weight of an acid: Molecular weight of acid/ No. of replicable Hydrogen atom i one molecule of acid (or basicity)

8. Standard solution: A standard solution is known concentration is known as standard solution.

(A) Primary standard solution: Any substance whose standard solution can be prepared directly by dissolving an accurately weight amount of it in water and making up the solution t known volume by diluting it with water. A primary standard substance possesses the followir characteristic

(a) It is available in highly pure state.

(b) It has high equivalent weight so that error in weighing is minimum.

(c) It is readily soluble in water or desire solvent.

(d) It is stable at oven temperature used for drying.(Hydrated substance are not used as primary standard)

(e) it is stable and unaffected by air

(f) Common used primary standard solution are crystalline oxalic acid, Anhydrous sodium carbonate, dried KCl, $AgNO_3$, $FeSO_4$ (NH_4) $6H_2O$.

(B) **Secondary standard solution**: The substance which does not fulfill the above requirement and their standard solution are not prepared directly by weighing are called secondary standard substance.

(a) Alkali hydroxide like NaOH, KOH etc as they are hygroscopic and weights keep on changi during weighing.

(b) $KMnO_4$ as it is not available in pure form and is associated with MnO2 which catalyzed the auto decomposition of per magnet solution on standing.

(c) Inorganic acid HCl, H_2SO_4 etc because their concentration are known only approximate.

Eriochrome black –T indicator is used in the determination of hardness of water.

Erichromeblack- [Sodium-1-(1-hydroxy-2-naphthylazo)-6-nitro-2-naphthol-4-sulphonate]

For simplicity EBT is represented as NaH_2in, EBT show different color in different pH range

$$\begin{array}{ccccc} & 7.0 & & 11.5 & \\ H_2in^- & \rightleftharpoons & Hin^{2-} & \rightleftharpoons & In^{3-} \end{array}$$

Red 5.5 Blue 11.0 Yellowish orange.

In the p^H range 7-11, indicator forms weak complexes with metal ions and imparts wine red color to the solution.

$$Hin^{2-} + M^{2+} \longrightarrow Min^- + H^+$$

During hardness determination p^H is maintained by adding p^H-10 buffer solution. When indicator is added to hard water sample containing buffer wine red colour generates due to the formation of Mg indicator complex.

$$Hin^{2-} + Mg^{2+} \longrightarrow \underset{\text{Wine red}}{Mgin^-} + H^+$$

Now during titration as EDTA is added, it forms complexes with free Ca^{2+} as Ca EDTA complex is more stable.

$$Ca^{2+} + H_2Y^{2-} \longrightarrow CaY^{2-} + 2H^+$$

Remaining free Mg^{2+} ions then from complex with EDTA forming Mg EDTA complex which is less stable as compare to Ca EDTA complex.

$$Mg^{2+} + H_2Y^{2-} \longrightarrow Mgy^{2-} + 2H^+$$

Mg EDTA complex is more stable than Mg Indicator complex so on further addition of EDTA Mg Indicator complex dissociate and formation of more stable Mg EDTA complex takes place this result in libration of free indicator in the solution which in this pH range give blue color.

$$Mgin^- + H_2Y^{2-} \longrightarrow MgY^{2-}\downarrow + Hin^{2-} +$$

1.3

$Mgin^{-} + H_2Y^{2-} \longrightarrow MgY^{2-}\downarrow + Hin^{2-} +$

Procedure:-

Step-1:-<u>Determination of total hardness</u>

1. Pipette out 25 mL of the hard water sample into a conical flask and add 2 mL buffer and few drops of EBT indicator to it.
2. Titrate the resulting solution against Std. EDTA solution till blue color appears.
3. Take three concordant readings and record the volume of EDTA used as B mL.

Observation Tables:

Table-1:-Determination of total hardness

Sl. No.	Initial burette reading	Final burette reading	Volume of EDTA used (mL)
1.			
2.			
3.			
4.			
		Concordant reading =	A mL

Calculations:-

Step-2:- Determination of total hardness

$$\text{(EDTA)}\ N_1V_1 = N_2V_2\ \text{(Hard Water)}$$

$$N_2 = \frac{N_1V_1}{V_2}$$

$$N_2 = \frac{1}{50} \times \frac{A}{25}$$

Strength in terms of $CaCO_3$ equivalents = Normality x Equivalent Wt. of calcium carbonate

$$= \frac{1}{50} \times \frac{A}{25} \times 50\ g/L$$

1.4 --

$$\text{Strength in } \frac{mg}{L} \text{ or ppm} = \frac{1 \quad A}{50 \quad x\,25} \; x\,1000 \; \frac{mg}{L} \text{ or ppm}$$

$$= \frac{1}{25} \; x\,A\,x\,200 \text{ ppm}$$

Result:-

Total hardness of the water sample = ppm

Hardness due to Calcium = ppm

Hardness due to Magnesium = ppm

VIVA – VOCE

Q.1. What is hardness of water? Define temporary and permanent hardness?
Q.2. What are buffer solutions?
Q.3. What are different unit of hardness of water and how they are related?
Q.4. What is the significance of hardness determination?
Q.5. Why metal-EDTA complex are more stable than metal-EBT complexes?
Q.6. What is the function of EBT in complex metric titration?
Q.7. What is a chelate?
Q.8. Why and how is the PH-value adjusted to about 10?

EXPERIMENT-2

Aim: - To determine of mixed alkalinity (a) Hydroxyl & Carbonate (b) Carbonate & Bicarbonate of given water sample.

Apparatus required: - Burette, pipette, conical flask, funnel and dropper etc.

eagent required: - 1. Hydrochloric acid (~N/50)

2. Na_2CO_3 (N/50)

3. Phenolphthalein indicator

4. Methyl orange indicator

Theory:-

Alkalinity is a measure of capacity of water to neutralize acid and is mainly caused by following ions present in water.

1. OH^-
2. CO_3^2
3. HCO_3^-

Alkalinity in water can be determined by titrating the water sample with standard acid solution using phenolphthalein and methyl orange indicator. All the alkalinity causing ions are sensitive to methyl orange, hydroxide and carbonate ion are sensitive to phenolphthalein indicator. Following set of reactions takes place on addition of acid.

$$\left.\begin{array}{l}\left.\begin{array}{l} OH^- + H^+ \longrightarrow H_2O \\ \\ OH_3^{2-} + H^+ \longrightarrow HCO_3^- \end{array}\right] P \\ HCO_3 + H^+ \longrightarrow H_2O + CO_2 \end{array}\right] M$$

Where P indicates phenolphthalein sensitive ions and M indicate methyl orange sensitive ions. Further hydroxide and bicarbonate ions cannot exist together in water due to the following reaction.

$$HCO_3 + OH \longrightarrow H_2O \quad + \quad CO_3^{2-}$$

2.2

Therefore there exist five possibilities of alkalinity in water on the basis of concentration of various ions as tabulated in the table.

TABLE-1:-SHOWING DIFFERENT POSSIBILITIES OF ALKALINITY

TITRATION RESULTS	VOLUME OF ACID USED FOR OH^- IONS	VOLUME OF ACID USED FOR CO_3^{2-} IONS	VOLUME OF ACID USED FOR HCO_3^- IONS
P = 0	ABSENT	ABSENT	M
P = M	P = M	ABSENT	ABSENT
$P=\frac{1}{2}M$	ABSENT	2P OR M	ABSENT
$P>\frac{1}{2}M$	2P - M	2(M – P)	ABSENT
$P<\frac{1}{2}M$	ABSENT	2P	M – 2P

Where P indicate phenolphthalein and M indicate methyl orange indicator.

Procedure:-

Step-1:- Determination of Alkalinity:-

1. Pipette out 50mL alkaline water in the conical flask add few drops of phenolphthalein indicator to obtain pink color.
2. Titrte the content for the flask with HCI solution till pink color disappear record the reading as Y ml.
3. Now add methyl orange indicator to the same flask to obtain yellow color.
4. Titrte the content of the flask with HCI solution till orange color appears record the reading as Z mL.
5. Take three concordant readings.

OBSERVATION TABLE-2:-

Sl. No.	Volume of water sample taken	Initial reading (X)	Reading up to P end point (Y)	Volume of acid used up to P end point (Y-X mL)	Reading up to M end point (Z)	Volume of acid used up to M end point (Z-XmL)
1						
2						
3						
					Concordant reading for M end point =	mL

CALCULATIONS:-

Step-1:- **Calculations for Determination of alkalinity** :-

Note: - before starting calculations first locate the type of alkalinity present in test sample form the tabnle-1 and the calculate alkalinity for the ions present.

(Alkaline water) $N_1V_1 = N2V2$ (HCI solution)

$$N_1 = \frac{N_2 \times N_2}{V_1}$$

Where

$N_2 = 1/50$

V_2 = Volume of acid consumed to be calculated for each iron using table – 1

V_1 = Volume of water sample taken i.e. 25 mL.

Strength in terms of Ca CO_3 equivalents = Normality x Eq. wt. of $CaCO_3$ (g/L)

Alkalinity in ppm = Normality x 50 x 1000 (mg/L or ppm).

2·4

RESULT:-

Alkalinity due to OH^- --

Alkalinity due to CO_3^{2-}--

Alkalinity due to HCO_3 --

VIVA – VOCE

Q.1. Define alkalinity . What is the significance of determining alkalinity in water?
Q.2. How much alkalinity is acceptable for domestic use/
Q.3. What is an indicator? What is phenolphthalein and methyl orange?
Q4. What is the difference between the end point and equivalence point?

Head
Dept. of Chemistry
College of Engineering
Teerthanker Mahaveer University
Moradabad

EXPERIMENT-3

JECT-PH-metric titration.

QUIREMENTS

emicals/Reagents: Buffer solution of pH 4 and 9 (acidic and basic), unknown solution of acid, 0, 1 N NaOH.

PARATUS

pH meter, glass electrode, reference electrode, beaker, stirrer, burette.

RINCIPLE

(a) The pH of a solution is defined as the logarithm of the reciprocal of $[H^+]$ ion concentration.

$$pH = \log 1/[H^+]$$

(b) The pH of the acidic solution increases slowly when an alkali is added, but near the equivalence pint, the rate of change of pH of the solution becomes very fast. From the sharp break in the curve one can find out the equivalence point, from which the strength can be calculated by normality equation.

ROCEDURE

Standardize the pH meter by dipping the electrode into the buffer solution of known (pH 4 for acidic solution and pH 9 for basic solution). Use basic buffer to determine the H of the basic solution and use acidic buffer to determine the pH of the acidic solution. ake out the electrode, wash with distilled water, dry and dip in the test solution. Switch the pH meter and mote down the pH of the solution directly.

ndardize the pH meter with acidic buffer (pH 4) as above. Wash the electrode with istilled water and dry. Take 50 ml of HCL solution in a beaker and dip the electrode ompletely into it. Note the pH of the pure acid solution. Add to this solution 1 ml of 0.1 N OH from the burette, stir well and note down the pH OF the solution. Similarly, go on dding the alkali up to say 9-10 ml. when pH change occurs rapidly (equivalence point), dd alkali infractions (0.2ml). you can also use the NaOH solution of different normality.

BSERVATION

3.2

OBSERVATION

Volume of HCL taken = 50 ml.

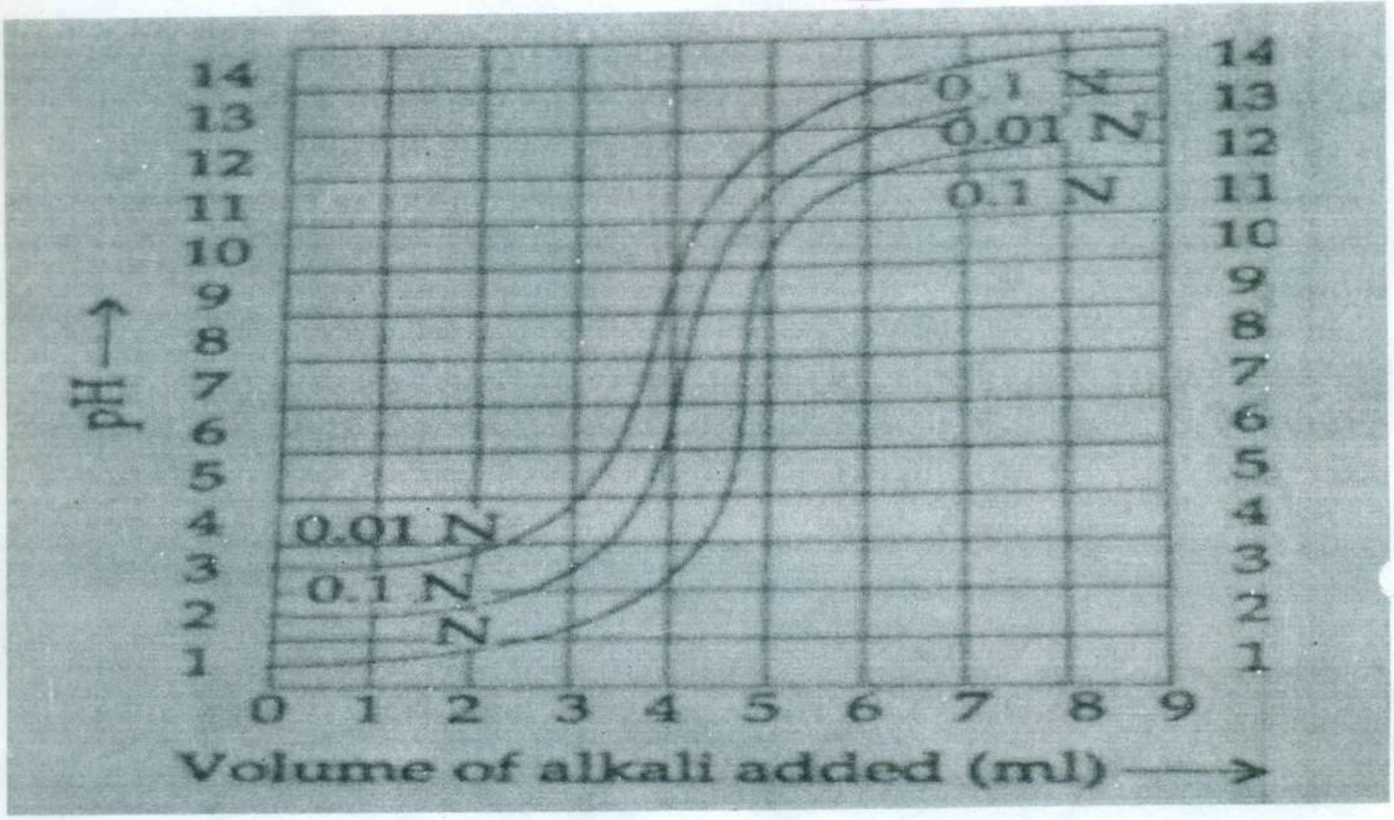
Volume of alkali added (ml)									

CALCULATION

Plot a curve with pH values as ordinate and the volume of the alkali added (ml) as abscissa. Find out the end point.

Suppose, volume of alkali used at the end point = v_2 ml

$$\underbrace{N_1 * V_1}_{Acid} = \underbrace{N_2 * V_2}_{Alkali}$$



RESULT:

(a)The Ph of the given solution is.........

(b }The strength of given acid solution.........gm/liters

PRECUTION:

1. The electrodes must be washed thoroughly and dried before dipping into solution.

-log

log 5x10

log5 + ~~log~~

log5

3.3

After each addition of alkali, the solution should be stirred thoroughly.

The knob of pH meter should be on standby position at the time of adding alkali in the acid solution.

After addition of alkali shift the knob from stand by position to pH side and at the time of taking the pH meter reading it should be constant.

VIVA-VOCE

Q.1 Define pH.

Ans. pH is a scale to measure the strength of H^+ ion.

$$pH = -\log_{10}[H^+]$$

Q.2 Name the electrodes used in pH titration.

Ans. Hydrogen electrode, Glass electrode, Quinhydrone electrode.

Q.3 What is the pH range for drinking water?

Ans. 7.0 – 8.5.

4.1

EXPERIMENT – 4

AIM: - To determine dissolved oxygen in given water sample.

Apparatus required: - Burette, pipette, iodine flask, conical flask, funnel and dropper etc.

Reagent required: -

1. Standard Sodium thiosulphate solution (N/40)
2. Potassium permanganate solution (N/10)
3. Potassium oxalate solution (2%)
4. Manganous sulphate solution (48%)
5. Alkaline potassium iodide solution.
6. Freshly prepared starch solution.

Theory:-

The iodometric (WINKLER) method is based on the fact that in alkaline medium dissolved oxygen oxidizes Mn_2^+ to Mn_4^+, which in acidic medium oxidizes 1^- to free iodine. The amount of iodine released, which can be titrated with standard sodium thiosulphate solution, is thus equivalent to D.O. originally present in water sample. Certain ions like NO_2^-, SO_3^2 etc are removed by treating the sample with an excess of $KMnO_4$ in acidic medium as they interfere in the D.O determination.

$$NO_2 + H_2O \longrightarrow NO_3^- + 2H^+ + 2e^-] \times 5$$
$$MnO_4 + 8H^+ + 5e \longrightarrow Mn^{2+} + 4H_2O] \times 2$$
$$2\,MnO_4 + 6H^+ + 5NO_2 \longrightarrow 5\,NO_3^- + 2Mn^{2+} \quad 3H_2O$$

$$SO_3^{2-} + H_2O \longrightarrow SO_4^{2-} + 2H^+ + 2e^-] \times 5$$
$$MnO_4^- + 8H^+ + 5e \longrightarrow Mn^{2+} + 4H_2O] \times 2$$
$$2\,MnO_4^- + 6H^+ + 5SO_3^{2-} \longrightarrow 5SO_4^{2-} + 2Mn^{2+} + 3H_2O$$

Excess of $KMnO_4$ is removed by adding potassium oxalate solution.

$$MnO_4^- + 8H^+ + 5e^- \longrightarrow Mn^{2+} + 4H_2O] \times 2$$
$$C_2O_4^{2-} \longrightarrow 2CO_2 + 2e^-] \times 5$$
$$2\,MnO_4^- + 16H^+ + 5C_2O_4^{2-} \longrightarrow 2Mn^{2+} + 10CO_2 + 8H_2O$$

On addition of $MnSO_4$ and alkaline Kl formation of white ppt. of takes place.

$$Mn^{2+} + 2OH^- \longrightarrow \underset{\text{White ppt.}}{Mn(OH)_2}$$

The oxygen present in water oxidizes Mn^{2+} to Mn^{4+} which is precipitated as brown hydrated manganese dioxide.

Head
Department of Chemistry
College of Engineering
Teerthanker Mahaveer University
Moradabad

4.2

$Mn^{2+} + 4OH^- \longrightarrow MnO(OH)_2 + H_2O + 2e^-] \times 2$

$O_2 + 2H_2O + 4e^- \longrightarrow 4OH^-$

$2Mn^{2+} + O_2 + \cancel{4\,OH} \longrightarrow MnO(OH)2 \text{ or } (2MnO_2H_2O)$

Brown ppt.

This process is sometimes reffered to as fixation of oxygen.

On acidifying the solution, $^{4+}$ oxidizes 1^- (Iodide ion) to free iodine.

$MnO(OH)_2 + 4H^+ + 2e^- \longrightarrow Mn^{2+} + 3H_2O$

$21^- \longrightarrow 1_2 + 2e^-$

$MnO(OH)_2 + 4H^+ + 21^- \longrightarrow Mn^{2+} + 1_2 + 3H_2O$

The iodine librated is titrated with standard $Na_2S_2O_3$ solution using freshly prepared starch solution as indicator.

$1_2 + 2e^- \longrightarrow 21^-$

$2\,S_2O_3^{2-} \longrightarrow S_4O_6^{2-} + 2e^-$

$1_2 + 2\,S_2O_3^{2-} \longrightarrow 21^- + S_4O_6^{2-}$

Procedure:-

1. Fill iodine flask (300mL) with tap water.
2. With the help of pipette add 0.9mL conc. H_2SO_4
3. Now add 0.2 m 4 drops $KMnO_4$ solution
4. Stopper the flask and shake to mix the contents
5. Leave the flask for about 5 minutes if pink color disappears then add one more drop of $KMnO_4$ solution.
6. Now add 0.5mL of potassium oxalate solution to the flask to remove excess of $KMNO_4$.
7. Leave the flask for about 10 minutes if pink color of $KMnO_4$ still persists add additional amount of potassium oxalate to discharge the color.
8. Now add2mL of $MnSO_4$ solution followed by 3mL of alkaline K I solution resulting in the formation of brown ppt. allow the ppt. to settle down.
9. Add 1mL of conc. H_2SO_4 to re-dissolve ppts.
10. Take 102.2mL of this solution in a titration flask and titrate slowly against N/40 $Na_2S_2O_3$ solution, when the color of this solution becomes light yellow add about 2mL freshly prepared starch solution and continue titration till the disappearance of blue color. Take two readings.

Head
Dept. of Chemistry
College of Engineering
Teerthanker Mahaveer University
Moradabad

4.3

OBSERVATION AND CALCUTATIONS:-

OBSERVATION TABLE:

Sl. No.	Initial Reading	Final Reading	Volume of $Na_2S_2O_3$ used mL
1.	0	3.5	3.5
2.	3.5	7.0	3.5
		Mean =	A mL

Total volume of sample taken = 300 mL

Reagents added during preparation of iodine solution

0.9ml Conc. Sulphuric acid.

0.2mL $KMnO_4$

0.5mL $K_2C_2O_4$

2.0mL $MnSO_4$

3.0mL alkaline KI

TOTAL = 6.6 mL

CALCULATIONS:-

Volume of the prepared solution taken = 102.2mL

Volume of the original sample that will be equivalent to 102.2mL of the prepared solution is

$$= \frac{\text{Total vol. of sample – Vol.displaced by reagent}}{\text{Total volume of sample}} \times 102.2$$

$$= \frac{300 - 6.6}{300} \times 102.2$$

100 mL

Thus iodine content of 102.2 mL of the prepared solution is equivalent to the D.O. of 100mL of the water sample taken.

Using normality equation.

(WATER SAMPLE) $N_1 \times V_1 = N_2 \times V_2$ $\left[\frac{N}{40} \text{ HYPO SOLUTION}\right]$

$$N_1 \times 100 = \frac{1}{40} \times A$$

$$N_1 = \frac{1}{100} \times \frac{1}{40} \times A$$

$$N_1 = \frac{1}{100} \times \frac{1}{40} \times A$$

Strength = Normality x Eq. Wt.

Strength = $\frac{1}{100} \times \frac{1}{40} A \times 8$ g/L

$= \frac{2^*}{1000} A * 1000$ mg/L

= 2A mg/L or ppm.

RESULT:-

Dissolved oxygen present in the given water sample = --------------------ppm

VIVA - VOCE

Q.1. What is dissolve oxygen? What is it's function? Name the method by which dissolve oxygen is determine. What is utility of finding dissolved oxygen?

Q.2. What are iodometric titration?

Q.3. How dissolved oxygen from water can be removed?

Q.4. What do you understood by fixation of dissolved oxygen?

Q.5. What is the effect of the presence of oxidizing impurities ($No2^-$ and fe^{+++}) and reducing impurities (fe^{x2}, So_3^{-2}, S^{--}etc) effect the D.O. determination?

Q.6. State Hunry's Law for the solubility of gas in a liquid. Outline the condition under which the law is applicable?

Experiment No-5

BJECT: - To determine chemical oxygen demand (C.O.D.) of a given sample.

PPARATUS:-

1. Sox let apparatus
2. C.O.D. flask 4 no's
3. Condenser-6 no's
4. Burette
5. Pipette 1 ml, 10ml, and 100ml cap.
6. Funnel etc.

Reagent:-

1. Potassium dichromate solution ($K_2Cr_2O_7$)-0.25 N
2. Standard of ferrous Ammonium Sulfate $Fe(NH_4)_2(SO_4)_2.6H_2O$-0.1 N
3. Ferro in indicator.
4. Mercuric Sulfate (Hg So_4)
5. COD Acid (Ag_2SO_4 mixed H_2SO_4)

inciple:

When organic matter present in sample are oxidized by strong oxidant and after mixing the oxidant sample is boiled then many such organic matter and other compound are oxidized by bacteria. Thus required oxygen for the chemical oxidation of organic matter and other impurity present in the sample is called chemical oxygen demand (COD).

This test complete in less time than BOD test. It takes about 5 hours while BOD test take 3 to 5 days. This test is sample. COD value can be determined very accurately. Interferes in this test are very much less.

In this test sample water is heated by mixing $K_2Cr_2O_7$ and Some H_2SO_4 in it. Some $K_2Cr_2O_7$ consume in oxidation and remaining $K_2Cr_2O_7$ is determined by titrate against ferrous sulfate. Consume $K_2Cr_2O_7$ Proportional to COD.

METHOD:-

1. Wash the all apparatus cleanly.
2. First determine the normality of ferrous ammonium sulfate accurately.
   For it take 10 ml $K_2Cr_2O_7$, 90 ml distilled water and 30 ml cons. H_2SO_4 in a conical flask.
3. Let the flask cool down. Now add 3-4 drops of Ferro indicator.

4. Now fill the burette with 0.1N ferrous ammonium sulfate and titrate the solution of flask till wine red color appears. Note the volume of ferrous ammonium sulfate in ml.
5. Calculate the normality of ferrous ammonium sulfate by the relation given below:
   Normality of Fe $(NH_4)_2(SO_4)_2.6H_2O$=volume of $K_2Cr_2O_7$ in ml X0.25/Volume of $Fe(NH_4)_2(SO_4)_2.6H_2O$ in ml

6. After this take 6 No. COD flask and paste 1 to 6 No. on them. Tke 0.4gm Hg_2SO_4 in flask No. 1 and 2 and add 20ml COD acid slowly. During addition of COD acid swirl the flask till eddy current generates. Now in the flask No. 1and blank solution is ready to use.

7. Now take 0.4ml Hg_2SO_4 crystal in flask No. 3 and No. 6 each and sake well by this weak chloride present in the sample change into ionized $HgCl_2$ add 10ml K2Cr2O7 , 30ml COD acid slowly Swirl the flask simultaneously. By this evaporation of volatile substances substance (like fat acid) is prevented. It color of solution turn green then again prepare this solution by taking more dilute sample and adding all above reagent till green color does not appear. Remember that the ratio of $K_2Cr_2O_7$ sample or dilution water and COD acid should remain in 1:2:3 ratios.

1 : 2 : 3
10 20 30

8. After that keep the entire flask on the mesh of Sox let heater and switch on heaters. Start the flow of cold water throws the condensers. Set the energy regulators of heaters such that the solution of the flask boils slowly. Boiling should not be fast. Due to acid these solution (Refluxing) continue for 2 hours.
9. Now stop the heaters and let the flask cool down. After cooling wash the condenser with distilled water from top and let the water to come in flask. Now remove the flask from Soxhlet apparatus and add 80ml distilled water in each flask. The solution will turn blue green. Now titrate with Fe ($NH_4)2(SO4)_2$ 6H_2O. Solution till wine red color appears and note the volume of Ferrous Ammonium Sulfate consumed in ml.
10. Take the average reading of flask No. 1 and No. 2. Let the value is 'a' ml. Now note reading which are same are same or approximately same in flask No. 3 and No. 6`. Let it is' b' ml. Now calculate by relation.

CALCULATION:

COD (mg/L) = (a-b). N. 8000/Volume of sample in water in ml

Where

a = Consumed Ferrous Ammonium Sulfate to blank solution (Average value)
b = Consume ferrous ammonium sulfate for sample water.(concordant reading)
N = Accurate Normality of standard ferrous ammonium sulfate

RESULT:

The COD of given sample is.......................mg/L.

PRECAUTION:

1. In titration Ferrous Ammonium Sulfate should be added slowly in drop wise and sake flask continuously during titration.
2. C O D acid be added slowly and shaking flask must be continue by this evaporation of volatile matter does not take place.
3. Quantity of $HgCI_2$ to be added in solution should be taken 0.4gm to 1.0gm according to the possible chloride in solution. This quantity need not to be taken accurately.

~~6.1~~ 5.1

EXPERIMENT – 6

AIM: - To determine Free chlorine in given water sample.

REQUIREMENT:

APPRATUS USED: Burette, pipette, Measuring flask, conical flask, funnel, morter ar paste.

Chemical/Reagents used: given sample of bleaching powder potassium iodide, dilu acetic acid(5%), N/10 Sodiumthiosulphate solution, distilled water Freshly prepar starch solution.

PRINCIPLE:

Bleaching powder is mixture of calcium hypo chloride [$Ca(OCI)_2$] and the basic chloride,

$CaCI_2 . Ca(OH)_2.H_2O$ and free calcium hypo chloride.[$Ca(OH)_2$]

The active constituent of bleaching powder is hypo chloride $Ca(OCI)_2$, which produc hypo chlorous acid(HOCI), Responsible for disinfectant property.

Available chlorine in bleaching powder is amount of chlorine liberated by action of dilu acid on bleaching powder.

$$CaOCI_2 \ \overset{Hcl}{\underbrace{+2CH_3\,COOH}} \rightarrow (CH_3\,COOH)_2Ca + H_2O + CI_2$$

The liberated chlorine react with K I and give free iodine.

$$2KI + CI_2 \rightarrow 2KCI + I_2$$

The free iodine is then titrated with standard sodium thiosulphate solution.

The free iodine is then titrated with standard sodium thiosulphate solution.

$$2Na_2S_2O_3 + I_2 \rightarrow 2NaI + Na_2S_4\,O_6$$

PRECEDURE:

1. Weight about 2-3 gm of bleaching powder in a clean and pre-weighted tube. Transf the sample into a mortar, crush with paste and make a paste and make of paste bleaching powder sample with water. Transfer the paste into 250 ml volumetric flask a rinse the mortar with distilled water and again transfer it to the volumetric flask. Aft

transferring all the bleaching powder to volumetric flask, add more distilled water make the solution up to the mark and shake until a homogeneous suspension is obtained.

2. Pipette out 25 ml of this in conical flask, add 2 gm of KI and halt test tube of dilute ace acid to the solution.

3. Titrate liberated iodine immediately with standard N/10 sodium thiosulphate soluti till light yellow color persist. Then add 5 drop of freshly prepared starch solution, a bl color appear. Again continue titration continue till blue color disappear, note down t reading of the burette.

4. Repeat the same procedure of titration to get two concordant reading.

OBSERVATION:

Weight of empty weighing tube = a gm.

Weight of empty weighing tube + Bleaching powder = b gm.

Weight of bleaching powder = (b - a)

Normality of hyposolution = N/10

OBSERVATION TABLE:

S. No.	Volume of water sample in ml	Iniitial reading of Burette.	Final reading of burette	Volume of hyposolution used in ml

CALCULATION

$$(\text{Water sample})\, N_1V_1 = N_2V_2\, (\text{Hypo solution})$$

$$N_1 = N_2V_2/V_1$$

Normality of available chlorine $N_1 = N_2/10 * V_2/25$

Amount of chlorine per liter = $N_1 * 35.5/1000$ gm/Liter

% of available chlorine = $N_1 * 35.5/1000 * 250 * 100/(b - a)$

5.3

Result;

The percentage of free chlorine present in given sample of water is ....gm/liter.

PRECUTION:

1. The solution of bleaching powder is very unstable and hence should be titrate immediately after its preoeration.
2. All the reagent must be freshly prepared.
3. The starch solution and acetic acid must be added in equal amount in each experiment

Head
Dept. of Chemistry
College of Engineering
Teerthanker Mahaveer University
Moradabad

Experiment No-7

Aim- To determination of chloride content in given water sample by Mohar's method.

APPRATUS: Burette, pipette, conical flask, volumetric flask , droper.

CHEMICAL : Standard (N/50) $AgNO_3$ Solution, N/20 NaCI solution,water sample.

INDICATOR: Potassium chromate (K_2CrO_4) as an internal indicator.

PRINCIPLE: Chloride usually occure in water as NaCI $MgCI_2$ and $CaCI_2$. In the determination of chloride content by Mohar's, a weak alkaline solution(pH 7-8) of the sample is titrated against standard silver nitrate solution using K_2CrO_4 internal indicator. CI^- Ions present in water, react with Ag^+ ions giving transient colour, which disappear due to the presence of greater concentration of CI^- Ions.

$$NaCI + AgNO_3 \longrightarrow AgCI + NaNO_3$$

$$2AgNO_3 + K_2CrO_4 \longrightarrow Ag_2CrO_4 + KNO_3$$

$$Ag_2CrO_4 + 2CI^- \longrightarrow AgCI + CrO_4^{2-}$$

As the titration proceeds all the chloride ions are removed as AgCI, any extra drop of $AgNO_3$ react with potassium chromate(K_2CrO_4) giving reddish brown color indicate the end point.

$$2AgNO_3 + K_2CrO_4 \longrightarrow Ag_2CrO_4 + KNO_3$$

PROCEDURE:

1. Pipette out 20ml of water sample in a 150ml conical flask. Now add 4 drop of K_2CrO_4 indicator solution. Titrate it against standard solution of N/50 $AgNO_3$ till brown color of Ag_2CrO_4 persist. Note the volume of $AgNO_3$ solution from burette. Repeat the experiment till two concordant reading are obtained.
2. Pipette out 20ml of distilled water in 150ml of conical flask. Now add 4 drop of K_2CrO_4 indicator solution. Titrate it against standard N/50 $AgNO_3$ solution till the reddish color of Ag_2CrO_4 persist. Note the volume of $AgNO_3$ solution from burette. Repeat the the experiment till concordant reading are obtaion.

OBSERVATION:

(I) Titration with water sample(unknown)

S No.	Volume of sample in ml	Initial reading	Final reading	Vol. of $AgNO_3$ used in ml

(Ii)Titrate with distill water(Blank titration)

S No.	Volume of sample in ml	Initial reading	Final reading	Vol. of $AgNO_3$ used in ml

CALCULATION:

Volume of sample water taken =20ml

Volume of $AgNO_{3\ used}$ $=V_1 - V_2$ml

$$N_1 V_1 \quad = \quad N_2 V_2$$

Water sample AgNO3 soluton

$$N_1 * 20 = N_2 * Z$$

$$N_1 = N_2 * Z/20$$

$$= 1/50\ X*Z/20$$

Strength of Cl^- ion = Z/50*20 *35.5*1000mg/lits

RESULT: The chloride content in the water sample................ppm

$N_1V_1 = N_2V_2$

$N_1 \times 10 = \frac{1}{50} \times 25$

$N_1 = \frac{25}{10 \times \cancel{50}_{2}}$

$S = N.E$

~~$= \frac{1}{50} \times 35.5$~~

$= \frac{1}{20} \times 35.5 = 1.77\ g/l \times 1000$

$= ppm$

7.1

EXPERIMENT NO. 08

Aim: To prepare Urea-formaldehyde and Phenol-formaldehyde resin.

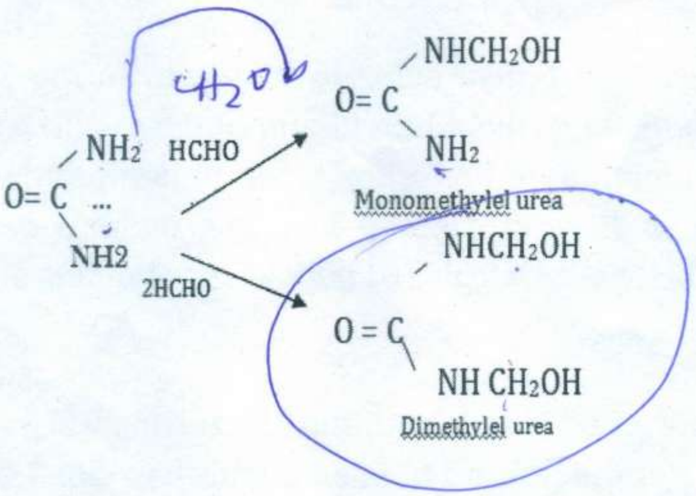
(a)Urea-formaldehyde:

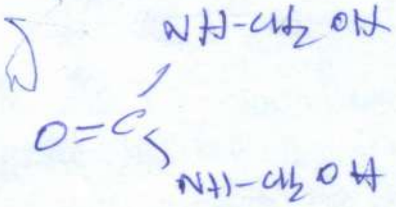
Requirements:

Urea, formaldehyde (40%), beaker, glass rod, measuring cylinder, funnel, filter paper, concentrated H_2S,

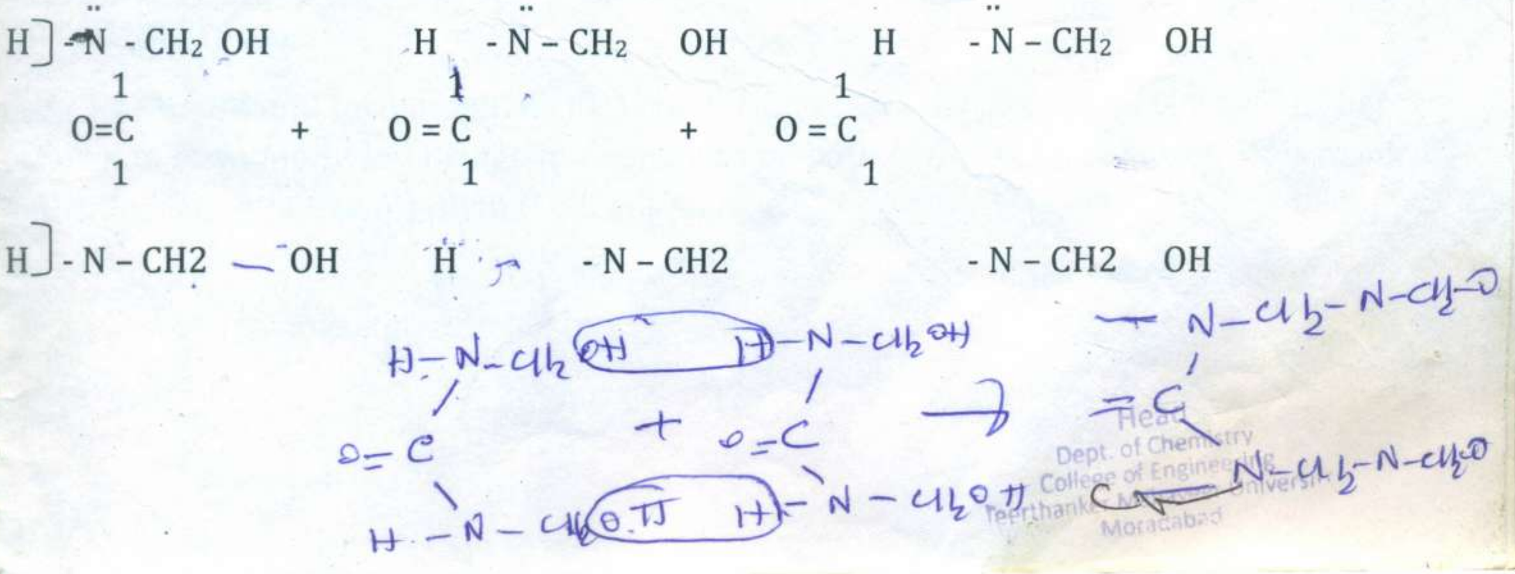
Theory: Urea formaldehyde resin is synthesized by the condensation polymerization reaction of urea with formaldehyde in acidic or basic medium. Reaction takes place in two steps.

Step - I Urea reacts with formaldehyde to form monomethylol urea and dimethylol urea.

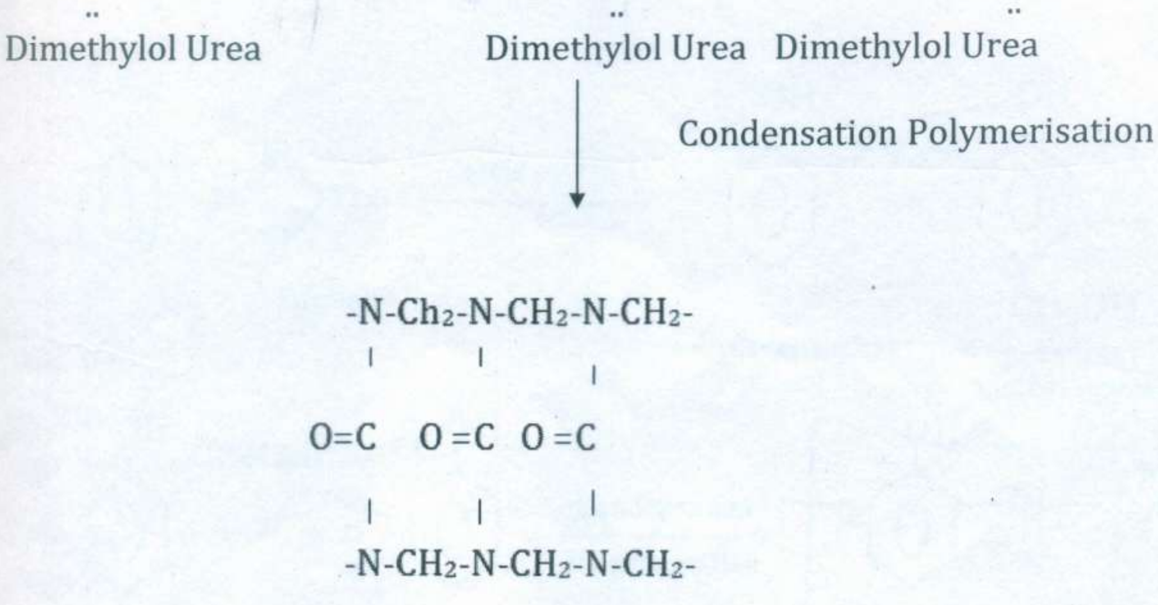




Step - II The mono methylol urea/dimethylol urea on further condensation polymerization formed a three dimensional network polymer called urea formaldehyde resin.



7.2



$$\begin{array}{ccc} \text{-N-Ch}_2\text{-} & \text{N-CH}_2\text{-} & \text{N-CH}_2\text{-} \\ | & | & | \\ \text{O=C} & \text{O=C} & \text{O=C} \\ | & | & | \\ \text{-N-CH}_2\text{-} & \text{N-CH}_2\text{-} & \text{N-CH}_2\text{-} \end{array}$$

Urea formaldehyde resin (cross linked polymer)

PROCEDURE :

Take 2 gm of urea and dissolve it in 40% formaldehyde till solution becomes saturated. To this reaction mixture add a drop of concentrated H_2SO_4 with continuous stirring till white precipitate appears. After completion of reaction, wash the residue with water to remove any acid or base present in it. Dry the precipitate of urea formaldehyde and note the weight to calculate the yield of product.

PRECAUTION:

1. Reaction is vigorous. All addition should be careful and with stirring.
2. Fuming cupboard should be used for preparation.
3. Concentrated H_2SO_4 should be used.
4. Solution of urea in formaldehyde should be saturated.

Result: Yield---------- g ✓

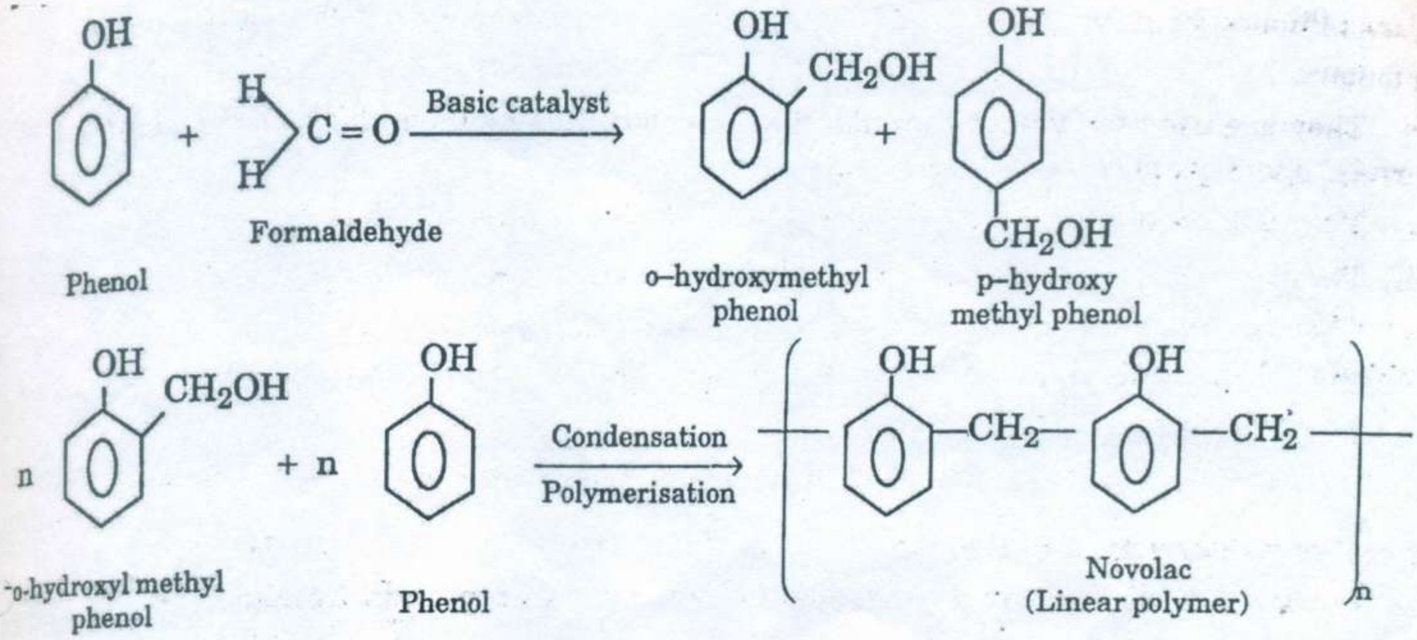
(b) Phenol Formaldehyde

8.1

One Stage Process:

Step – I Phenol and formaldehyde (1:1) or 1:1:25 reacts in the presence of acid or base catalyst to form Methylen bridge in ortho,para or both ortho, para position. Thes result in the formation of linear polymer called(navolack).

8·2

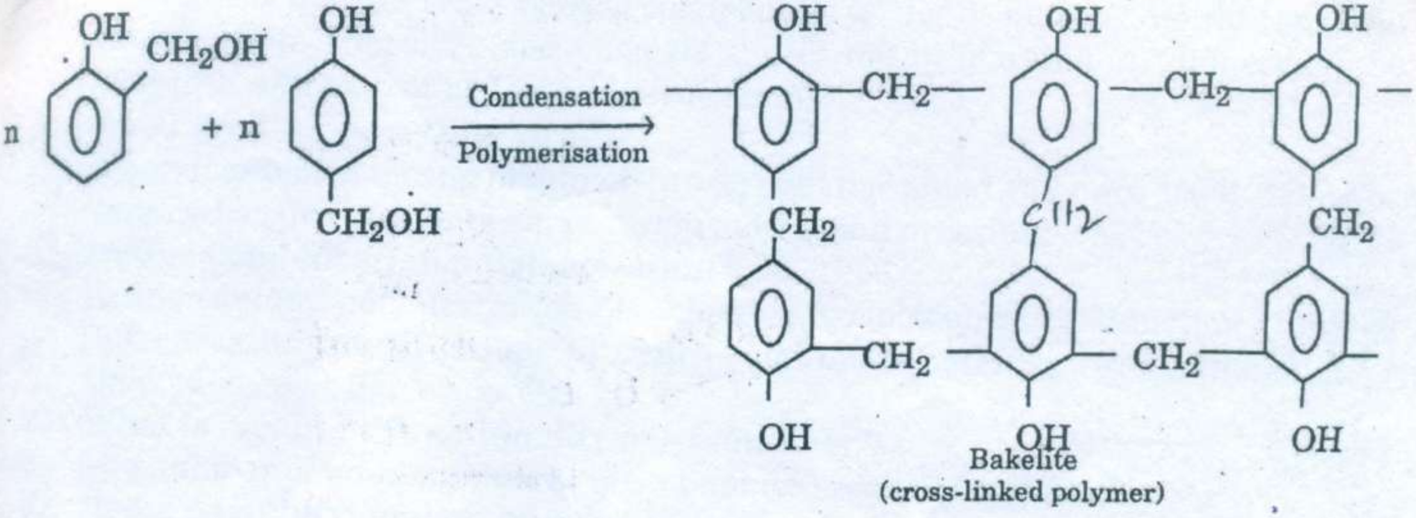


Figure

Two Stage Process:

Step – 2 Novolac resin can undergo further polymerization to yield three dimensional network polymers only when excess of formaldehyde is added. Formaldehyde makes the soluble and fusible mass of novolac into hard and insoluble Bakelite. Formaldehyde attacks at the vacant Para position of phenol.

8.3



Figure

Procedure:

Take 5 ml of glacial acetic acid and 2.5 ml of 40% formaldehyde solution in a beaker. To this add 2 gm of phenol. Add concentrated, HCI to the reaction mixture drop wise with continuous stirring till the pink colored mass appears. Residue obtained is washed with water several times till it is free from acid. Filter and dry the product and note its weight.

Precaution:

1. Reaction is vigorous. All additions should be careful and with stirring.
2. Fuming cupboard should be used for preparation.

Result: Yield ------------ g.

VIVA – VOCE

Q.1. Define a polymer?

Ans. A giant molecule of high molecular mass formed by linking together a large number of small molecules (monomers) by chemical bond.

Q.2. What is meant by degree of polymerization ?

Ans. The numbers of repeating units of monomers in a chain of a polymer.

Q.3. What is the common name of phenol-formaldehyde resin?

Ans. Bakelite is the common name for phenol-formaldehyde resin.

Q.4. Why is Bakelite used in electrical appliances?

Ans. Urea-formaldehyde resin is used for manufacturing of buttons, bottle caps, and containers in adhesives and enamels and in the preparation of dinnerware by mixing it with melamine formaldehyde resin.

Q.6. What are thermosetting resins?

Ans. A polymer which during moulding process get hardened and once set, it cannot be softened again even on the application of heat and pressure.

Q.7. Define condensation polymerization?

Ans. Condensation polymerization is the inter-molecular combination through difference functional groups present in the monomers, with the elimination of small molecules like water etc.

Q.8. What is meant by functionality of a monomer ?

Ans. The number of bonding sites in the monomer is called functionality.

Q.9. Name a trifunctional monomer?

Ans. Phenol

Q.10. What are resins?

Ans. It is the basic polymeric material which is formed by the process of polymerization and hole different constituents in a plastic.

Q.11. What is a monomer?

Ans. Small molecules which combine with each other to form polymer, are called monomers.

Q.12. Define addition polymerization?

Ans. Addition polymerization is a reaction which yields a product which is exact multiple of the original monomeric molecule.

Q.13. What are thermoplastics?

Ans. Polymers which can be softened on heating and gets hardened on cooling are called thermoplastics e.g., PVC.

Q.14. What are homochain polymers?

Ans. Polymers having same species of atoms e.g., carbons in polyethylene chain are called homochain polymers.

Q.15. What are heterochain polymers?

Ans. Polymers whose chain is made up of different atoms is called a heterochain polymer e.g., Nylon-6 whose main chain is composed of C and N atoms.

Q.16. Name the monomers of Bakelite?

Ans. Phenol (C_6H_5OH) and formaldehyde (HCHO)

Q.17. Who invented the first synthetic polymer?

Ans. Wallace Carothers in Feli. 1953 invented Nylon (polyamide)

Q.18. Why plasticizers are used in the manufacture of plastics?

Ans. The plasticizers improve flexibility and plasticity of plastics by decreasing Vander Waal forces of attraction between the chains so that moulding becomes easy.

Q.19. What is Co-polymerization?

Ans. Polymerization of two or more monomer species yield Co-polymer e.g., butadiene and styrene react together to give styrene-butadiene rubber (SBR).

EXPERIMENT NO. 9

AIM:To determine the concentration of unknown sample of Iron Spectrophotometrically.

APPARATUS: Spectrophotometer

CHEMICAL: Stock solution of Fe^{3+}ion, con. HCI, Potasium thiocynate.

THEORY: The calorimetric determination of iron using KCNS as color-developing agent is based on the formation of red colored complex between Fe^{3+} and CNS^-Ions.

$$Fe^{3+} + 3CNS \Longrightarrow [Fe\ (CNS)]^{2+}\ (Red)$$

In the determination of Iron in water sample, a series of standard solution having iron is treated with KCNS to get iron-thiocynate complex. The absorbance of all the standard solution prepared is noted λ_{max} 480nm because Iron-thiocynate complex show maximum absorbance at this wavelength. Now the absorbance for the unknown solution is also determined. A graph is plotted for absorbance (OD) against the concentration of the known solution. From the graph the concentration of unknown solution can be found.

PROCEDURE:

1. Spectrophotometer must initially read zero on transmittance scale.
2. Connect the instrument to the mains and put on the power switch.
3. Adjust the wavelength knob to the required region on scale. Chose either 340-400nm or 400-960nm.
4. Adjust the needle to 100% transmission scale and 100 on OD scale.
5. Open the lid of the cell compartment and insert a cuvette containing a blank solvent(distilled water)
6. Adjust the needle to 100% transmittance or zero optical density.
7. Prepare the stock solution of Fe^{3+}ion by dissolving 2.0gm of ferric ammonium sulphate in 50ml of distilled water and 10ml of con HCI. HCI is added to suppress the hydrolysis of ferric ammonium sulphate.
8. Take the stock solution in burette and prepare 5 solution of different known concentration by diluting the above stock solution in five different flasks.
9. Transfer 1ml of stander stock solution to 5ml measuring and add 10ml thiocynate solution. Dilute the solution to 50ml.

Head
Dept. of Chemistry
College of Engineering
Teerthanker Mahaveer University
Moradabad

10. Repeat the process (step-9) with the solution of other concentration prepared above and also with unknown sample.

11. Keep the solution at rest for five minutes.

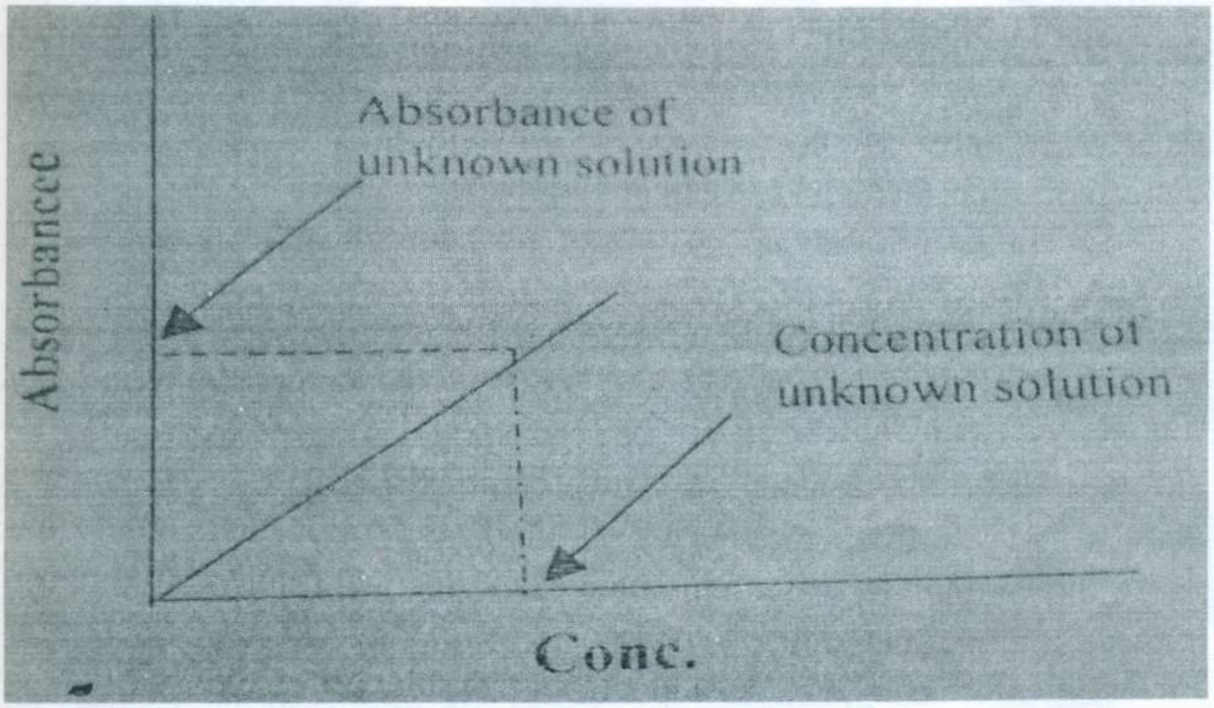
12. Note down the absorbance of all the solution against blank solution spectrometrically at 480nm.

13. Prepare a calibration curve by plotting absorbance against concentration.

14. From the calibration curve, the concentration of the unknown solution corresponding to its absorbance can be found out.

OBSERVATION:

S No	Concentration	absorbance



Result: The amount of Fe (III) in water was found to be = ...g/lit

EXPERIMENT NO. 10

AIM-To determines the viscosity of lubricating oil using Redwood Viscometer (No. 1 and No. 2).

APPARATUS REQUIRED AND REAGENT REQUIRED

Redwood Viscometer No. 1 and No. 2, stop watch, lubricating oil.

THEORY- Viscosity is the property of a liquid or a fluid by which a layer of liquid offers resistance to the flow of another layer. If two layers of a liquid separated by a distance 'd' and moving with a relative velocity difference V then force per unit area (F) required to maintain this velocity difference is given by

F = $\underline{\eta V}$, where η is the coefficient of viscosity.

Description of the Viscometer

It consists of the following parts:

(1) **Oil Cup** :

Oil cup made up of silver plated brass (having 90 ml in height and 46.5 mm in diameter. The bottom of the cup is fitted with an agef ject which opened or closed by a value nod. The level of the oil to be filled is indicated by a painter. The lip of the cup is fitted with a thermometer which indicates the oil temperature.

(2) **Heating bath :**

The oil cup is surrounded by a cylindrical copper bath containing water. A thermometer is fitted in this bath to maintain the temperature.

(3) **Stirrer :**

A stirrer containing four blades is fitted in water bath to maintain uniform temperature of water bath as well as oil cup.

(4) **Spirit Level :**

A Spirit level is provided at the lid of the cup for the vertical leveling of jet.

(5) **Leveling Screw :**

The entire apparatus stands on three legs provided at their bottom with leveling screw.

(6) **Kohlrausch Flask :** It is 50 ml flask for receiving the oil from jet outlet which is kept below the bottom of the cup.

CO-EFFICIENT OF VISCOSITY

Co-efficient of viscosity=(η) is defined as the tangential force required per unit area to maintain unit velocity gradient to maintain unit velocity difference between two parallel planes in the fluid unit distance.

Viscosity Index:

The variation in the viscosity of lubricating oil with temp is usually expressed in terms of viscosity index. When the viscosity index is high. The viscosity of oil changes at a very low rate with change in temp on the other hand, if the viscosity index is low, the viscosity of oil changes rapidly with change in temperature.

C.G.S. System: 1 poise = 1 dyne/sec/cm^2

And 1 centipoise = $\frac{1}{100}$ poise

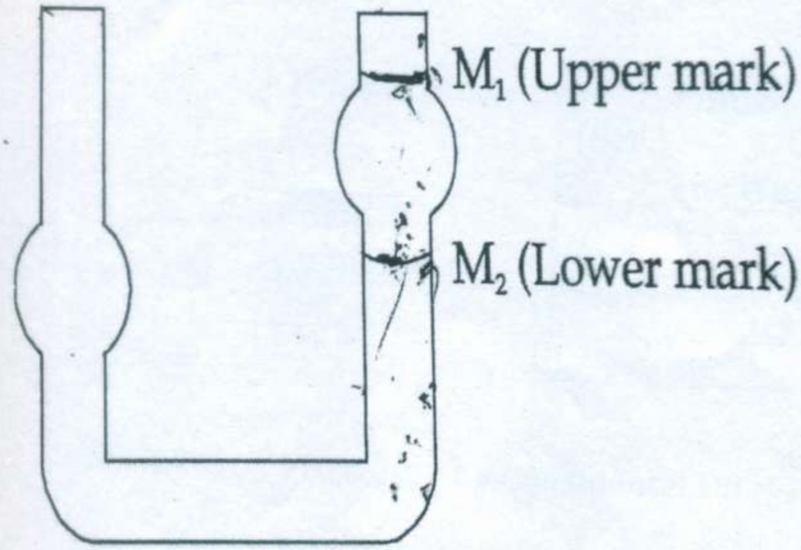
S.I. System: Newton second/m^2 (NS/m^2

1 NS/m^2 = 10 poise

Viscosity is the most important single property of all lubricating oil. If the viscosity of the oil is too low, a liquid oil film cannot be maintained between two moving surfaces and excessive wearing takes place. On the other hand if the viscosity of lubricating oil is too high excessive friction due to sharing of oil would result. In case of heavy pressure and low speed machines less viscous oils will be easily squeezed out hence thick viscous oils are used. Similarly, in light machines less viscous oils are used. In industry viscosity of lubricating oil is determined by Redwood Viscometer. The measure of viscosity of oil is the time in seconds for 50 ml. of oil to flow through standard orifice under a given set of conditions. Two types of Red wood viscometers are used for determining viscosity. The difference between Red Wood Viscometer 1 and 2 is in the Jet length and bore diameter as given below.

	Jet Length	**Bore Diameter**
Redwood Viscometer -1	10 mm	1.62 mm
Redwood Viscometer – 2	50 mm	5.8 mm

Depending upon the viscosity of sample appropriate viscometer in used to determine viscosity.



PROCEDURE

(a) Depending upon the nature of lubricating oil, select the appropriate viscometer, either Redwood viscometer No. 1 or No. 2

(b) With the help of suitable solvent, CCl_4 ether, petroleum sprit or benzene clean the viscometer cup.

(c) Remove any trace of solvent by proper drying.

(d) With the help of screws, provided on the tripod stand, level of the viscometer.

(e) Depending on the temperature of the experiment, fill bath with water or oil. [Use water for determining viscosity at $T \leq 80^0$ C and use oil for determining viscosity at higher temperature]

(f)Seal the orifice properly by means brass ball. Carefully pour the lubricating oil under test into the oil cup up to the tip of indicator.

(g) Below the jet, put the Kohlorausch's flask.

(h) Stirrer the content of bath and cup. Maintain the desire temperature, room temp.

(i) Lift the ball and simultaneously start stop watch when equilibrium temperature has been attain.

(j) For collecting the oil up to the mark in Kohlrausch's flask, note the time(in second) using stop watch

(k) Refill after sealing and repeat the experiment to get nearly reproducible result.

(l) Report the mean value as Redwood 1 and 2 viscosity at room temperature in second.

(m) Repeat the experiment at five temperature and note respective time efflux as described above.

(n) Plot a viscosity temperature curve and from slope find viscosity index.

OBSERVATION

Sr. No.	Temperature °C	Efflux Time (Sec.) For Water	Efflux Time (Sec.) For Oil
1	Room temperature		
2	45°		
3	65°		

RESULT : Viscosity of Experimental Oil = ------------------- Redwood Seconds (R)

Viscosity of water = -------------------------------- Redwood Seconds (R)

VIVA – VOCE

Determination of viscosity of Lubricant by Red Wood viscometer (No. 1 and No. 2).

Q.1. What is viscosity and viscosity index?
Q.2. How does the viscosity of liquid change with temp.?
Q.3. What is principle involved for measurement of viscosity? Name the instrument commonly used to measure viscosity of liquid?
Q.4. How the viscosity indexes (VI) indicate the performance of oil? How the V.R. of oil can be improved?
Q.5. What is oiliness of lubricating oil? How can oiliness of mineral oil be increased?
Q.6. What is meant by the term viscous-static? How can viscous-static lubricant be prepared?
Q.7. What is meant by all weather lubricant?

6.1

EXPERIMENT NO. 11

AIM-To determines flash point fire point of the given lubricating oil by Penskey-Marten's apparatus.

APPARATUS REQUIRED AND REAGENTS REQUIRED

Given lubricating oil, Penskey-Marten's apparatus.

THEORY

Good lubricating oil should not volatilize under the working temperatures. Even if some volatilization takes place, the vapors formed should not form inflammable mixture with air under the conditions of lubrication. From this point of view, the flash point and fire point of lubricating oil are of significance.

The flash point of oil is defined as the minimum temperature at which the oil gives off sufficient vapor that ignite for a moment. When a flame of standard dimension is brought near the surface of the oil. The fire point of an oil is the lowest temperature at which the lubricant gives off sufficient vapor that ignite continuously for at least five seconds, when a flame is brought near it. The fire point of an oil is about 5° to 40° C higher than its flash point. If the liquid is having flash point less than 140° F, they are called flammable liquid and those with flash point about 140° F are called combustible liquid

Lubricating oil selected for a job should have a high flash and fire point which is reasonably above its working temperature. This ensures safety against fire hazards during the storage, transport and use of the lubricating oil. In addition, the flash point of oil is used as a means of identification and also for detection of contamination of the lubricating oils.

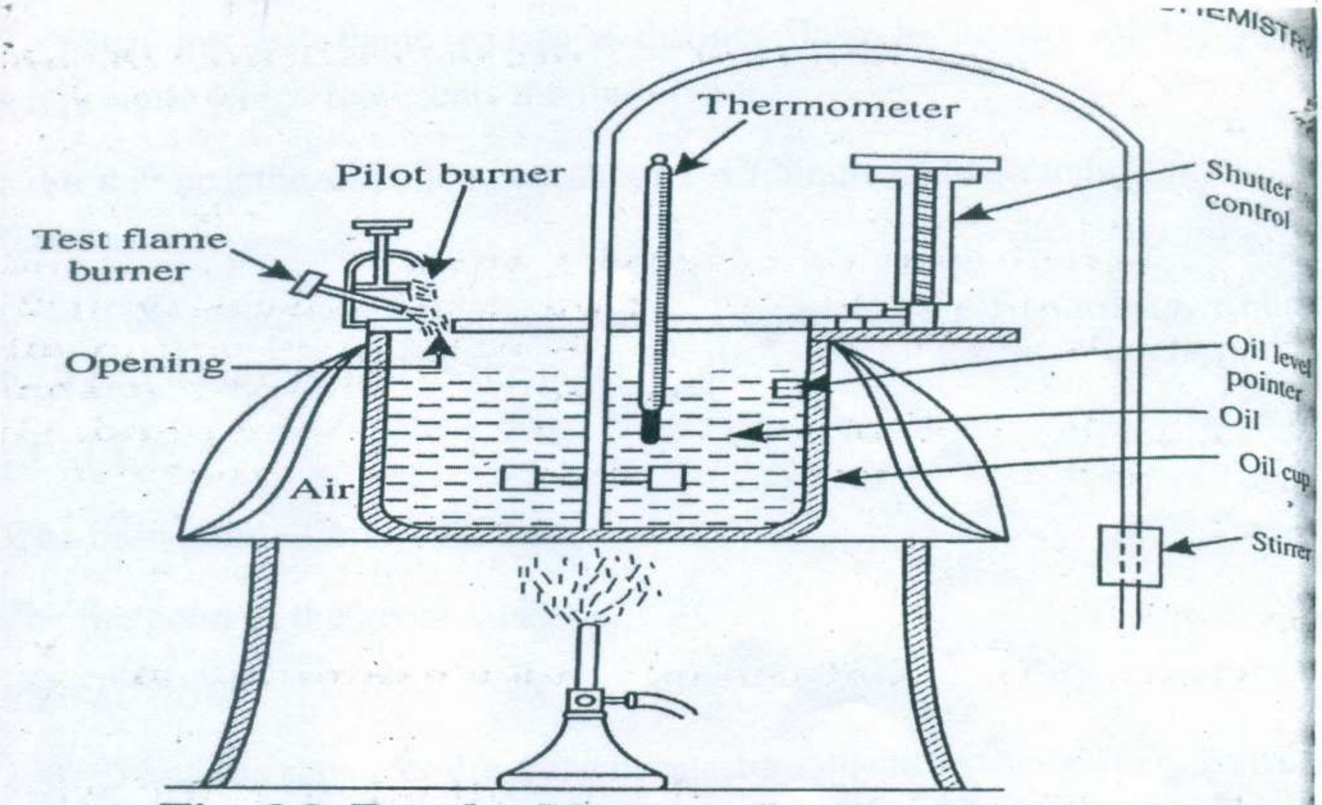


Fig. 6.8. Pensky-Martens-flash point apparatus

PROCEDURE

1. Clean and dry all part of the apparatus

2. Fill the oil cup with the test oil up to the mark.

3. Fixed the lid on the top through which are inserted a thermometer and stirrer. Ensure that the flame exposure device is fixed on top.

4. Light the test flame and adjust it to about 4 mm in diameter.

5. Heat the apparatus by the gas burner so the temperature of the test increase by about five to six degrees per minute while the stirrer is rotated at approximately sixty revolution per minute,

6. At every one degree rise of temperature introduced the test flame for moment into the oil vapor. This is done by operating the Sutter. On moving the knob of shutter, the test flame spring back to its original position and opening is closed.

6.3

7. When the test flame causes a distinct flash in interior of the cup, note temperature which represents the flash point.

8. Further heat the test oil at the rate of 1^0 C/minute and continue applying the test flame as before

9. The temperature at which the vapor of oil give a clear and distinct blue flash for 5 second is recorded as fire point of oil.

RESULT:

The flash point of given oil = ……..^{0}C

The fire point of the given oil is = ……^{0}C

PRECAUTIONS

1. The oil cup should be dried and no moisture should be allowed to remain inside it.
2. The bulb of the thermometer should dip inside the oil sample.
3. Stirring of the oil should be stopped at the time of the application of test flame.
4. Breathing over the surface of oil should be avoided.
5. A bluish halo sometimes found around the test flame should not be confused with the flash.

RESULT

(i) The flash point of given oil sample = t_1° C

(ii) The fire point of the given oil sample = t_2° C

VIVA-VOCE

1. What are lubricants?
2. Define flash point and fire point.
3. What is meant by flash point "closed" and flash point "open" ? Which one is reproducible and why?
4. What is the significance of flash point and fire point measurement?

5. Mention the contaminants which can increase or decrease the flash of lubricating oil.

Description of the Apparatus:

It consists of the following parts.

1. **Oil Cup**: It consists of brass having diameter 5 cm and depth 5.5 cm fitted with a lid.

   The lid is provided with opening holes of standard size. A thermometer is introduced into one hole. Other holes are used to passing air and having stirrer. There is a mark inside the cup at the top to fill the lubricating oil.

2. **Shutter:** The shutter provided at the top of the cup has a lever mechanism. It may be Opened or closed with the help of handle known as shutter controller. The holes on lid are opened for bringing the flame over the oil surface with the help of a shutter.

3. **Air Bath** : An oil cup is placed in air bath and heated by electricity.

4. **Pilor Burner** : Pilot is always lighted. To support the burning. The burning of test flame burner, as the test flame. Is introduced into the oil surface through the hole it gets extinguished but when the test flame is returned to its original position it is automatically lighted by the pilot burner.

5. **Flame Test Burner** :It is burner by which the test flame is introduced into cup Containing lubricating oil.

Experiment No-12

B. CALAROFIC VALUE OF A FUEL BY BOMB CALORIMETER

Bomb Calorimeter provides an accurate method for determination of heat of combustion, calorific value of solid and liquid fuels. [IS 1359-1995, BS 1016 Part 5 1967, IP 12/63 T]

A known amount of the sample is burnt in a sealed chamber, called "Bomb". The sample is ignited electronically. As sample burns, heat is produced. The rise in temperature is determined. Since barring loss of heat, amount of heat produced must be equal to the amount of heat absorbed by the calorimeter assembly. Hence the amount of heat produced can be calculated by the temperature rise.

If Mass of sample = M

Water equivalent of calorimeter assembly = W calories/oC

Rise in temperature = ΔT

$$Calorific\ Value\ or\ Heat\ of\ Combustion = H = \frac{W\ \Delta T}{M}$$

Gross calorific valur or Higher heating value at constant volume, $C_{G:}$ It is the quantity of heat liberated by combustion of unit quality of fuel. Water vapour the original material and final production of combustion being at a temperature of 25°Cand the water obtained from the fuel being in the liquid state'

Gross calorific value at constant pressure is similar to the former expression except that the combustion takes place at constant pressure.

In laboratory determination, solid and liquid fuels are burnt at constant volume and gaseous fuel in constant pressure. The difference in the two methods is small. For coal CV at constant pressure exceeds the CV at constant volume by 5.5kcl/kg.

Net calorific value (Lower heating value) at constantvolume , C_N is the quantity of heatevolved when unit quantity of fuel is burnt at constant volume in oxygen saturated water vaour, the original material and final products of combustion being at a reference temperature of 25°C and the water obtained from the fuel being in the vapour state.

Considering Latent heat of condensation of water vapour at 25°C is 583.5kcal/kg, approximate relation between HHV and LHV for coal is

$$C_N = C_G - 52.5H,\ \text{where His \% hydrogen, } C_N \text{ and } C_G \text{ in kcal/kg}$$

Energy/Water Equivalent of a calorimeter is the heat required to effect 1°C rise in temperature of the entire calorimeter system (Calorimeter vessel containing a specified weight of water, calorimeter, bomb charge oxygen, firing wire and water, in calorimeter and stirrer)

Cooling Corrections: Cooling correction includes the heat interchange between the vessel a the jacket, due to conduction, convection and radiation and the heats of stirring and evaporati This can be minimized by having

a) The temperature of the water in the vessel below that of the water in the jacket at the ti of firing and within ± 0.5°C,of it at the end of the chief period
b) Low heat of stirring
c) Low loss by evaporation.

Cooling Correction is predicted by Renault-Pfaundler formula or the Dikinson Formula[**In th present experiment we will assume it to be 0.01oC]**

Sulphur Correction: This takes into account the extra heat released in the bomb where, und pressure and in the presence of oxides of nitrogen and water, the sulphur in the fuel is convert into sulphuric acid. The correction is the difference between the heat released due to this and tha due to the conversion of the sulphur into gaseous sulphur dioxide. It amounts to 3.6 calories mL of 0.1N Sulphuric acid formed.

Nitrogen Correction : The nitrogen correction takes into account of the fact the pa the nitrogen of the fuel and of that in the air originally present in the bomb is converted into acid in the bomb. The correction is the heat released due to the reaction, which does not occur the atmospheric pressure, it amounts to 1.43 calories per mL of 0.1N nitric acid formed.

APPARATUS:

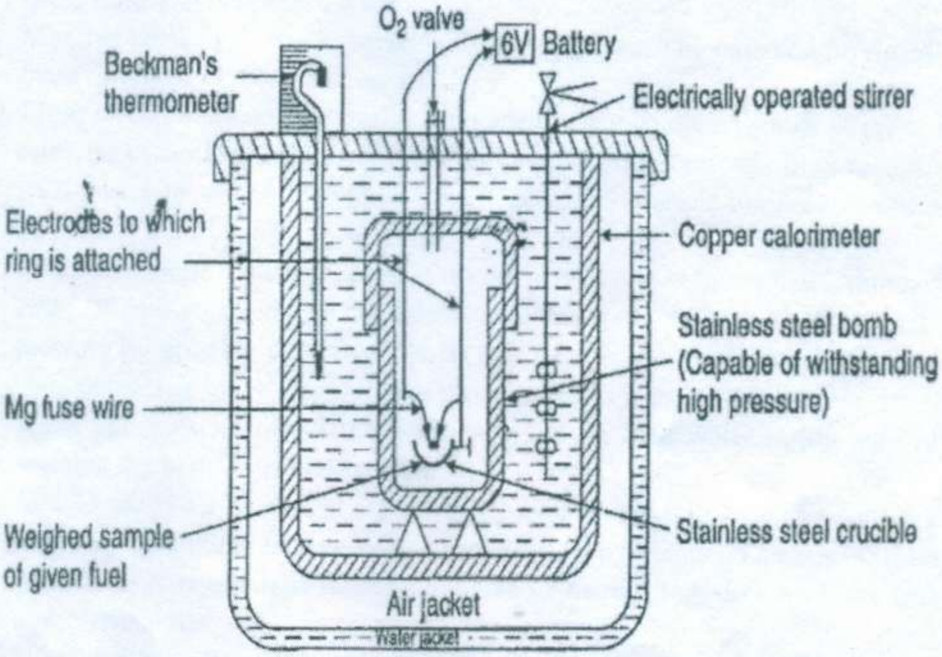
The steel bomb is tested as per requirement of the Institute of petroleum (IP 12/63 T)It certified by hydraulic testing under a pressure of 300 atmospheres, and maintained for a period of ten minutes without sign of leakage.

Calorimeter vessel: It is made of copper and is chrominum plated or SS. It includes a Bomb Support that ensures proper positioning of Bomb in the vessel.

Water Jacket: The outer container of jacket is made of copper and is chrominum plate inside and outside or SS. The top is closed by an ebonite cover. A terminal block is fitted to t top of the container so that connection can be made from the firing control

Offset Stirrer: The stirring mechanism supplied gives sufficient turbulence for effective stirring whilst no heat is imparted to the calorimeter water. It consists of an impeller stirrer driven a constant speed of approximately 800 rpm. The offset arrangement where by the motor drives the impeller via a belt precludes any possibility of heat transfer between motor and calorimete contents.

Electronic firing unit: The firing a calorimeter bomb AC supply main 230 Volts. It consists of two parts - Electrical Box and Electronics Box for thermometer reading. The Electric is provided with socket for stirrer and the terminals for the fuse wire. The unit has provision for test firing wire resistance or open circuit. A LEFT HAND SIDE BULB INDICATES THE CONTINUITY WHILE RIGHT HAND SIDE BUB INDICATED THE OPEN CIRCUIT (Firing).



PROCEDURE:

1. Take 1 gm of coal -20 mesh(210 micron), preferably compressed into a cylindrical pellet taking precaution the pellet ins not hard, which may cause spalling and ejection of fragments during heating.
2. Stretch a piece of the firing wire across the electrodes within the Bomb
3. Tie 15 cm length of sewing cotton around the wire;place crucible in position and arrange the loose ends of the thread so that they are in contact with the material [the same amount of thread in each determination]
4. Introduce into the body of the Bomb two ml of distilled water. Reassemble the bomb screw avoiding excessive pressure.
5. Charge bomb slowly with oxygen to a pressure of 25 atm.without displacing its original air content. [If rapid, the sample may be blown out of the cup]
6. Close the valve effectively, using as little pressure as possible and detach the bomb from the oxygen supply

7. Weigh into the calorimeter vessel a quantity of water sufficient to submerge the cover of the bomb to a depth of at least two cm leaving the terminals projecting. [Use same weight of water in all tests.]
8. Transfer the calorimeter vessel to the water jacket, lower the bomb carefully into the calorimeter vessel. Adjust the stirrer, place the thermometer and covers in position and start the stirring mechanism, which must be kept in continuous operation at a constant speed during the experiment.
9. Note temperature every 30 seconds for five minutes. The temperature rise should not be more than 0.00172°C. per minute.
10. Close circuit momentarily to fire charge and continue observations of the temperature at intervals of similar duration in this Chief period .which extends from the instant of firing until time after which the temperature is constant. [Jacket and room temperature should be close to calorimeter temperature]
11. Continue to note the temperature in the AFTER Period at interval of 1 minute.
12. After 30 minutes remove the bomb, allowing the acid mist to settle and release the pressure by opening the valve. Verify that there is no sooty deposit, to ensure complete combustion has taken place. If soot is there, reject the experiment.
13. Wash out the contents of the bomb with hot distilled water into a hard glass beaker washing the bomb cap, and crucible.
14. Add 25 ml of 0.1 N sodium carbonate solution .and boil to 10 ml to convert any metallic sulphates or nitrates to less soluble carbonate or hydroxide. **The consumption of alkali carbonate is equivalent to the sulphates or nitrate together with the free-sulphuric and nitric acids.**
15. Filter, wash to make upto 100ml/50 ml. Determine total acidity by titrating with 0.1N hydrochloric acid using methyl orange as indicator.[the titre representing excess alkali of the quantity of sodium carbonate solution added to the washings]

OBSERVATIONS:

Particle size of coal : 210 micron
Hydrogen % = 4%
Sulphur % = 0.7%
Amount of coal: g
Weight of firing wire = mg
Weight of fuse wire left after the experiment= mg
Weight of fuse wire burnt= mg [fw]
Weight of thread= mg [th]
Room Temperature =
Temperature of Calorimeter jacket =
Water Equivalent of the Calorimeter = [W] 5334.89 cal/g
Cooling Correction = 0.01°C [r]
Nitric Acid Correction: 1.43 calories per mL of 0.1N nitric acid formed
Sulphuric Acid Correction: 3.6 calories per mL of 0.1 N sulphuric acid formed
Firing wire correction : Nichrome wire = 0.335 cal/mg,
Platinum wire = 0.100 cal/mg
Thread Correction = 4.2cal/mg
Rise in temperature [apparent or uncorrected] =
Volume of 0.1 N sodium carbonate solution added to the treated washing = 25 ml [x]
Volume of 0.1 N HCl used for neutralization= [y]

% Sulphur in sample= Amount of sample=
Amount of Sulphur in sample= [S]

$$cc\ of\ \frac{N}{10} H_2SO_4 = \frac{S}{0.0016} = \qquad [A]$$

$$cc\ of\ \frac{N}{10}\ HNO_3 = x - y - \frac{S}{0.0016} = \qquad [B]$$

$$Gross\ CV\ in\ cal/g$$
$$= \frac{W\ x\ [\Delta t + r] - [A\ x\ 3.6 + B\ x\ 1.43 + fw\ x\ 0.335 + th\ x\ 4.2}{mass\ of\ coal}$$

$$Net\ CV = Gross\ CV \frac{cal}{g} - \left[\frac{H}{100}\ x\ 9\ x\ 600\right]$$

Head
Dept. of Chemistry
College of Engineering
Teerthanker Mahaveer University
Moradabad

Sl No	Time, minutes	Temperature, °C	Observations
PRELIMINARY PERIOD			
	.5		
	1		
	1.5		
	2		
	2.5		
	3		
	3.5		
	4		
	4.5		
	5		
CHIEF PERIOD			
	.5		
	1		
	1.5		
	2		
	2.5		
	3		
	3.5		
	4		
	4.5		
	5		
AFTER PERIOD			
	1		
	2		
	3		
	4		
	5		
	6		

EXPERIMENT NO. - 13

AIM: Determination of calorific value of gaseous fuel by Junker's gas calorimeter.

DISCUSSION: The Junker's calorimeter is of the flow type in which the calorific value is ascertained from the rise in temperature imparted to a measured quantity of flowing water by burning a measured value of gas. The heat of combustion of a gaseous fuel is required in heat balance and thermal efficiency calculations of gas engines and gas fired furnaces.

Heat of combustion=Heat gained

C.V. = Mt

Where C is the calorific value, V the volume of gas burnt, M the wt. of water and t, the rise of temperature.

APPARATUS: The basic apparatus consists of:

A. Junker's calorimeter body
B. Gas meter for measurement of gas consumed in cubic feet during the test
C. Balance governer for controlling pressure of gas
D. Graduated glass measurements for water collected during test and for condensed products of combustion
E. Thermometers for temperature measurement of inlet and outlet water and products of combustion
F. Flexible plastic tubing for connections.

Junker's gas calorimeter has a vertical cylindrical combustion chamber surrounded by an annular water heating space and heats inter change coils fitted inside the outer flues. The whole is enclosed in an outer jacket heavily chromium plated on the outside. The annular water heating space is connected at its lower and to an internal water way which in turn is connected to a constant heat reservoir through an adjustable value moving on a graduated arc. The upper end of this space leads to allowing water into a hot water mixing box fitted with baffle plates. From the box the heated water goes out into the sink or a measuring cylinder through an overflow outlet funnel and a swinging arm attached to it. The constant head reservoir is provided with an inlet pipe which at the bottom is connected to an outlet funnel fixed in the overflow discharge cup. To discharge the outlet water from the apparatus into the sink. the swinging arm can be turned to bring it above the discharge cup. There are two opening provided at the top of the apparatus for two thermometers to record the inlet and outlet water temperatures. A gas burner clamped at the bottom can be easily taken out of the combustion chamber or pushed wall up into it. The products of combustion having given up their heat to the flowing water in the annular space and in the heat inter changer coils scape through the waste gas products outlet near the base and any condensate formed is run out into a graduated cylinder through a small outlet tube at the

√ 1

bottom. There is also a drain cock provided at the base to the drain the whole of water from the apparatus after the experiment. There is gasometer provided to measure the gas and a balance governor to maintain it at a constant pressure. The waste gas outlet is provided with a thermometer socket and a throttle value to control the admission of air or the draught.

SETTING UP OF THE APPARATUS:

Place the calorimeter body, preferably near a sink so that waste water can be disposal conveniently. Place the gasometer near calorimeter for ease of reading. Using suitable lengths of the plastic tubings supplied, make following connections:

A. As service to inlet of balance governor
B. Outlet of balance governor to inlet of meter (centre of back0
C. Outlet of meter (top of back to burner this tube connections should be long enough to allow burner to be removed from its working position in calorimeter body.

Connect the water supply to the inlet pipe on calorimeter body. Attach a flexible tube to the outlet pipe in order to discharge waste water into sink. (Soft and filtered water to be used to avoid scale format in the calorimeter. Variations pressure cause trouble hence the water supply should be from small tank mounted above the branch).

Fit the two long thermometers into the sockets at the top of the calorimeter with rubber bungs attached and see that the bulbs are low enough but not touching any metal parts. Swing the swing arm into the discharge cup. Place the short thermometer in outlet. Place the baffle with butterfly value over outlet. Remove the encased thermometer and fill the meter until the water level is to water line on sight glass. Replace thermometer and adjust the meter level by means of leveling screw, using spirit level on glass plate on top of the meter. Run out the water from the meter by drain cock until the lower menisous coinsides with the line on sight glass. Remove the plug on side of governor tank. Pour water into tank until it overflow from socket on side. Replace the plug. The calorimeter is now ready for making the test.

PROCEDURE:

Turn on water by opening the control cock and see that there is a small but continuous overflow. Remove the burner from calorimeter and turn on the gas. Allow the gas to blow oil for two or three revolutions of the meter to eliminate air from meter and connections and to saturate the water in meter and governor. Light the burner and adjust air slide to get acquient and non luminous flame. Shut oil the gas at the burner and watch the needle on the meter for any leakage. Adjust gas rate by cook on burner, to approximately 6 to 7 cm. ft. (about 0.2 cu.meter) per hour for gas of about 4500 kgm. cal per cu meter. Adjust water central control to give water flow through calorimeter of about one litre per minute. These adjustments should normally give a temperature difference of 12-15+C between inlet and outlet water. Insert the burner well up into the combustion chamber and observe, with a mirror, the condition of flame. Excess air can be controlled by means of the throttle value at the gas exit end. Adjust the passage of air in the combustion chamber, with the help of the throttle valve, so as to record the maximum temperature on the water outlet thermometer. The temperature of air, gas, inlet water and the flue gas should be about the same. Allow the calorimeter to run for 3-40 mts. To attain a steady temperature condition. Place the large glass measure conveniently to collect water from the swing water outlet tube. Place a small glass measure to collect any condenses water from the products of combustion. As the meter pointer passes chosen mark turns on outlet water into glass measure. Read inlet and outlet water temperatures. As the meter pointer passes the chosen mark at the completion of one revolution divert water to waste. Read the barometer, manometer, wetter temperature and volume of heated water collected. From this data the calorific value is calculated.

ADDITIONAL PRECAUTIONS:

A. Always turn on water first before starting up.
B. Always turn off gas first before shutting down.
C. Always keep water control cock partly open.
D. Check water line of meter before starting up

Draw a neat and simple diagram showing the balance governor, gasometer and Junker's and Junker's calorimeter and as connected for the experiment.

Calculations:

$$\text{Calorific value of gaseous fuel} = \frac{\text{Heat produced on combustion}}{\text{Volume of fuel burnt at } 15.5^{0}\text{C and 1 atm.}}$$

Since the date supplied are at room temperature and pressure the volume of fuel burnt at 15.5^{0}C and 1atm be calculated by the application of usual gas equation $\frac{P1\ V_1}{T_1} = \frac{P_2 V_2}{T_2}$

$$\frac{(76-1.3)V_1}{273+15.5} = \frac{(B + \quad h/13.6 - q)\ V_2}{(T + 273)}$$

Where V_1 = Volume at gas burnt in std. Condition (at 15.5^{0}C and 1 atm.)

B = Barometric pressure (cm)

H = Manometric pressure (cm)

V_2 = Volume of fuel burnt in laboratory condition t^{0} C

q = Aqueous tension at (thus V can be calculated)

Approximate G.C.V. = a x b / V Kcal / ft^3

Where, a = kg of water collected

b = rise of temp. (Outlet – inlet)

Head
Dept. of Chemistry
College of Engineering
Teerthanker Mahaveer University
Moradabad

EFFLUENT GAS CORRECTION:

The gaseous combustion product (effluent gas) takes away a part of sensible heat and thus being unutilized in heating the calorimetric system. This sensible heat constitute heat constitute the effluent gas correction. Effluent gas correction:

$F = 6.4\ (t_F - t)$ Kcal/m^3

t_F = temperature of effluent gas

t =temperature of fuel gas

This F has to be added in the approximate gross calorific value (in proper unit)

Approx. G.C.V. = a* b/ V Kcal/ft^3

= a * b /V * 35.31 Kcal m^3

Akcal/m^3

Corrected G.C.V. = (A + F) = Bkcal/m^3 (i)

B / 35.31 =C Kcal/ft^3 (ii)

C * 1000 = D ~~Kcal/ft^3~~ cal/ft (iii)

D/252 =E BTU/ft^3 (iv)

(1 BTU = 252) cal
1BTU = 0.252 Kcal

Net C.V. =heat of condensation per unit volume = (C-D * 600/V * 1000) Kcal/ft^3
(Then convert into other three units).

****

D x 600 / V x 1000

EXPERIMENT NO.-14

AIM: To report the volumetric analysis of flue gas in terms of oxygen and CO_2 & Co using orsat apparatus.

THEORY:

Orsat apparatus consist essentially of water jacketed measuring burette connected by means of glass manifold to three absorption bulbs usually called pipette. The burette has a capacity of 100 ml. from zero at the bottom to 100 mark on the capillary tube at the top. The lower end of the burette is connected by rubber tubing with the pressure bottle.

The pipettes are partly filled with glass tubes to increase the liquid surface in contact with gas. Care should be taken that the pipette is not exposed to the air, as the solution.

PIPETTE:

$$2\,C_6H_3\,(OH)_3 + 7O_2 \longrightarrow 2H\,COOH + 2\,CH_3COOH + 6\,CO_2$$

$$2\,H\,COOH + 2KOH \longrightarrow 2H\,COOK + 2H_2O$$

$$2\,CH_3COOH + 2KOH \longrightarrow 2CH_3COOK + 2H_2O$$

$$6\,CO_2 + 6KOH \longrightarrow 6KOHCO_3$$

SUMMING UP:

$$2C_6H_3\,(OH)_3 + 7O_2 + 10KOH \longrightarrow 6KOHCO_3 + 2HCOOK\ \ 2CH_3COOK + 4H_2O$$

PROCEDURE:

Before proceeding with an analysis, test the apparatus to check that it is air tight. This is done by raising the pressure bottle and filling the burette with water and close with a stopcock No.4 to air, then lowering the bottle. Any change in the water level in the burette will show whether there is a leakage. Finally, again raise water in the burette to the mark on the upper capillary tube and close the apparatus.

Take the sample by opening stopcock No.4 and lowering the pressure bottle and allow the water to fall in the burette until it's slightly below the zero of the scale, then close stopcock No.4 to its first position. Now lift the pressure bottle sufficient to raise its water level just above that the water in the burette and continuously loosen then plug of the 4th stopcock from its seating. Allow the water to reach zero of the burette, then immediately replace the stopcock plug. The water level will then be exactly the same in both pressure bottle and burette and the latter will contain 100ml of the gas at atmospheric pressure. Now pass the air into the pipette by raising the bottle and opening the stopcock of the pipette. Take it back into the burette and take the reading at atmospheric pressure. Send the gas again into the pipette and repeat till concordant reading are obtained.

Head
Dept. of Chemistry
College of Engineering
Teerthanker Mahaveer University
Moradabad

Observation Table

For CO_2

S. No.	Initial Reading	Final Reading	Different	Concordant
				4
1	[illegible]	[illegible]	4	
2	[illegible]	[illegible]	4	
3				

For O_2

S. No.	Initial Reading	Final Reading	Different	Concordant
1	[illegible]	[illegible]	16	[illegible]
2	[illegible]	[illegible]	16	
3				

For CO

S. No.	Initial Reading(ml)	Final Reading(ml)	Different(ml)	Concordant
1	80	79.2	0.8	0.8
2	80	79.2	0.8	
3				

Calculation

Result-

Precaution-

****

Experiment No. 15

M: To determine moisture, volatile, fixed carbon and ash contents in a given coal sample by proximate analysis.

PRATUS: Electrical or Analytical balance, electric oven, desiccators, silica crucible with lid, tongs.

TERIALS REQUIRED: Given coal sample (powdered).

EORY: proximate analysis of coal includes analysis of the following:

1) Moisture content – Owing to its nature and origin, coal is always associated with moisture. When wet coal is exposed to atmosphere, the external moisture evaporates but the apparently dry coal still contains some moisture is referred to as air dried moisture.

2 Volatile matter- The volatile matter in coal consists of complex mixture of gaseous and liquid products resulting from the thermal decomposition of the coal. Volatile matter does not include moisture of coal. It consists mainly of combustible gases such as H_2, CO, CH_4 and other hydrocarbons.

3) Ash- coal contains inorganic mineral substances which are converted into ash by chemical reactions during the combustion of coal. Ash usually consists of silica, alumina, iron oxide and small quantities of lime, magnesia etc.

Ash can be classified as intrinsic and extrinsic ash. The mineral matter originally present in vegetable matter from which the coal was formed is called intrinsic ash. It consists of oxides of Na, K, Mg, Ca and silica. On the other hand, the mineral matter, like clay, gypsum, dirt which gets mixed up during mining and handling of coal consists of the extrinsic ash which remains as a residue after the combustion. The extrinsic ash may consist of anhydrous $CaSO_4$, $CaCO_3$, FeO_3 etc.

The minerals responsible for the formation of intrinsic ash cannot be remover by any mechanical means. Fortunately this mineral matter content is low (about 2-3%) where as the extrinsic ash content is relatively higher.

4) Fixed carbon- Fixed carbon content increases from low ranking coals such as lignite to high ranking coals such as anthracite. Higher the percentage of fixed carbon, greater is its calorific value and better is the quality of coal. This represents the quantity of carbon that can be burnt by a primary current of air.

PROCEDURE:

(I)Determination of moisture (air dried):

. Dry the coal (powdered) in air or in the moisture oven at 10 to 15^0C above room temperature.

1. Weigh the silica crucible in an analytical or electrical balance accurately.

Transfer l g of air dried coal sample to the crucible and weigh it.

Keep the crucible in an electric oven maintained at 105 to 110⁰C for an hour.

5. Remove the crucible with a pair of tings and keep it in a desiccators for cooling.

6. After it is cooled weigh it. Note down the observations.

(II) Determination of volatile matter.

Take the dried sample of the moisture free coal (obtained from steeple) in a weighed and covered platinum crucible. The cover should have a vent for escape of volatile matter.

Place the crucible in a muffle furnace maintained at 950+200⁰C and heat it exactly for 7 min.

3. Remove the crucible with the help of tongs very carefully and bring down its temperature rapidly by placing it on a cold iron plate.

4. Place the crucible in a desiccators so that it attains room temperature.

5. Weigh it and note down the readings.

(III) Determination of ash content.

1. The residual coal (after step II) is kept in a weighed silica or platinum crucible.
2. The crucible is heated in a muffle furnace maintained at 725 + 25⁰C for nearly half an hour, without cover. Occasionally stir the residue with platinum wire.
3. The crucible is then cooled in a desiccators and weighed.

(IV) Determination of fixed carbon.

The fixed carbon is calculated as:

% Fixed Carbon = [100-(%moisture + % ash + volatile matter)]

OBSERVATIONS AND CALCULATIONS:

(i) Moisture

Weight of empty crucible = a g

Weight of crucible + coal sample = b g

Weight of crucible + coal sample after heating = c g

$$\% \text{ moisture} = \frac{(b-c)}{(b-a)} \times 100$$

(ii) Volatile matter

Weight of empty crucible = a g

Weight of crucible + sample (moisture free) = c g

Head
Dept. of Chemistry
College of Engineering
Teerthanker Mahaveer University
Moradabad

Weight of crucible after the removal of volatile matter = d g

% volatile matter on oven dried basis = $\frac{(c-d)}{(c-a)} \times 100$

% volatile matter (on air dried basis) = $\frac{(c-d)}{(c-a)} \times 100$

(iii) Ash content

Weight of empty crucible = a g

Weight of crucible + coal sample = b g

Weight of crucible + ash = e g

% ash = $\frac{(e-a)}{(b-a)} \times 100$

IV) Fixed carbon

% Fixed Carbon = [100- (%moisture + %ash + volatile matter)]

RESULT: The proximate analysis of coal is as follows:

Moisture = ... %, Volatile matter = ... % Ash = ... % and fixed carbon = ... %.

PRECAUTIONS:

1. After air drying the coal, the moisture analysis should be done speedily so as to prevent moisture change.
2. In the determination of volatile matter, the temperature should be raised at a slow rate.
3. If black particles appear during the determination of ash content due to the present of unhurt carbon continue ignition for another 15 min.