Dronacharya Group of Institutions,
Greater Noida

CHEMISTRY PRACTICAL MANUAL

LIST OF EXPERIMENTS

1. Determination of alkalinity in the given water sample.

2. Determination of temporary and permanent hardness in water sample using EDTA as standard solution.

3. Determination of available chlorine in bleaching powder.

4. Determination of chloride content in water sample by Mohr’s method.

5. To determine the iron content in the given sample by redox titration using external indicator.

6. pH-metric titration.

7. Viscosity of an addition polymer like polyester by viscometer.

8. Determination of iron concentration in sample of water by colorimetric method. The method involves the use of KSCN as a colour developing agent and the measurements are carried out at λ_{max} 480nm.

9. Element detection and functional group identification in organic compounds.

10. Preparation of Bakelite and urea formaldehyde resin.

SUBJECT CODE

NAS102/NAS202: ENGINEERING CHEMISTRY (THEORY)

NAS152/NAS-252: ENGINEERING CHEMISTRY (PARACTICALS)

To Write in Practical File

Left Hand Side with Pencil

Date

Aim:

Observation Table:

S. No	Volume in the Titration Flask (ml)	Burette Reading (ml)		Volume of Solution used from burette (ml)
		Initial	Final	
1				
2				
3				

(This table is only for volumetric titrations)

Graph if required:

Calculations:

Result:

Right Hand Side with Pen (blue only)

Date

Object:

Apparatus:

Chemicals:

Theory:

Procedure:

Precautions:

Result:

EXPERIMENT NO:- 1

OBJECT: To determine the alkalinity of given water sample by neutralization titration.

APPARATUS: Beaker, Pipette, Burette, Conical Flask

CHEMICALS: Alkalinity Sample, 0.1N HCl, Methyl Orange and Phenolphthalein.

THEORY: The alkalinity in water is due to the presence of hydroxyl ion (OH^-), carbonate ion (CO_3^{2-}) and bicarbonate ion (HCO_3^-) present in the given sample of water. These can be estimated separately by titration against standard acid, using phenolphthalein and methyl orange indicators. The chemical reaction involved can be shown by the equations given below:

i) $OH^- + H^+ \rightarrow H_2O$
ii) $CO_3^{2-} + H^+ \rightarrow HCO_3^-$
iii) $HCO_3^- + H^+ \rightarrow H_2O + CO_2$

PROCEDURE: Take 25ml of the sample solution in conical flask with help of a pipette. Add 2-3 drop of phenolphthalein indicator. Titrate this sample against HCl solution until the pink colour caused by phenolphthalein just disappears. Note down this reading as phenolphthalein end point. Now add 2-3 drops of methyl orange indicator in the same solution. Continue the titration until yellow colour change into pinkish orange. Note the volume of acid used. This is methyl orange end point.

OBSERVATION: Normality of HCl solution = N/10

1. Indicator used : Phenolphthalein
   End point: Pink→ Colorless
2. Indicator used : Methyl Orange
   End point: Yellow→ Pinkish orange.

OBSERVATION TABLE:

For Phenolphthalein Indicator

S. No	Volume of alkalinity sample in the Titration Flask (ml)	Burette Reading (ml)		Volume of N/10HCl Solution used from burette (ml)
		Initial	Final	
1				
2				
3				

Volume of N/10HCl Solution used from burette (ml) to neutralize25 ml of alkalinity sample =V_1ml

For Methyl Orange Indicator

S. No	Volume of alkalinity sample in the Titration Flask (ml)	Burette Reading (ml)		Volume of N/10HCl Solution used from burette (ml)
		Initial	Final	
1				
2				
3				

Volume of N/10HCl Solution used from burette (ml) to neutralize10 ml of alkalinity sample = V_2ml
(This is total volume starting with pink colour to colourless and then yellow to pinkish orange)

CALCULATIONS:

Volume of water sample solution taken in titration flask = 25 ml.
Volume of HCl used for Phenolphthalein end point = V_1ml
Volume of HCl used for methyl orange end point = V_2ml
Equivalents of OH^-+ equivalents of ½ CO_3^{2-} = Equivalent of HCl in V_1 of N/10 HCl –(1)
Equivalents of OH^-+ equivalents of CO_3^{2-} = Equivalent of HCl in V_2 of N/10 HCl –(2)
Solving equation (1) & (2)
Equivalents of CO_3^{2-} = $2(V_2 - V_1)$ of N/10 HCl
Equivalents of OH^- = $(2 V_2 - V_1)$ of N/10 HCl

1) Calculation of alkalinity due to OH^-

   Strength of OH^- = $(2 V_2 - V_1)/(25\times10)$ equivalent/L
   Strength of OH^- in terms of $CaCO_3$ = $((2 V_2 - V_1)/250)\times50\times1000$ mg/l
   $=500(2 V_2 - V_1)$ ppm (in terms of $CaCO_3$)

2) Calculation of alkalinity to Na_2CO_3

   Strength of CO_3^- = $2(V_2 - V_2)/25\times10$ equivalent/L
   $=(2(V_2 - V_2)/250)\times50\times1000$ mg/l
   $=1000\times (V_2 - V_2)$ ppm (in terms of CaCO3)

RESULT: Alkalinity due to NaOH = ppm.
Alkalinity due toNa_2CO_3 ion = ppm.
Total Alkalinity = ppm

PRECAUTONS:

1. All the solutions should be freshly prepared
2. In each titration same amount of indicator should be added
3. There should not be any leakage from burette
4. While taking reading from burette, eye should be parallel to it.
5. Before using any solution, shake it properly

QUIZ QUESTIONS:

Q1. What is the suitable range for methyl orange and phenolphthalein to act as Indicators ?
Q2. Why OH^- and HCO_3^- ions can not exist together?
Q3. Give the reason to find the alkalinity in a given water sample?
Q4. Express all different type of alkalinity
Q5. Why do we use two indicators to find alkalinity in given water sample?

EXPERIMENT NO:- 2

OBJECT: To determine the temporary and permanent hardness of a given water sample by complexometric titration using EDTA.

APPARATUS:- Burette, Pipette, Conical Flask, Beaker Measuring cylinder.

CHEMICALS: N/100 EDTA Solution, Water sample, Eriochrome Black-T, Buffer Sol. (pH = 10)

THEORY: When Eriochrome black T (indicator) is added to hard water solution at around pH 10, it forms wine red colored unstable complex with Ca^{2+} and Mg^{2+} ions of the sample water. When this wine red colour solution is titrated against EDTA solution, the colour of the solution changes from wine red to blue colour at the end point.

Ethylene diamine tetraactic acid (EDTA) is a well knowing complexing agent which is widely used in analytical work, on account of its powerful complexing action and commercial availability.

PROCEDURE: Take 25 ml of hard water sample in a conical flask with help of a pipette. Add 5 ml of buffer solution. Add 5 drops of Eriochrome black-T indicator. Colour of the solution turns wine red. Titrate the solution against EDTA until the colour changes from wine red to blue. Repeat the titration for two concordant readings. This reading corresponds to total hardness.

Take about 250 ml of the hard water in 500 ml beaker and boil it for half an hour, cool it and titrate the solution as mentioned above for permanent hardness. Repeat the titration for two concordant readings

OBSERVATION:

Normality of EDTA solution = N/100

1. Indicator used Erichrome Black- T
   End point: Wine red → Blue

OBSERVATION TABLE:

For Tap Water

S.No	Volume of tap water in the Titration Flask (ml)	Burette Reading (ml)		Volume of N/100EDTA Solution used from burette (ml)
		Initial	Final	
1				
2				
3				

Volume of N/100EDTA Solution used from burette (ml) to neutralize10 ml of water sample = $v_2$ml

For Boiled Water

S.No	Volume of boiled water in the Titration Flask(ml)	Burette Reading(ml)		Volume of N/100 EDTA Solution used from burette(ml)
		Initial	Final	
1				
2				
3				

Volume of N/100EDTA Solution used from burette (ml) to neutralize10 ml of boiled water = $v_3$ml

CALCULATION:

a) Calculation of Total Hardness :-

Equivalents of Ca^{+2}&Mg^{+2} in the water sample = Equivalents of EDTA consumed.

$$N_1V_1=N_2V_2$$

(water) (EDTA)

$$N_1= (1/100 x v_2 ml)/25ml$$

Strength of total hardness causing ions in terms of $CaCO_3$ ions = N1x50 gm/litre

$= N_1\times50 \times1000$mgm/litre (in terms of calcium carbonate)

$= N_1\times50\times1000$ppm

b) Calculation of Permanent Hardness :-

Equivalents of Ca^{+2}&Mg^{+2} in the water sample = Equivalents of EDTA consumed.

$$N_1V_1=N_2V_2$$

(water) (EDTA)

$$N_1= (1/100\times V_3 ml)/25ml$$

Strength of Permanent Hardness hardness causing ions in terms of $CaCO_3$ ions = N_1x50 gm/litre

$= N_1\times50 \times1000$mgm/litre (in terms of calcium carbonate)

$= N_1 \times50\times1000$ppm

c) Calculation of Temporary Hardness = Total Hardness-Permanent Hardness

RESULT:

Total Hardness S_1 = ____________________ ppm

Permanent Hardness S_2 = ____________________ ppm

Temporary hardness $S_1 - S_2$ = ____________________ ppm

PRECAUTONS:

1. All the solutions should be freshly prepared
2. In each titration same amount of indicator and buffer solution should be added
3. There should not be any leakage from burette
4. While taking reading from burette, eye should be parallel to it.
5. Before using any solution, shake it properly

QUIZ QUESTIONS:

Q1. Why and how the pH value is adjusted to about 10?

Q2. What do you mean by the term hardness and how many types of hardness is there?

Q3. What are the differences between temporary and permanent hardness?

Q4. Why hardness is generally expressed in terms of $CaCO_3$ equivalents?

Q5. Write the full name and formula of EDTA. Also explain the mechanism of color change from wine red to blue.

EXPERIMENT NO:- 3

OBJECT: To determine the percentage available chlorine in given bleaching powder sample.

APPARATUS: Beaker, Pipette, Burette, Conical Flask, Measuring Cylinder

CHEMICALS: Bleaching powder sample, potassium iodide, dilute acetic acid, N/20 sodium thiosulphate solution, freshly prepared starch solution

THEORY: The amount of chlorine liberated by the action of dilute acids on bleaching powder is termed as available chlorine and expressed as percentage weight of bleaching powder. When dilute acetic acid reacts with bleaching powder than free chlorine is liberated. The liberated chlorine reacts with potassium iodide solution to give free iodine. This liberated free iodine is than titrated against n/20 sodium thiosulphate solution using freshly prepared starch solution.

$CaOCl_2 + 2CH_3COOH \rightarrow (CH_3COO)_2Ca + H_2 + Cl_2$

$Cl_2 + 2KI \rightarrow 2KCl + I_2$

(Liberated iodine)

$Starch + I_2 \rightarrow$ Starch iodine complex

(Dark blue colour)

$I_2 + 2NaS_2O_3 \rightarrow Na_2S_4O_6 + 2NaI$

PROCEDURE: Weighed 3 gm bleaching powder is dissolved in 250ml of tap water and shaken properly to obtain a homogeneous solution. Fill the burette after rinsing with hypo solution and note down initial burette reading. Pipette out 10ml of bleaching powder solution in 100ml titration flask. Add 3gm solid KI and 5ml of acetic acid, solution becomes dark brown.Titrate liberated iodine against sodium thiosulphate solution till light yellow colour persists. Add 6-7 drops of freshly prepared starch solution. Solution turns blue. Continue adding hypo solution till blue colour disappears. Repeat the same process to get three concordant readings.

OBSERVATION:

Normality of Sodium thiosulphate solution = N/20

1. Indicator used: Freshly prepared starch solution
   End point: Blue → Colorless

OBSERVATION TABLE:

S. No	Volume of bleaching powder solution in the Titration Flask (ml)	Burette Reading (ml)		Volume of N/20 sodium thiosulphate Solution used from burette(ml)
		Initial	Final	
1				
2				
3				

Volume of N/20 sodium thiosulphate Solution used from burette (ml) to neutralize10 ml of bleaching powder sample solution =$v_2$ml

CALCULATION:

$$N_1V_1 = N_2V_2$$

(Bleaching powder solution) (Hypo solution)

$$N_1 = (1/20 \times V_2 \text{ml})/10\text{ml}$$

Strength in terms of available chlorine = $N_1 \times 35.5$ gm/litre
250 ml of sample contains = 3gms of bleaching powder
% available chlorine in the given sample of bleaching powder= $(N_1 \times 35.5 \times 3 \times 100)/250$

RESULT: Percentage available chlorine in given sample of bleaching powder is--------------

PRECAUTONS:

1. Starch solutions should be freshly prepared
2. In each titration same amount of indicator, KI and acetic acid should be added
3. There should not be any leakage from burette
4. While taking reading from burette, eye should be parallel to it.
5. Lumps of bleaching powder should be powdered before making solution
6. Before using any solution, shake it properly

QUIZ QUESTIONS:

Q1. What is difference between available chlorine and chlorine content in bleaching Powder?
Q2. Why is a fresh solution of starch used?
Q3. For what purpose bleaching powder is used and why do we estimate chloride content in given Sample?
Q4. Why is starch indicator added near the end point?
Q5. Why blue color disappears at the end point in iodine titration?

EXPERIMENT NO:- 4

OBJECT: To determine the chloride content in given water sample by Mohr's method.

APPARATUS: Burette, Pipette, Conical Flask and Beaker.

CHEMICALS: Standard Silver Nitrate ($AgNO_3$) solution, Water sample, Potassium chromate (K_2CrO_4) indicator

THEORY: Mohr's method is used to determine chloride content in a water sample. In this method, chloride ion solution is titrated against standard silver nitrate solution using potassium chromate as indicator. As the titration proceeds, the chloride ions present react with $AgNO_3$ forming insoluble white precipitate of AgCl. The extra drop of $AgNO_3$ reacts with the indicator, forming red silver chromate. The appearance of distinct reddish brown colour over white precipitate marks the end point.

$$NaCl + AgNO_3 \rightarrow \underset{\text{White ppt.}}{AgCl} + NaNO_3$$

$$2AgNO_3 + K_2CrO_4 \rightarrow \underset{\text{Reddish brown}}{Ag_2CrO_4} + 2\,KNO_3$$

PROCEDURE: Pipette out 10 ml of water sample in a conical flask. Add 5 drops of freshly prepared K_2CrO_4 solution. Titrate it against standards $AgNO_3$ solution until the brick red colour persists. Repeat the titration till two concordant readings are obtained.

OBSERVATION:

Normality of $AgNO_3$ solution = N/50

1. Indicator used — Freshly prepared K_2CrO_4 solution
   End point: — colourless → Brick red precipitates

OBSERVATION TABLE:

S.No	Volume of water sample in the Titration Flask(ml)	Burette Reading(ml)		Volume of N/50$AgNO_3$Solution used from burette(ml)
		Initial	Final	
1				
2				
3				

Volume of N/50$AgNO_3$Solution used from burette (ml) to neutralize10 ml of water sample =V_2ml

CALCULATION:

$$\underset{\text{(water sample)}}{N_1V_1} = \underset{(AgNO_3\text{ solution})}{N_2V_2}$$

$$N_1 = (1/50 \times v_2 ml)/10ml$$

$$\text{Strength in terms of chloride content} = N_1 \times 35.5 \text{ gm/litre}$$

$$= N_1 \times 35.5 \times 1000 \text{mgm/litre}$$

$$= N_1 \times 35.5 \times 1000 \text{ppm}$$

RESULT: Amount of chloride content in given water sample = ----------ppm

PRECAUTONS:

1. In each titration same amount of indicator should be added
2. There should not be any leakage from burette
3. While taking reading from burette, eye should be parallel to it.

QUIZ QUESTIONS:

Q1 Why does $AgNO_3$ combine first with chloride ions in the water and not with K_2CrO_4
Q2 Can tap water be used for the preparation of $AgNO_3$ solution and why a bottle containing $AgNO_3$ is dark brown or always wrapped with carbon paper?
Q3Name the sources of chlorides in water
Q4 Name the indicator used in this experiment and what is the end point
Q5What is the principle of Mohr’s method

EXPERIMENT NO:- 5

OBJECT: To determine the iron content in the given sample by redox titration using external indicator.

APPARATUS: Burette, Pipette, conical flask, glass rod, beaker, measuring cylinder.

CHEMICALS: Mohr's salt, N/20 $K_2Cr_2O_7$, Potassium ferricyanide indicator, dilute sulphuric acid

THEORY: Potassium dichromate in acid medium oxidizes ferrous sulphate present in Mohr's salt into ferric sulphate .In this titration, potassium ferricyanide is used as an external indicator which gives a blue colour due to the formation of ferro ferricyanide.

$$Cr_2O_7^{2-} + 14\,H^+ + 6\,e^- \longrightarrow 2Cr^{3+} + 7H_2O$$

$$[Fe^{2+} \longrightarrow Fe^{3+} + e^-]\ X\ 6$$

$$Cr_2O_7^{2-} + 14\,H^+ + 6Fe^{2+} \longrightarrow 2Cr^{3+} + 7H_2O + 6Fe^{3+}$$

$$3Fe^{2+} + 2[Fe(CN)_6]^{3-} \longrightarrow Fe_3\,[Fe(CN)_6]_2$$

(Ferro ferricyanide)
(Blue complex)

PROCEDURE: Pipette out 10 ml of the sample solution in a conical flask and add 5 ml of dilute H_2SO_4. Titrate the solution against N/20 $K_2Cr_2O_7$ solution, using potassium ferricyanide as an external indicator. Take 1 drop of the solution from the conical flask and put it over a drop of potassium ferricyanide solution placed on a white glazed tile. If a blue colour appears, then the end point has not reached. Add more $K_2Cr_2O_7$ solution till a drop of solution does not changed to blue. This is the end point. Repeat till three concordant readings are obtained.

OBSERVATION:

Normality of $K_2Cr_2O_7$solution = N/20

1. Indicator used   Freshly prepared Potassium ferricyanide solution
   End point: Yellow → Brown

OBSERVATION TABLE:

S.No	Volume of Mohr's salt solution in the Titration Flask(ml)	Burette Reading(ml)		Volume of N/20 $K_2Cr_2O_7$ Solution used from burette(ml)
		Initial	Final	
1				
2				
3				

Volume of N/20 $K_2Cr_2O_7$ Solution used from burette (ml) to neutralize10 ml of Mohr's salt solution = $v_2$ml

CALCULATION:

$$N_1V_1 = N_2V_2$$

(Mohr salt sample) ($K_2Cr_2O_7$ solution)

$$N_1 = (1/20 x v_2 ml)/10ml$$

Strength in terms of Mohr's salt = N_1x392 gm/litre

392gm of Mohr's salt contains =56gm of iron

N_1x392 gm of Mohr's salt contains = 56/392× (N_1x392)gm of iron

= $56N_1$ gm of iron

$= 56 \times N_1 \times 1000$mgm of iron
$= 56 \times N_1 \times 1000$ppm of iron

RESULT: The iron content in the given sample of iron is………ppm.

PRECAUTONS:

1. In each titration same amount of indicator and dilute sulphuric acid should be added
2. There should not be any leakage from burette
3. While taking reading from burette, eye should be parallel to it.

QUIZ QUESTIONS

Q1. Why is ferrous ammonium sulphate solution used in the titration and not direct ferrous sulphate?
Q2. What is the formula of ferrous ammonium sulphate and any other name for it?
Q3. Why is dilute H_2SO_4 added while preparing standard solution of ferrous ammonium solution?
Q4. Why is $K_3Fe(CN)_6^-$ potassium ferricyanide is used as external indicator in ferrous ammonium sulphate *vs* $K_2Cr_2O_7$ titration?
Q5. Why is dilute H_2SO_4 and not HCl/HNO_3 used in redox titrations?

EXPERIMENT NO:- 6

OBJECT: Determination of concentration of HCl solution by titrating it against standard NaOH solution using a pH-meter.

CHEMICALS: HCl, N/10 NaOH, Solutions of pH = 4 & 9

APPARATUS: pH meter, burette, pipette, beakers, measuring cylinder

THEORY: When an alkali is added to an acid solution, the pH of the solution increases slowly, but at the vicinity of the equivalence point, the rate of change of pH of the solution is very rapid. From the sharp break in the curve, we can find the equivalence point, from which the strength can be calculated by normality equation.

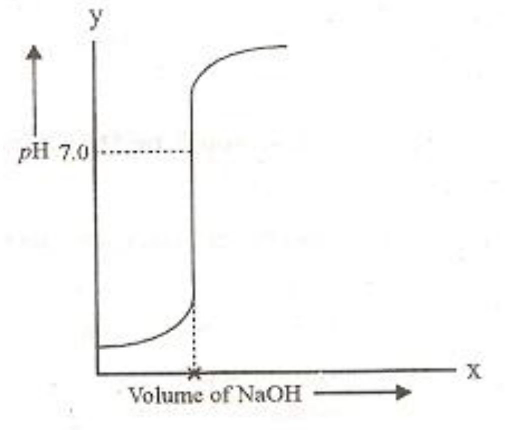
PROCEDURE: First standardize the pH meter with a buffer of pH 4 & 9.Take 50ml of HCl solution in a 100 ml. beaker so that the glass electrodes is completely dipped. Note the pH of pure acid solution. Now add 2 ml of N/10 NaOH from the burette in the beaker. Stir the contents well. Note the pH of the solution. Now go on adding NaOH solution from the burette and note the pH of the solution after each addition of 2 ml of N/10 NaOH. After equivalence point there will be sudden change in pH value i.e it will jump to basic range. After completion of experiment draw a graph between volumes of NaOH added vs pH values. Join two curves and find the volume of N/10 NaOH required to neutralize at pH=7 which is equivalence point

OBSERVATION:

Volume of HCl taken = 50ml
Normality of NaOH solution=N/10

SNO	Vol. of N/10 NaOH	pH	SNO	Vol. of N/10 NaOH	pH
1	0.0		14	26.0	
2	2.0		15	28.0	
3	4.0		16	30.0	
4	6.0		17	32.0	
5	8.0		18	34.0	
6	10.0		19	36.0	
7	12.0		20	38.0	
8	14.0		21	40.0	
9	16.0		22	42.0	
10	18.0		23	44.0	
11	20.0		24	46.0	
12	22.0		25	48.0	
13	24.0		26	50.0	

GRAPH:



Plot between pH and volume of NaOH

Volume of N/10 NaOH Solution used from burette (ml) to neutralize50 ml of HCl solution (From Graph) =$v_2$ml

CALCULATIONS: $N_1V_1 = N_2V_2$

(HCl sample) (NaOH solution)

$N_1 = (1/10 x v_2 ml)/10ml$

Strength HCl solution = N_1x36.5 gm/litre

= N_1×36.5 ×1000mgm/litre

= N_1×36.5×1000ppm

RESULT: The strength of the given HCl solution = …………….. ppm.

PRECAUTIONS:

1. pHmeter should of switched on 15 minutes before performing the experiment
2. pH meter should be standardized properly
3. Temperature should be kept at 25^{o}C
4. Each time equal amount of NaOH should be added

QUIZ QUESTIONS

Q1. Define pH and mention the pH of distilled water at 25^{o}C.
Q2. Do you use any indicator in pH titration?
Q3. Which method is more accurate to find the end point volumetric or Ph method and why?
Q4. How is the end point determined in pH metric titration?
Q5.Why is there a sudden jump from acidic value to basic value?

EXPERIMENT NO: 7

OBJECT: To determine Viscosity of an addition polymer like polyester by viscometer

APPARATUS: Ostwald viscometer, Beaker, Wash bottle, Stop watch, Sucker, Pipette, Electronic balance

CHEMICALS: Water, Polystyrene

THEORY: Viscosity also known as coefficient of viscosity is the tangential force per unit area required to move one horizontal plane of the fluid at unit velocity with respect to another maintained at a unit distance apart by the fluid. The units of viscosity are poise or centipoises. In 1844 Hagen–Poiseuille did their work concerning the interpretation that liquid flow through tubes and he proposed an equation for viscosity of liquids. This equation is called Poiseuille's equation.

$$\eta = \frac{\pi r^4 P t}{8VL} \qquad \text{....(1)}$$

Where η is called the viscosity coefficient, t is the time of flow of liquid, V is the volume of the liquid, P is the hydrostatic pressure, and L is the distance travelled by the liquid during time t. In the honour of Hagen–Poiseuille the unit of viscosity is called the Poise (P). The official SI unit for absolute viscosity is kg/m s (or Pascal-seconds, Pa s).

Ostwald viscometer is a commonly used viscometer, which consists of a U-shaped glass tube held vertically. For more accurate measurements it is held in a controlled temperature bath. It is also known as a glass capillary viscometer. A liquid is allowed to flow through its capillary tube between two etched marks and the time of flow of the liquid is measured using a stopwatch.

In an Ostwald viscometer the measured distance the liquid travels, L, will be always a constant; the radius, r will always be a constant; and by procedure the volume of liquid, V will also be constant. Equation (1) can then be simplified to:

$$\eta = KPt \qquad \text{---------(2)}$$

Where K is a constant and

$$K = \frac{\pi r^4}{8VL}$$

The hydrostatic pressure is P proportional to the density of the fluid being measured. In this experiment we will be measuring the mass of equal volumes of liquid so that the viscosity will be proportional to the masses measured. Therefore relation will be:

$$\eta \ \alpha \ Kmt \qquad \text{---------(3)}$$

Where K and t are defined above and m is the mass of the liquid.

For finding the viscosity of liquids it is important to calibrate the viscometer using a reference liquid. Water is a commonly used reference liquid. The viscosity of water at 30.0 °C is 0.8007 centipoise (cP). Knowing the values for the reference liquid and relation (3), we get:

$$\frac{\eta}{\eta_r} = \frac{mt}{m_r t_r} \qquad \text{----------(4)}$$

where: η_r is viscosity coefficient of the reference sample (water), m_r is the mass of the reference sample, and t_r is the time flow of the reference sample. Note that K cancels out. The other variables are the viscosity coefficient, mass, and time flow of the sample respectively.

With an Ostwald viscometer we can measure the time flow of a liquid (mass can be measured using standard laboratory procedures, e.g. a relative density bottle and a scale) and determine its viscosity by solving equation (4) for η.

$$\eta = mt\left(\frac{\eta_r}{m_r t_r}\right) \qquad \text{------------(5)}$$

PROCEDURE: The viscometer is fixed vertically on the stand and 10 mL or 20mL of water is pipetted into the lower bulb. The volume of water (10 mL or 20 mL) is chosen so that the liquid can be conveniently sucked into the upper bulb leaving some in the lower bulb. It is sucked up into the other bulb to a point about the mark above the bulb. Now it is released and stop clock is started when the meniscus crosses the mark. The clock is stopped when the mark below the bulb is passed. The time is recorded at the moment. The same procedure is repeated twice or thrice and their average is used in calculations.

Similarly, the experiment is repeated with the given polymer solution. Using the specific gravity bottle, one can determine the specific gravity of the liquid and calculate the viscosity.

OBSERVATION TABLE AND CALCULATIONS:

Solvents	**Flow Time (sec)**		
	i	ii	Mean
Water			
Polystyrene			

Room temperature = ……….^{O}C
Mass of water =
Mass of Polystyrene =
Time of flow of water =...........................sec
Time of flow of polystyrene solution =......................sec
Absolute viscosity of water = 0.8007 centipoise
Absolute viscosity of the polystyrene =........................centipoise

RESULT: Absolute viscosity of the polystyrene = ...centipoise

PRECAUTIONS:

1. Always wear lab coat and gloves when you are in the lab. When you enter the lab, switch on the exhaust fan and make sure that all the chemicals and reagents required for the experiment are available. If they are not available, prepare the reagents using the components for reagent preparation.
2. Make sure to clean all your working apparatus with chromic acid and distilled water and ensure that all the apparatus are free from water droplets while performing the experiment.
3. Make sure to calibrate the electronic weigh balance before taking the measurements.
4. Ensure that the stop watch and hot air gun are working properly.
5. Clean all glassware with soap and distilled water. Once the experiment is completed recap the reagent bottles. Switch off the light, and exhaust fan before leaving the lab.

QUIZ QUESTIONS:

Q1. What do you mean by viscosity?
Q2. What is the effect of temperature on viscosity? Explain the reason
Q3. How many types of viscometers are there and what is the difference between them?
Q4. What is the principle involved for measurement of viscosity?

EXPRIMENTNO:- 8

OBJECT: Determination of iron concentration in sample of water by colorimetry using KCNS as colour developing agent.

APPARATUS: Colorimeter, Burette, Pipette, Measuring Cylinder and Beaker, test tubes, Cuvette

CHEMICALS: 0.1% Fe^{3+} -ion solution (with excess HCl), 20% KCNS in distilled water.

THEORY: Spectroscopy is that branch of science which deals with the interaction of matter with electromagnetic radiations, which are different waves of energy. It covers a wide range of wavelengths or energies and visible light is part of electromagnetic radiations. When monochromatic visible light falls on the homogeneous medium, the intensity of transmitted light is less than that of incident light. A part of incident light has been absorbed.

$$I_o=I_a+I_t$$

I_o- intensity of incident light, I_a- intensity of absorbed light, I_t- intensity of transmitted light

A Spectrometer is a device which detects the percentage transmittance of light radiation, when the light of certain intensity and frequency range is passed through the sample. It is based on Lambert Beer Law which states that "when a monochromatic light is passed through the sample medium, the intensity of that light decreases exponentially with the increase in concentration and thickness of absorbing medium."

$$I_t=I_o e^{-ct}$$

Absorbance or optical density is defined as:

$$A=\log I_o/I_t= €CT$$

A- Absorbance

€- Molar Extinction coefficient or Molar absorptivity

T- Thickness of medium

C- Concentration of the absorbing medium

If the same sample cell i.e. T is constant is used for measurement of absorbance of solution having different concentrations then extent of absorbance (A) is directly proportional to the concentration (C) of the medium.

Thus, if a graph is plotted between A and C, we get a straight line in compliance with Lambert Beer Law. This is known as calibration curve.

For measuring the concentration of given solute, the calibration curve is obtained by measuring the absorbance of standard solution of different concentration. This calibration curve is then used to measure the conc, of unknown solution.

PROCEDURE:

a) **Setting of colorimeter**:

Insert a cuvette containing distilled water in the sample holder adjust the control knob to get zero absorbance

b) **Determination of λ_{max}**:

Insert the cuvette containing given colored solution and note the absorbance reading at different wavelengths or filters. At a specific wavelength maximum absorbance will be observed with given coloured solution

c) **Determination of concentration of sample solution**:

Once λ_{max} is fixed. Prepare the following solution from given sample solution and distilled water using a measuring cylinder

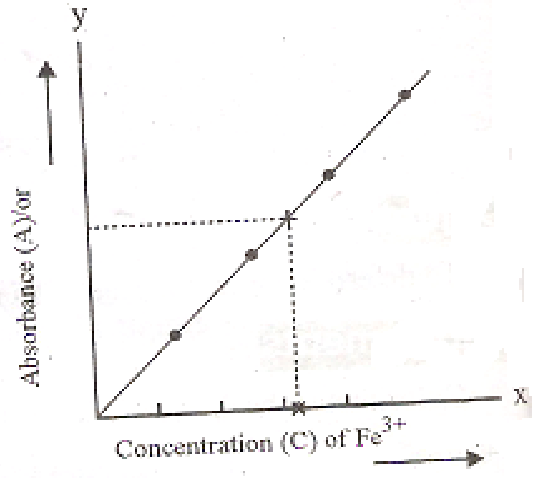
Solution	I	II	III	IV	V	VI	VII	VIII	IX	X
Given sample(ml)	1	2	3	4	5	6	7	8	9	10
Distilled water(ml)	9	8	7	6	5	4	3	2	1	0
% concentration	10	20	30	40	50	60	70	80	90	100

Note down the value of absorbance of above prepared solutions in series. Plot a graph of values of absorbance so obtained and respective concentration should be a straight line. Now note down the absorbance of unknown concentration solution.

OBSERVATION TABLE:

Solution. No.	% Conc. of given sample solution	Absorbance at λ_{max}
I	10	
II	20	
III	30	
IV	40	
V	50	
VI	60	
VII	70	
VIII	80	
IX	90	
X	100	
Unknown solution		

GRAPH:



CALCULATIONS: Concentration of unknown solution from graph= A gm/litre

392 gm of given sample contains=56gm of iron

A gm of given sample contains=56/392×Agm/litre

= (56xA×1000)/392mgm/litre

RESULT: The concentration of Fe (III) in the given sample is obtained from the graph & calculated to be ………ppm

PRECAUTIONS:

1) For the preparation of calibration curve, dilute solutions of unknown concentration should be used
2) λ_{max} should be carefully observed
3) Cuvette should be handled carefully as it is fragile
4) Cuvette should be clean and dry from outside
5) Colorimeter should be switched on 15 minutes before the start of experiment

QUIZ QUESTIONS

Q1 Explain the law on which this experiment is based and how do we prove it.

Q2 What is the advantage of colorimetric method?

Q3 What is visible spectrum? What happens when visible spectrum is made to fall upon a colored solution?

Q4 Filters are invariably used in absorption spectroscopy. why?

Q5 What is the source of light in calorimeter?

EXPERIMENT NO:- 9 A

OBJECT: To detect the elements in given organic samples.

APPARATUS: Porcelain dish, test tubes, beaker

CHEMICALS: Pieces of sodium metal, distilled water, Samples containing S, N, Cl, Br, I etc

THEORY AND PROCEDURE:

Test for N, S, and halogens elements

SL.	Experiment	Observation	Inference
I	**For Nitrogen**		
1	Take 2 ml of Sodium Extract + 2 ml. of freshly prepared aqueous Sol. of $FeS0_4$+ 1-2 drops of NaOH.	A Green precipitate is obtained	N present
2	Boil the above mixture cool and add sufficient dilute. H_2SO_4 to dissolve the $Fe(OH)_2$ add 2-3 drop of $FeCl_3$ solution.	a) Blue or green colour is obtained b) Appearance of Red blood colour	a) N present b) N & S present
3	Take 2 ml of Sodium extract , acidify with CH_3COOH add 2-3 drops of 1% solution of benzidine in 50% acetic acid and 1-2 drops of 1% solution of copper sulfate	A blue colour or precipitate	Nitrogen present
II	**FOR SULPHUR**		
1	Take 2 ml of sodium extract an add 1-2 ml of freshly prepared solution of sodium Nitroprousside.	A violet colour	Sulfur present
2	Take 2 ml of sodium extract acidify with acetic acid and add a few drop of lead acetate solution	A black precipitate of lead sulfide	Sulfur present
III	**FOR HALOGEN**		
1	Take 1 ml of sodium extract + 0.5 ml conc. HNO_3, boil cool and add silver nitrate solution	A curdy white precipitate soluble easily the ammonium hydroxide and insoluble in HNO_3	Chlorine present
		If gives a yellowish white or yellow precipitatet respectively 1. White AgBr precipitate, is soluble only in great excess of NH_4OH with difficulty 2. Completely insoluble in NH_4OH	Bromine present Iodine present
2	Sodium Extract + CCl_4 chlorine water. Shake well. Observe the colour of CCl_4 layer	1) Violet colour 2) Yellow, Brown or red	Iodine Confirmed Bromide Confirmed

RESULT:

1. Sample-1 contains--------------
2. Sample-2 contains--------------

3. Sample-3 contains--------------
4. Sample-4 contains--------------
5. Sample-5 contains--------------

PRECAUTIONS:

1. All the test tubes should be cleaned properly.
2. All the reagents should be freshly prepared.
3. While preparing sodium extract ignition tubes should be hold away from the face.

EXPERIMENT NO:- 9 B

OBJECT: To identify the functional groups in given organic samples.

APPARATUS: Test tubes, beaker, Test tube holder

CHEMICALS: Distilled water, Samples with required functional groups
Sodium bicarbonate solution, Neutral Ferric chloride solution, Sulphuric Acid, Sodium Hydroxide, Phthalic anhydride, Sodium Metal, Na_2SO_4, Ethanol, Sodium nitroprusside, etc.

Theory: Test for Functional Groups:-

SL.	Experiment	Observation	Inference
I	**For Carboxylic Acid**		
2	Take a few drops sample and add 5 ml of cold $NaHCO_3$ solution. in water	Effervescence due to the evolution of CO_2	-COOH
3	To a part of original solution add 5 drops of ethyl alcohol and conc. H_2SO_4 and lead acetate	A fruity smell of ethyl ester	-COOH
II	**For Phenols**		
1	To a part of alcoholic solution of original solution and 2-3 drops of aq. $FeCl_3$ solution.	Violet, green, red, blue colour indicate the presence of phenolic OH group	Violet phenol (O-cresol or P-cresol) Blue –Violet (Resorcional)
2	Take part of original solution and add phthalic anhydride in a dry test tube, add 0.5 ml of conc H_2SO_4 and heat. Pour the content of the test tube into 50 ml water containing about 1 ml dilute aq. NaOH.	A green fluorescence	Shows the presence of Phenol.
III	**For Carbohydrate**		
1	Take part of original solution in a dry test tube and 1-2 drops of conc. H_2SO_4	A red or crimson colour	presence of carbohydrate
2	Heat part of original solution with 1 ml of conc. H_2SO_4	An immediate charring	presence of carbohydrate
3	Take 2 ml of aqueous solution of original sample and add 1 ml of Molischis reagent and then add carefully 1 ml of conc. H_2SO_4 from the side of the test tube.	A reddish violet ring at the junction of the two liquid is formed.	presence of carbohydrate
IV	**For Ketones**		
1	Take 2 ml of aqueous or alcoholic solution of	A yellow or red colour	presence of a

	original solution, and add 0.5 ml of Sodium nitroprusside and 2-3 drops of aq. NaOH		Ketone
2	Take 1 ml alcoholic solution of original solution and add 1 ml of 2;4-dinitro phenyl hydrazine solution, boil and cool	A yellow orange or red precipitate	presence of a Ketone
V	**For Aldehyde**		
1	Take 1 ml of a mixture of equal amounts of Fehling solution A & B, and add a few drops of original solution and heat on a water bath for 5 minutes.	A red precipitate of Cu_2O is obtained	presence of and aldehyde
2	Take 1 ml of Tollen's reagent and a few drops of original solution and heat on a water bath for 5-10 minutes.	A silver mirror is obtained due to deposition of metallic silver on the side of the test tube.	presence of an aldehyde.

RESULT:

1. Sample-1 contains-------------
2. Sample-2 contains-------------
3. Sample-3 contains-------------
4. Sample-4 contains-------------
5. Sample-5 contains-------------

PRECAUTIONS:

1. All the test tubes should be cleaned properly.
2. All the reagents should be freshly prepared.
3. Test tubes should be hold away from the face.

QUIZ QUESTIONS:

Q1 What do you mean by functional group?
Q2 what is the difference between a functional group and an element?
Q3 Which one is more acidic, phenol or carboxylic acid?
Q4 Why do you fuse sodium with organic compounds?
Q5 Why is metallic sodium kept in kerosene oil? Can potassium be used instead of sodium

EXPERIMENT NO: 10

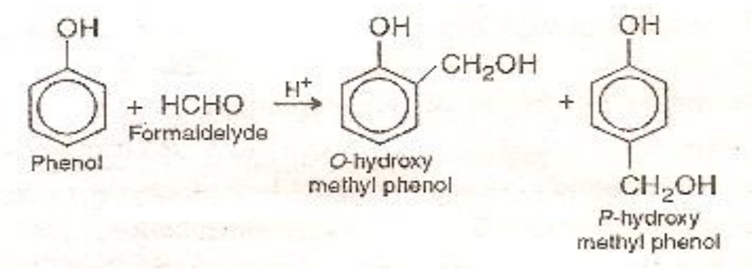
OBJECT: To prepare phenol formaldehyde (Bakelite) resin

APPARATUS: Measuring cylinder, beakers, glass rod, funnel, filter papers

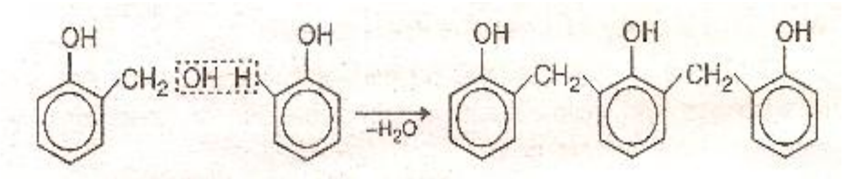
CHEMICALS: Phenol, Glacial acetic acid, Formaldehyde, and conc. HCl acid

THEORY:

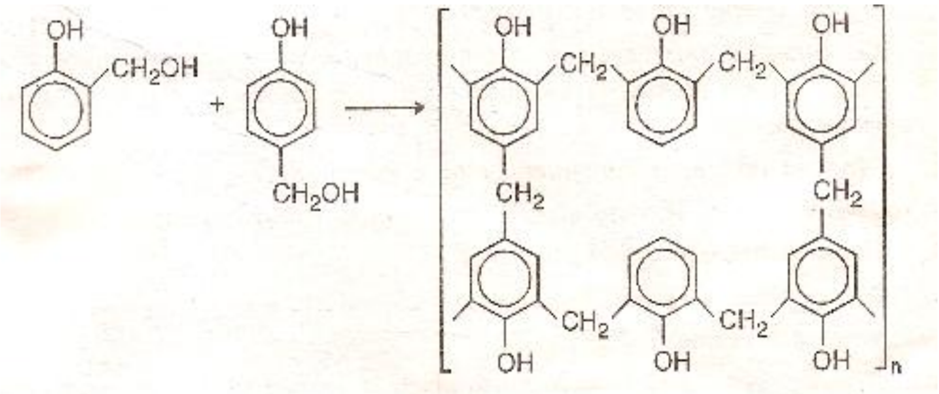
Phenol formaldehyde resin can be preparared by condensation polymerization phenol with formaldehyde in acidic medium. Reaction takes place in three steps;
Step1: Phenol reacts with formaldehyde in acidic medium



Step2: Hydroxy methyl phenol reacts with phenol to give linear polymer.



Step3: It polymerizes at high temperature into a three dimensional cross-linked polymer.



PROCEDURE FOR PREPATION OF BAKELITE: Take 5ml of glacial acetic acid and 2.5ml of 40% formaldehyde solution in a beaker. To this add 2ml of phenol. Add 5ml of Conc. HCl acid to the reaction mixture drop wise with continuous stirring till pink colour 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.

PROCEDURE FOR PREPATION OF UREA FORMALDEHYDE RESIN: In a 250ml Round bottom flask take 35ml.of formaline and adjust the pH to 7.0-7.5 using 1M NaOH and B.D.H. Universal Indicator. Now add 12.5gms of urea and 2ml of liquor ammonia. Reflux the content of the flask until the first bubble by slightly exothermic reaction appears. Then heat it gently under reflux for one hour. From the start of the exothermic reaction care should be taken that the content of the flask are agitated whilest refluxing the solution, and then cool it to room temp. Transfer the resin syrup in to previously weighted test tube.
RESULT: Weight of Phenol formaldehyde and urea Formaldehyde resin is-----------gm

PRECAUTIONS:

1) Reaction is vigorous. All addition should be done carefully and with continuous stirring
2) Fuming cupboard should be used for preparation.

QUIZ QUESTIONS:

Q1 Define a polymer?
Q2 What is the common name for phenol formaldehyde resin?
Q3Why is bakelite used in electrical appliances?
Q4 Define condensation polymerization and resins
Q5 Name a trifunctional monomer?