EN 14 104(P)

ENGINEERING CHEMISTRY

LABORATORY MANUAL

List of Experiments

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OBSERVATIONS

Record of weighing

Article weighed	*weights added*		*Rider at*	*Weight due to rider*	*Total Weight*
	grams	*milligrams*			
Weighing bottle+$K_2Cr_2O_7$					
Weighing bottle alone					

Estimation of ferrous iron

Ferrous iron solution Vs $K_2Cr_2O_7$ (N-phenyl anthranilic acid)

Serial No:	*Volume of ferrous iron solution(ml)*	*Burette reading*		*Volume of $K_2Cr_2O_7$ (ml)*
		Initial	*Final*	

EXPERIMENT NO. 1

ESTIMATION OF FERROUS IRON USING STANDARD POTASSIUM DICHROMATE

AIM

Estimate the amount of ferrous iron in the whole of the given solution of ferrous ammonium sulphate, supplied with A. R. potassium dichromate.

PRINCIPLE AND OUTLINE

The estimation is based on the reaction of potassium dichromate in acid medium with ferrous ammonium sulphate. The dichromate oxidises ferrous sulphate to ferric sulphate, itself getting reduced to green chromic salt

$$K_2Cr_2O_7 + 4H_2SO_4 \rightarrow K_2SO_4 + Cr_2(SO_4)_3 + 4H_2O + 3[O]$$

$$2FeSO_4 + H_2SO_4 + [O] \rightarrow Fe_2(SO_4)_3 + H_2O$$

A standard solution of potassium dichromate is prepared by dissolving requisite amount of A.R. quality crystals. The given ferrous salt solution is made up to a known volume and it is standardised using the standard potassium dichromate solution. N-phenyl anthranilic acid is used as internal indicator. On the addition of the indicator the solution becomes colourless, after adding a few milliliters of the dichromate solution, it becomes green and at the end point the colour changes from green to violet red.

PROCEDURE

(a) Preparation of standard potassium dichromate solution

1.2 g of A.R. potassium dichromate crystals is accurately weighed and transferred into a 250ml standard flask. It is dissolved in water and made up to the mark.

CALCULATION

Mass of potassium dichromate solution = W g

Normality potassium dichromate solution,

$$N_1 = \frac{W \times 4}{49.04} = \text{.....N}$$

Volume of ferrous salt solution taken, V_1 = 20 ml

Volume of potassium dichromate solution used up, V_2 = ... ml

Let the normality of ferrous salt solution solution be = N_2

$V_1 x N_1 = V_2 x N_2$

$N_2 = (V_1 x N_1)/V_2$

Mass of ferrous iron per litre = $N_2 x 55.84$ g/L

Mass of ferrous iron in the whole of the given solution = $(N_2 x 55.84)/10$ g

(b) Estimation of ferrous iron.

The given ferrous salt solution is transferred to a 100 ml standard flask and made up to the mark and well shaken. 20 ml of the made up solution is pipetted out into a conical flask. About 20 ml of 2N sulphuric acid is added followed by 10 drops of N-phenyl anthranilic acid as indicator. The solution is then titrated with vigorous shaking against the standard potassium dichromate solution until green solution changes to violet red. The titration is repeated till concordant values are obtained.

From the titre values, the mass of ferrous iron in the whole of the solution is calculated.

RESULT

Mass of ferrous iron in the whole of the given solution = ...g

OBSERVATIONS

Record of weighing

Article weighed	*weights added*		*Rider at*	*Weight due to rider*	*Total Weight*
	grams	*milligrams*			
Weighing bottle+$Na_2S_2O_3$					
Weighing bottle alone					

Estimation of dissolved oxygen

Sample water Vs $Na_2S_2O_3$ (Starch)

Serial No:	*Volume of sample water (ml)*	*Burette reading*		*Volume of $Na_2S_2O_3$ (ml)*
		Initial	*Final*	

EXPERIMENT 2

DETERMINATION OF DISSOLVED OXYGEN IN THE GIVEN SAMPLE OF WATER

AIM

To determine dissolved oxygen in the given sample of water.

PRINCIPLE

5 to 7 ppm of dissolved oxygen is present in unpolluted water and is a must for supporting aquatic lives. In presence of a good amount of dissolved oxygen (>8 ppm), aerobic bacteria lead to oxidation of organic compounds present in water. This kind of oxidation is called 'aerobic oxidation'. But if the dissolved oxygen is <5 ppm, anaerobic oxidation of organic compounds present in water takes place by anaerobic bacteria. If the water is polluted with large amount of organic matter, a large amount of dissolved oxygen is rapidly consumed in the biological aerobic oxidation. The decrease in the dissolved oxygen in turn decreases the population of aquatic life.

Dissolved oxygen is usually determined by Winkler's method. It is based on the fact that dissolved oxygen oxidises potassium iodide (KI) to iodine. The liberated iodine is titrated against standard sodium thiosulphate (also known as hypo) solution using starch as indicator. Since dissolved oxygen in water is in the molecular state, it as such cannot oxidise KI. Hence, manganese hydroxide is used as an oxygen carrier to bring about the reaction between KI and oxygen. Manganese hydroxide, in turn, is obtained by the action of KOH on manganese sulphate ($MnSO_4$).

$$MnSO_4 + 2\,KOH \rightarrow Mn(OH)_2 + K_2SO_4$$

$$2\,Mn(OH)_2 + O_2 \rightarrow 2\,MnO(OH)_2$$

$$MnO(OH)_2 + H_2SO_4 \rightarrow MnSO_4 + 2\,H_2O + [O]$$

CALCULATIONS

Weight of thio in 100ml = W g

Normality of thio solution ,N_1 = W x 10/ 248.18

= .....N

Volume of thio equivalent to 100ml of water,V_1 =..... ml

Volume of sample water taken for titration ,V_2 = 100 mL

Normality of water sample, N_2 = $N_1 x V_1 / V_2$

Since, equivalent weight of O_2 =8 g

Hence, strength of oxygen = N_2 x 8 g/L

Amount of dissolved oxygen = $N_2 x 8 x 1000$ ppm

$$2KI+H_2SO_4+[O] \rightarrow K_2SO_4+H_2O+I_2$$

$$2\ Na_2S_2O_3+I_2 \rightarrow Na_2S_4O_6+2NaI$$

PROCEDURE

a) Preparation of standard thiosulphate solution

0.25g of sodium thiosulphate crystals are accurately weighed.It is transferred into a 100ml standard flask and made upto the mark.

b) Estimation of dissolved oxygen

Take 500 ml of sample water in a bottle avoiding as far as possible contact with air. Immediately add 4 ml of manganese sulphate solution and 4 ml of alkaline KI solution. Stopper the bottle and shake the contents thoroughly. Add 4 ml of concentrated H_2SO_4 when the precipitate is settled. Shake the bottle until the precipitate has completely dissolved. Allow the solution to stand for 5 minutes. Take 100 ml of this solution and titrate against N/100 thio solution using starch as indicator. At the end point, the blue colour will disappear.

RESULT

The amount of dissolved oxygen = ... ppm.

OBSERVATIONS

Record of weighing

Article weighed	*weights added*		*Rider at*	*Weight due to rider*	*Total Weight*
	grams	*milligrams*			
Weighing bottle+ $ZnSO_4.7H_2O$					
Weighing bottle alone					

Standardisation of EDTA Solution

$ZnSO_4.7H_2O$ Vs EDTA (Eriochrome black-T)

Serial No:	*Volume of $ZnSO_4.7H_2O$ (ml)*	*Burette reading*		*Volume of EDTA (ml)*
		Initial	*Final*	

Estimation of total hardness of the water sample

Hard water Vs EDTA (Eriochrome black-T)

Serial No:	*Volume of hard water (ml)*	*Burette reading*		*Volume of EDTA (ml)*
		Initial	*Final*	

EXPERIMENT No. 3

ESTIMATION OF TOTAL HARDNESS OF A GIVEN WATER SAMPLE

AIM

To estimate the total hardness of a given sample of water by using EDTA. [Ethylene Diamine Tetra Acetic acid]

PRINCIPLE

The hardness of water is due to soluble salts of calcium and magnesium. The concentration of these metallic salts can be determined by using a dye stuff Eriochrome black-T indicator. When the indicator is added to the sample of water containing Ca and Mg ions, red coloured metal indicator complexes are formed at a pH of 10. On adding EDTA, the indicator ions are replaced by EDTA ions to form more stronger metal-EDTA complexes. At the end point ,when all the metal-indicator complex change into metal -EDTA complex ,the free indicator gives a blue colour.

$$M^{2+} + H_2Y^{2-} \longrightarrow MY^{2-} + 2H^+$$

A pH of 10 is required during the titration for the stability of the metal - EDTA complex as well as for the sharp colour change of the indicator at the end point.

The hardness is estimated in terms of weight of $CaCO_3$ per litre of water in ppm.

PROCEDURE

a) Standardisation of EDTA Solution

Weighed out accurately 0.72g of $ZnSO_4.7H_2O$ and made upto 100ml, in water. 20ml of the std $ZnSO_4.7H_2O$ is pipetted out into a conical flask. 2ml

CALCULATION

Weight of $ZnSO_4.7H_2O$ in100ml =Wg

Molarity of $ZnSO_4.7H_2O$,M_1 =Wx10/287.54= .....M

(a) Volume of $ZnSO_4.7H_2O$, V_1 = 20 ml

Vol of EDTA ,V_2 = ...ml

Molarity of EDTA, M_2 =M_1V_1/V_2

Volume of hard water =20ml

(b) Volume of EDTA = V_3

Molarity of hard water M_3 =$M_2V_3/20$

Wt. of $CaCO_3$ per litre of the water = M_3x100= A g/l

Hardness of sample of water = Ax1000 ppm

of buffer of pH = 10 is added to it. 8 drops of indicator is added to it. The solution turned into wine red colour. It is titrated against EDTA solution, taken in the burette, The end point is the colour change from wine red to blue. Titrations are repeated for concordant values.

b) Estimation of total hardness of the water sample

The given solution is made upto 100ml. 20ml of the sample of hard water is pipetted out into a conical flask, 2 ml of buffer is added to it. 8 drops of indicator are added to get a wine red colour. It is then titrated against standard EDTA solution till the solution gives a blue colour. Titrations are repeated for concordant values. From the titre value, concentration of hard water and hardness can be calculated. The hardness of water is expressed in parts per million of $CaCO_3$.

RESULT

Hardness of the given sample of water =.....ppm

OBSERVATIONS

Record of weighing

Article weighed	*weights added*		*Rider at*	*Weight due to rider*	*Total Weight*
	grams	*milligrams*			
Weighing bottle+$K_2Cr_2O_7$					
Weighing bottle alone					

Standardisation of Sodium thiosulphate solution

$K_2Cr_2O_7$ Vs $Na_2S_2O_3$ (Starch)

Serial No:	*Volume of $K_2Cr_2O_7$ (ml)*	*Burette reading*		*Volume of $Na_2S_2O_3$(ml)*
		Initial	*Final*	

Estimation of copper

$CuSO_4$ solution Vs $Na_2S_2O_3$ (Starch)

Serial No:	*Volume of $CuSO_4$ solution(ml)*	*Burette reading*		*Volume of $Na_2S_2O_3$ (ml)*
		Initial	*Final*	

EXPERIMENT NO. 4
ESTIMATION OF COPPER IN A GIVEN SAMPLE

AIM

To determine the amount of copper in the given sample, being provided with A.R. $K_2Cr_2O_7$ and approximately N/20 solution of sodium thiosulphate.

PRINCIPLE

The given copper sulphate solution is made up to a definite volume. A definite volume of this solution is pipetted out and potassium iodide is added when an equivalent amount of iodine will be set free.

The liberated iodine is then titrated against the sodium thiosulphate solution using starch as indicator. From the titre values the mass of copper in the whole of the given solution can be calculated.

$$CuSO_4 + 2KI \rightarrow CuI_2 + K_2SO_4$$

$$2\ CuI_2 \rightarrow Cu_2I_2 + I_2$$

$$I_2 + 2Na_2S_2O_3 \rightarrow 2\ NaI + Na_2S_4O_6$$

PROCEDURE

1) Preparation of std $K_2Cr_2O_7$

About 0.25g of $K_2Cr_2O_7$ is accurately weighed out and transferred into a 100ml standard flask. It is made up to the mark with distilled water.

CALCULATIONS

Weight of $K_2Cr_2O_7$ in 100ml = Wg

Normality of $K_2Cr_2O_7$, N_1 = (Wx10/49.04)= .....N

(a) Volume of $K_2Cr_2O_7$ pipetted, V_1 = 20 ml

Volume of thio used, V_2 = ...ml

Normality of thio, N_2 = $(V_1xN_{1)} / V_2$

= .....N

(b) Volume of copper solution used, V_3 = 20 ml

Volume of thio used ,V_4 = ...ml

Normality of copper sulphate solution,N_3 = $(N_2xV_4) /V_3$

= .....N

Equivalent weight of Cu = 63.54g

Weight of copper in the whole of the given solution =$(N_3x63.54)/10$ g

2) Standardisation of Sodium thiosulphate

20m1 of $K_2Cr_2O_7$ solution is pipetted out into a conical flask. Add 3 ml of con. HC1 and 10 ml of 5% KI. It is then titrated against thio using starch as indicator until the colour changes from blue to pale green.

3) Estimation of copper

The given copper solution is made up to 100ml. 20 ml of the solution is pipetted out into a conical flask. Add ammonia solution drop by drop till a permanent precipitate is formed. The precipitate is redissolved in acetic acid. 15 ml of 5% KI is added. The liberated iodine is titrated against thio using starch as indicator towards the end. Titration is continued till blue colour is discharged. Repeated for concordant values.

RESULT

Mass of copper in the whole of the given solution =.....g

OBSERVATIONS

Record of weighing

Article weighed	*weights added*		*Rider at*	*Weight due to rider*	*Total Weight*
	grams	*milligrams*			
Weighing bottle+$K_2Cr_2O_7$					
Weighing bottle alone					

Standardisation of sodium thiosulphate solution.

$K_2Cr_2O_7$ Vs $Na_2S_2O_3$ (Starch)

Serial No:	*Volume of $K_2Cr_2O_7$ (ml)*	*Burette reading*		*Volume of $Na_2S_2O_3$(ml)*
		Initial	*Final*	

Estimation of bleaching powder

Bleaching powder solution Vs $Na_2S_2O_3$ (Starch)

Serial No:	*Volume of bleaching powder solution (ml)*	*Burette reading*		*Volume of $Na_2S_2O_3$(ml)*
		Initial	*Final*	

EXPERIMENT NO. 5

ESTIMATION OF BLEACHING POWDER

AIM

Estimate the amount of chlorine in bleaching powder, being supplied with approximately N/20 sodium thiosulphate solution and A.R potassium dichromate crystals.

PRINCIPLE AND OUTLINE

Bleaching powder is a mixture of calcium hypochlorite, $CaOCl_2$ and the basic chloride, $CaCl_2Ca(OH)_2.H_2O$ The active constituent is the hypochlorite, which is responsible for the bleaching action.

$$CaOCl_2 + H_2SO_4 \longrightarrow CaSO_4 + H_2O + Cl_2$$

A definite mass of bleaching powder suspended in water is treated with potassium iodide in acid medium. An equivalent amount of iodine is set free which is titrated against the sodium thiosulphate solution.

$$2CaOCl_2 + 4KI + 4CH_3COOH \longrightarrow 2CaCl_2 + 4CH_3COOK + 2H_2O + 2I_2$$

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

A standard solution of potassium dichromate is prepared and using this sodium thiosulphate solution is standardised.

$$K_2Cr_2O_7 + 6KI + 14HCl \longrightarrow 2CrCl_3 + 8KCl + 7H_2O + 3Cl_2$$

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

CALCULATION

Mass of potassium dichromate taken = W g

Normality of potassium dichromate solution, N_1 = (Wx10)/49.04

(a) Volume of potassium dichromate solution , V_1 = 20 ml

Volume of sodium thiosulphate solution used, V_2 = ...ml

Normality sodium thiosulphate solution, N_2 = $(N_1 x V_1)/V_2$

(b) Volume of bleaching powder, V_3 = 20 ml

Volume of sodium thiosulphate solution used , V_4 = ...ml

Normality of chlorine from bleaching powder solution , N_3 = $N_2 x V_4 / V_3$

Mass of chlorine in whole of the solution = $(N_3 x 35.46)/10$ =..... g

From the equation, it is clear that the equivalent mass of potassium dichromate = Molecular mass/6 = 294.2/6 = 49.04. In both the titrations, starch is used as the indicator.

PROCEDURE

(a) Preparation of standard N/20 potassium dichromate solution. About 0.25 g of A.R potassium dichromate is weighed accurately by using a chemical balance. It is transferred to a 100ml standard flask, dissolved in water and made up to the mark.

(b) Standardisation of sodium thiosulphate solution.

20 ml of the potassium dichromate solution is pipetted out into a conical flask. About 10 ml of 5% potassium iodide and 3 ml of concentrated hydrochloric acid are added.It is then titrated against sodium thiosulphate solution taken in the burette. When the solution becomes pale yellow, about 2 ml of freshly prepared cold solution of starch is added. A blue colour will then develop. Sodium thiosulphate solution is then added drop by drop until the colour just changes from blue to light green. The light green colour of the solution at the end point is due to the chromic salt formed by the reduction of the dichromate solution. The titration is repeated till concordant values are obtained.

Estimation of bleaching powder

The given solution is made upto 100ml. 20 ml of the solution in a state of very fine suspension is pipetted out into a 250 ml conical flask. 10 ml of 5% potassium iodide solution is added followed by 10 ml of glacial acetic acid. The liberated iodine is titrated against standard sodium thiosulphate solution. About 2 ml of freshly prepared starch solution is added when the solution has a pale yellow colour and the titration continued dropwise untithe colour just disappears. Titratons are repeated till concordant values are obtained.

RESULT

Mass of chlorine in the given sample of solution =.....g

OBSERVATIONS

Record of weighing

Article weighed	*weights added*		*Rider at*	*Weight due to rider*	*Total Weight*
	grams	*milligrams*			
Weighing bottle+$K_2Cr_2O_7$					
Weighing bottle alone					

Estimation of Ferric iron

serial No:	*volume of solution in (ml)*	*concentration ((Y x V)/100) ml*	*Absorbance*
1	Blank		
2	2 ml		
3	4 ml		
4	6 ml		
5	8 ml		
6	10 ml		
7	unknown		

EXPERIMENT NO. 6

ESTIMATION OF FERRIC IRON COLORIMETRICALLY

AIM

Estimate the mass of iron in the given ferric alum solution.

PRINCIPLE AND OUTLINE

Traces of iron are usually determined colorimetrically with thiocyanate as reagent. Ferric iron reacts with thiocyanate to give an intensely red coloured compound. More than one product can be formed. Depending upon the thiocyanate concentration a series of complexes represented by Fe(CNS)n + 3-n, where n=l ..... 6. can be obtained. The colour of all these complexes are red. Therefore in the colorimetric determination of iron, it is desirable to use a large excess of the reagent.

Owing to strong hydrolysis, solutions of ferric salts in water contain relatively few ferric ions. Addition of a strong acid suppresses hydrolysis and therefore intensifies the colour obtained with thiocyanate. The effect of the acid depends upon its kind and its concentration. Sulphuric acid should not be used because sulphate ions have a tendency to form complexes with ferric ions and consequently above a certain acidity the colour intensity decreases with increasing sulphuric acid concentration.

PROCEDURE

A standard solution of iron containing 0.1 mg per ml of iron is prepared as follows. 0.864 g of ferric alum is weighed into a 100 ml standard flask. 10 ml of conc. hydrochloric acid (iron free) is added to prevent hydrolysis. It is then made up to 100ml (solution A). 10 ml of this solution is pipetted into

CALCULATION

Molecular mass of ferric alum = 482.19

Mass of ferric alum taken = W g

Mass of iron present in 1 ml of standard solution =(Wx 55.85)/(482.19x1000)

= X g

=X x 10^3= Y mg

Concentration of unknown solution from the graph = ...mg/l

Mass of iron in the whole of the given solution =.....mg

another standard flask and made up to 100 ml (solution B) . Solution B now contains 0.1 mg per ml.

2, 4, 6, 8 and 10 ml of this standard solution are taken in different 100 ml standard flasks. To each 5ml of 4N hydrochloric acid and 10 ml of 20% potassium thiocyanate are added and the volume is made up to 100ml. A blank is also prepared by taking 5ml of 4N hydrochloric and 10 ml of 20% potassium thiocyanate in a 100ml standard flask. The intensities of colour developed are compared by using a photoelectric colorimeter.

The given unknown solution is taken in 100ml standard flask and the colour is developed as above.

A standard calibration curve is drawn by plotting concentration on the X-axis against absorbance on the Y-axis. A straight line graph is obtained. From the graph the concentration of the unknown solution with respect to the standard solution is calculated.

RESULT

Mass of iron in the given solution = ... g

EXPERIMENT NO. 7

TO PREPARE BUFFERS AND STANDARDISATION OF pH METER

AIM

To standardize a pH meter

PROCEDURE

Buffers at the pH range 4.0, 7.0 and 9.2 are prepared. Dip the electrode in 7.0 pH solution. The reading will show 7.0 + or - some counts. Adjust the reading to 7.0 pH by calibrate control. Take out the electodes and clean the surface with distilled water . Rinse it properly and do not wipe it. Dip the electode in 4.0 pH solutions. The reading will show 4.0 + or - some counts. Adjust the reading to 4.0 pH using slope control on rear side. Take out and clean the electrodes.

The pH meter is now calibrated to make any pH measurement. To confirm the calibration, dip the electrodes in 9.2 pH solution. The reading should show 9.2 pH. Take out and clean the electrodes.

RESULT

The pH meter is calibrated.

EXPERIMENT NO. 8

PREPARATION OF UREA -FORMALDEHYDE RESIN

AIM

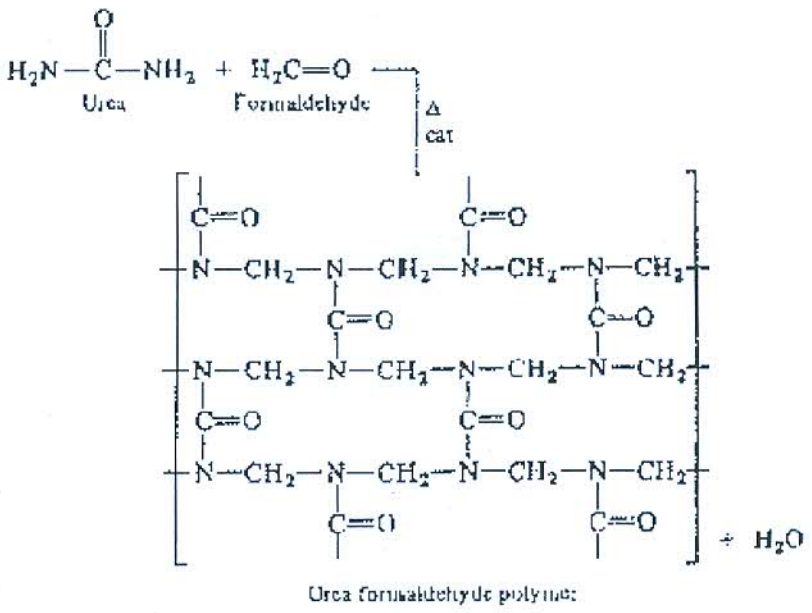
To prepare a polymer of Urea and formaldehyde

APPARATUS AND REAGENTS REQUIRED

100ml beaker, glass rod, graduated pipette, 40% formaldehyde, urea, conc. H_2SO_4, distilled water

PRINCIPLE

Amino resins are condensation products which are obtained by the reaction of urea with formaldehyde . Commercially important ammonia resin is urea-formaldehyde, which is prepared by the reaction between 2 parts of urea and 1 part of formaldehyde in basic medium, about 50 ^{0}C in stainless steel vessel,The structure of UF resin:



Urea formaldehyde polymer

For moulding , the methylol derivatives are compounded and then cured. During the process of curing long C-N-C-N- chains are formed.

PROCEDURE

In 100ml beaker take 2ml of 40% HCHO. To this add about 10g urea with continuous stirring until a saturated solution is obtained. Add a few drops of conc.H_2SO_4 continuously. All of a sudden a voluminous white mass appears in the beaker. When the reaction is complete, wash the residue with water and dry the product and calculate the mass of the product formed.

RESULT

Mass of the product formed = ... ***g***

EXPERIMENT NO. 9

PREPARATION OF PHENOL FORMALDEHYDE RESIN

AIM

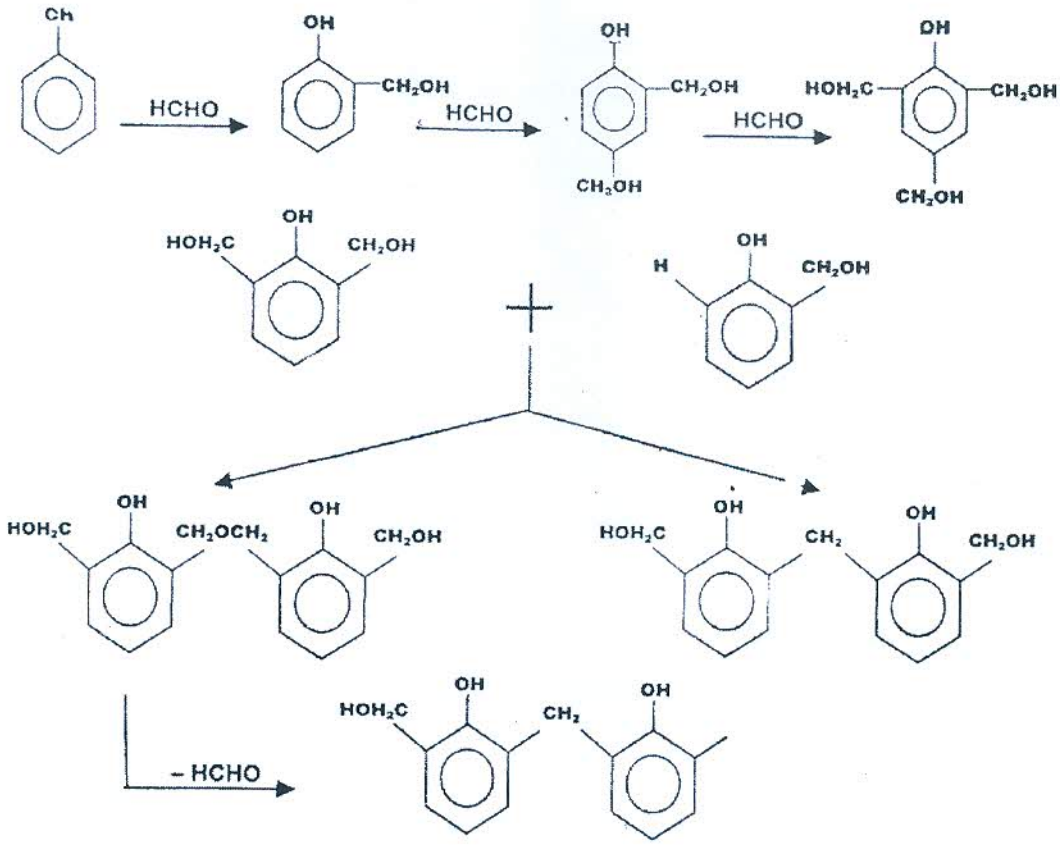
To prepare a polymer of phenol and formaldehyde.

Apparatus and reagents required

Glacial Acetic acid, 40% formaldehyde Solution, phenol, conc.HCl, Glass rod, beakers, glass funnel, filter paper.

Theory

Phenolic resins are condensation polymerization products of phenolic derivative with aldehydes. Most important member of this class is Bakelite or phenol formaldehyde resin. It is prepared by condensing phenol with formaldehyde in presence of acidic/alkaline catalyst. The structure of resin is as given below.



PROCEDURE

Place 5 ml of glacial acetic and 2.5 ml of 40% formaldehyde solution in a 500 ml beaker and add 2ml of phenol. Add a few ml of con.HCl in to the mixture carefully and stir for at least 10 minutes. Heat it on a water bath, stirring should continue during heating .Within 5 mints a large mass of pink plastic is formed. The residue obtained is washed several times with distilled water and dried . The yield is calculated.

RESULT

Mass of the product formed = ***g***