

EXPERIMENT: 1

Aim: To prepare glyptal resin.

Requirements: Phthalic anhydride, Glycerol, Round bottom flask, Reflux condenser, Oil bath.

Process: In a 250ml Round bottom flask, take 15gms of Phthalic anhydride and 5ml.of glycerol. Attach an condenser to the neck of flask. Reflux the content of the flask on oil bath at 150°C to 170°C for two hours. Transfer the brown yellow resin in to a previously weighed test tube. Note down the weight of glyptal resin formed.

Result: Weight of glyptal resin formed = ----- gms.

Observations:

(1) Weight of Phthalic anhydride taken = ----- gms.

(2) Volume of glycerol taken = ----- ml.

Equation:

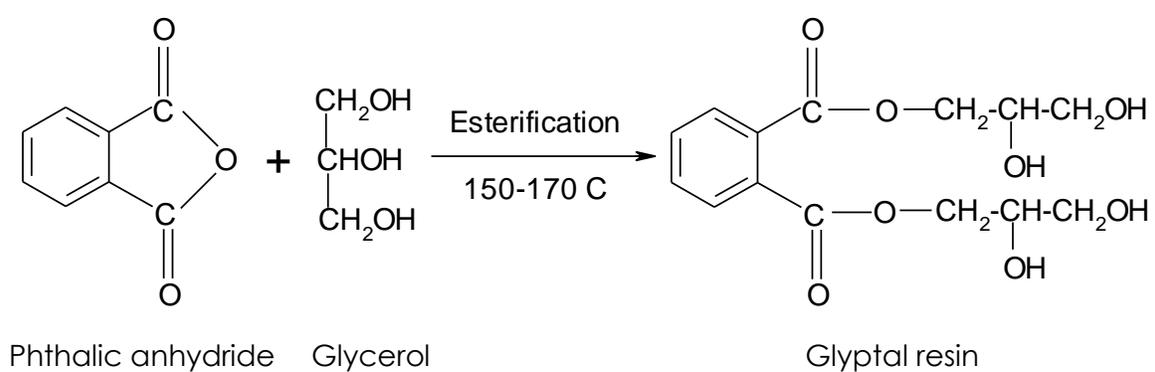


Figure:

EXPERIMENT: 2

PREPARATION OF PHENOL FORMALDEHYDE RESIN

Aim: To prepare phenol formaldehyde resin (Novalak).

Requirements: Phenol, formaline, oxalic acid, Round bottom flask, Reflux condenser.

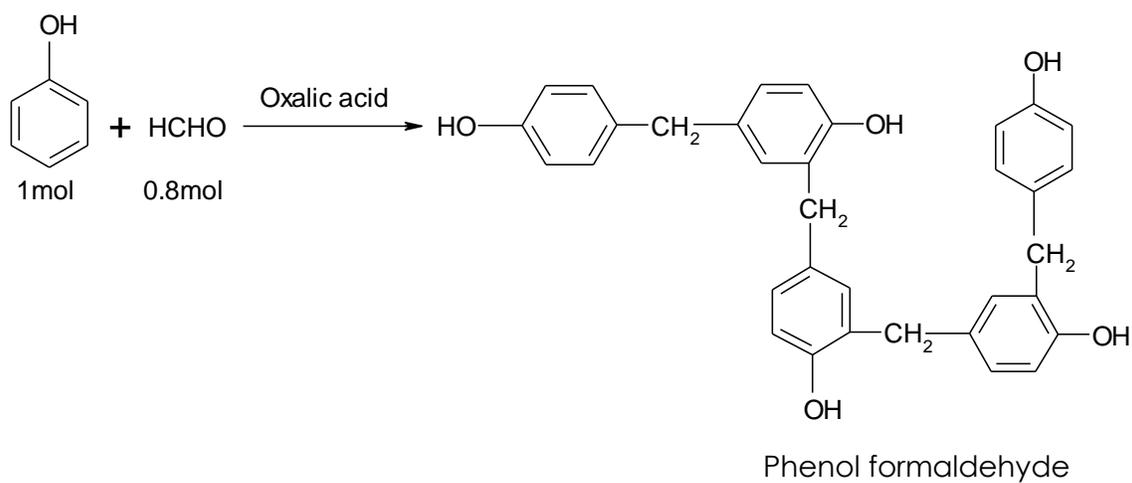
Process: In a 250ml Round bottom flask, take 25gms phenol and 16ml.of formaline and 0.375gms Oxalic acids. The liquor is warmed continuously under reflux by means of a low Bunsen flame applied by hand directly to the flask. As soon as bubble start to rise, showing that the exothermic reaction has begun the burner is taken away and the liquor is allowed to boil on its own. Heat the flask to 60-80°C for at least two to three hours. A time will reach when the bubble will escape very difficultly from the surface of the solution. At that time stop heating and pour immediately the content of flask in previously weighed test tube.

Result: Weight of phenol formaldehyde resin formed = ----- gms.

Observations:

- (1) Weight of phenol taken = ----- gms.
- (2) Volume of formaline taken = ----- ml.

Equation:



EXPERIMENT: 3

PREPARATION OF PHENOL FORMALDEHYDE RESIN

Aim: To prepare phenol formaldehyde resin (Resol).

Requirements: Phenol, formaline, 40% NaOH, Round bottom flask, Reflux condenser.

Process: In a 250ml Round bottom flask, take 8gms of phenol, 15ml.of formaline and 1ml of 40% NaOH and mixed them together. Attach reflux condenser to the flask. Reflux the content of the flask directly on wire gauze. First heat slowly then strongly for 30min. After this period the mixture acquires red color and it becomes viscous on heating. A time will reach when the bubble will escape very difficultly from the surface of the solution. At that time stop heating and pour immediately the content of flask in previously weighted test tube.

Result: Weight of phenol formaldehyde resin formed = ----- gms.

Observations:

(1) Weight of phenol taken = ----- gms.

(2) Volume of formaline taken = ----- ml.

Equation:

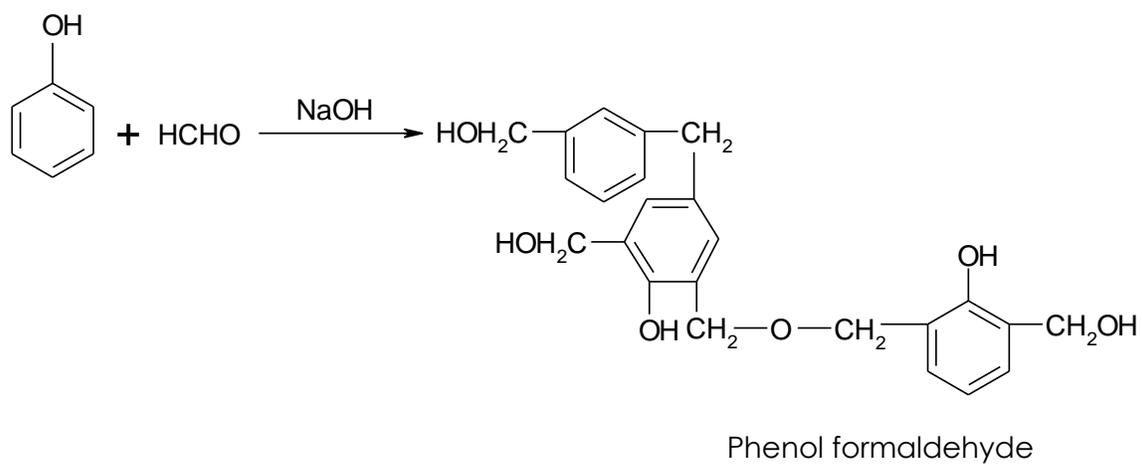


Figure:

EXPERIMENT: 4

PREPARATION OF UREA FORMALDEHYDE RESIN

Aim: To prepare urea formaldehyde resin.

Requirements: Urea, formaline, 1M NaOH, Universal Indicator, ammonia,
Round
bottom flask, Reflux condenser.

Process: 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 Urea formaldehyde resin formed = ----- gms.

Observations:

- (1) Weight of Urea taken = ----- gms.
- (2) Volume of formaline taken = ----- ml.
- (3) Volume of Ammonia taken = -----ml.

Equation:

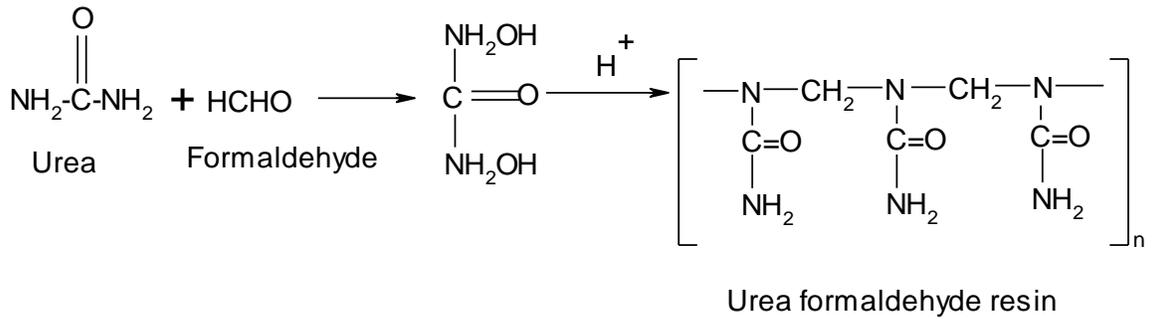


Figure:

EXPERIMENT: 5

PREPARATION OF MELAMINE FORMALDEHYDE RESIN

Aim: To prepare melamine formaldehyde resin.

Requirements: Melamine, formaline, 1MNaOH, Universal Indicator, liquor ammonia, Round bottom flask, Reflux condenser.

Process: In a 250ml Round bottom flask take 35ml.of formaline and adjust the pH of it to 8.0-8.5 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 of slightly exothermic reaction appears. If necessary take away the flask. 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 is agitated while refluxing the solution, and then cool it to room temp. Transfer the resin syrup in to previously weighted test tube.

Result: Weight of Melamine formaldehyde resin formed ----- gms.

Observations:

- (1) Weight of Melamine taken = ----- gms.
- (2) Volume of formaline taken = ----- ml.
- (3) Volume of Ammonia taken = -----ml.

Equation:

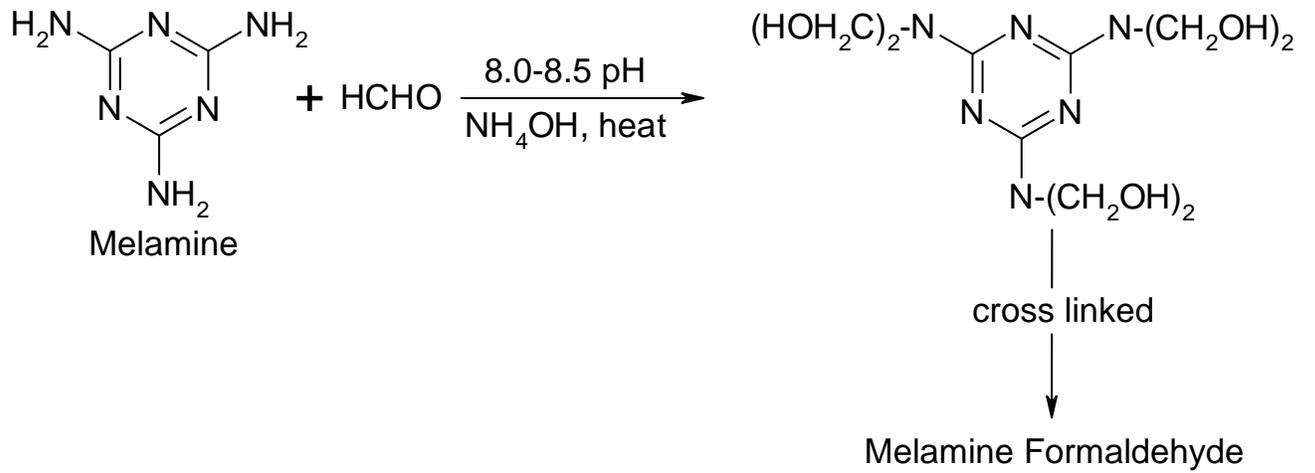


Figure:

EXPERIMENT: 6

ESTIMATION OF % FORMALINE

Aim: To determine % Formaline in a given formaline solution.

Requirements: 10ml. 10% Hydroxyaminehydrochloride, conical flask, burette, pipette, 0.5N solution, Phenolphthalein indicator.

Process: Weight accurately about 1-1.4gm of the given sample in the capsule and transfer it into a conical flask. Add exactly 10ml. of 10% Hydroxyaminehydrochloride soln. along with 50ml of water. Titrate it against 0.5N NaOH by adding bromophenol blue as an indicator. End point will be yellow to purple colour. Find out % formaldehyde using given formula.

Result: % Formaline in the given solution = -----

Observations:

(1) Weight of sample = ----- gms.

In burette: 0.5N NaOH solution

In conical flask: Solution of sample.

Indicator: Bromophenol blue

End point: yellow to purple.

Calculations:

$$\% \text{ formaline} = \frac{\text{B.R.} \times \text{Normality of NaOH} \times 3.602}{\text{Weight of sample}}$$

EXPERIMENT: 7

PREPARATION OF CELLULOSE NITRATE POLYMER

Aim: To prepare cellulose nitrate polymer from cellulose using Nitrating mixture.

Requirements: Mercerized cotton, Concentrated HNO_3 , Concentrated H_2SO_4 , Acetone, conical flask, beaker, water bath, and thermometer.

Process: Prepare a nitrating mixture by taking 8ml. water and 60ml. of concentrated HNO_3 acid in a clean dry conical flask. Cool the flask in ice bath to 10°C and add 60ml. of concentrated H_2SO_4 in such a way that temperature should not rise above 20°C .

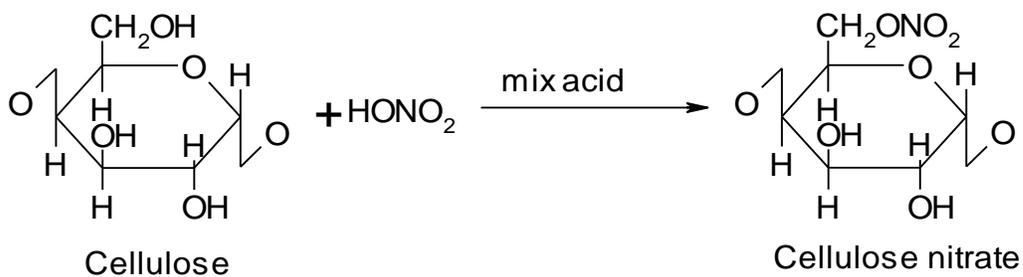
Now add 1gms of mercerized cotton piece into the nitrating mixture. Place the flask for 3 hours at room temperature with occasional stirring. After three hour, removes cotton from nitrating mixture, wash with water so that it is free from acid. Test with litmus paper. Dissolve the cotton in minimum quantity of acetone. Pour the solution of acetone into the ice cold water taken in a beaker. Precipitates of cellulose nitrate are separated out, filter and wash it with cold water. Dry it and determine the weight of the polymer formed.

Result: Weight of Cellulose nitrate formed = ----- gms.

Observations:

- (1) Weight of Mercerized cotton taken = -----
gms.
- (2) Volume of Con.HNO₃ taken = ----- ml.
- (3) Volume of Con.H₂SO₄ taken = -----ml.

Equation:



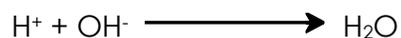
EXPERIMENT: 8

ACID VALUE

Aim: Determination of acid value of a given polymer sample.

Requirements: Capsule, conical flask, burette, pipette, Acetone, 0.2M KOH soln., Phenolphthalein indicator.

Theory: Total acidity, Acid number or Acid value of polymer is the amount of titrating base, expressed as mg of KOH, required to neutralize all acidic constituents of 1 gm of the sample.



Process: Weight accurately about 1gm of the given resin sample in a previously weighted glass capsule. Transfer the capsule with its content in a clean conical flask, add 75ml. acetone. Warm the solution of flask for complete dissolution of resin in acetone. Cool and titrate the content of flask as rapidly as possible against standard alkali solution from burette using Phenolphthalein indicator until the pink color appears.

Result: Acid value of the given sample = -----

Observations:

(1) Weight of capsule + resin = ----- gms.

(2) Weight of capsule = ----- gms.

(3) Weight of resin = -----gms

In burette: 0.2M KOH solution

In conical flask: Solution of polymer in acetone.

Indicator: Phenolphthalein

End point: Colorless to pink.

Calculation:

$$\text{Acid value} = \frac{56.1 \times \text{Burette reading} \times \text{Morality of KOH}}{\text{Weight of sample taken}}$$

EXPERIMENT: 9
HYDROXYLVALUE

Aim: Determination of Hydroxyl value of a resin.

Requirements: Water bath, water condenser, conical flask, burette, pipette, Acetic anhydride- pyridine mixture, 1M KOH solution, Phenolphthalein indicator.

Theory: (Total) Basicity, Base value or hydroxyl value or base number or hydroxyl number of a polymer is the amount of titrating acid, expressed as mg of equivalent KOH, required to neutralize all the basic constituents of 1gm of the sample.

Process: Weigh accurately about 1gm of the given resin sample and transfer it into a conical flask. Add exactly 8ml. of acetylating reagent (1 vol. of acetic anhydride + 3 vol. of pyridine). Attach a reflux condenser to the flask and reflux over water bath for 3-2 hours. Cool the flask and add 20-30ml of distilled water slowly through the condenser. The flask is allowed to cool again and titrate the content of flask as rapidly as possible against standard alkali solution from burette until the pink color appears using Phenolphthalein indicator.

Result: Hydroxyl value of the given sample = -----

Observations:

(1) Weight of capsule + resin = ----- gms.

(2) Weight of capsule = ----- gms.

(3) Weight of resin = -----gms

(4) Weight of Acetylating reagent taken = -----ml.

In burette: 1M KOH solution

In conical flask: Solution of polymer.

Indicator: Phenolphthalein

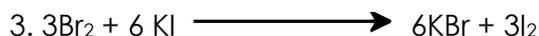
End point: Colorless to pink.

EXPERIMENT: 10
ESTIMATION OF PHENOL

Aim: Estimate the amount of phenol present in the given solution.

Theory: Phenol (-OH group) is estimated by bromination method. It is a method of quantitative precipitation of tribromophenol method. The solution is acidified; the bromine liberates and reacts with phenol to form the tribromo derivative. Unconsumed bromine reacts with KI; displaced I₂ is titrated with standard Na₂S₂O₃ solution. The reading gives the quantity of bromine used.

Equation:



Process:

Part-I Standardization: Take 10ml. of brominating solution from a burette in a glass stoppered bottle and one test tube of water. Add 5ml. concentrated HCl and one test tube of 10% KI solution. Cork the bottle and shake well. Titrate immediately the liberated I₂ against 0.1N Na₂S₂O₃ solution

from the burette using starch as an indicator. End point will be from blue to colorless.

Part-II Estimation: Dilute the given solution to 250ml in a standard measuring flask with distilled water. Pipette out 25ml of it in a glass stoppered bottle. Add 5ml of concentrated HCl and 25ml of brominating solution from the burette. Cork the bottle, shake it well and keep for 10min., add one test tube of 10% KI soln., cork the bottle and shake well. Titrate immediately the liberated I_2 against 0.1N $Na_2S_2O_3$ soln. from the burette, using starch indicator. End point will be from blue to colorless.

Results:

- (1) 25 ml. of diluted solution required (C) = ----- ml. of brominating solution in terms of 0.1N $Na_2S_2O_3$ solution.
- (2) Amount of phenol present = -----gm.

Calculations:

10ml. of brominating solution required X ml. of 0.1N $\text{Na}_2\text{S}_2\text{O}_3$ solution
 $25\text{ml.} = X \times 25 / 10 = A \text{ ml}$

Quantity of brominating soln. added = A ml. (in terms of 0.1N $\text{Na}_2\text{S}_2\text{O}_3$ soln)

Quantity of brominating solution unused = Y ml.

(Back titration reading from the burette)

Quantity of brominating soln used up by 25ml. of diluted phenol solution. = (A-Y) = C ml.

250 ml. diluted soln. used (in terms of 0.1N $\text{Na}_2\text{S}_2\text{O}_3$ solution) brominating solution = $10 \times C \text{ ml}$.

Since 6 $\text{Na}_2\text{S}_2\text{O}_3$ solution = 1 phenol

Then, 1 $\text{Na}_2\text{S}_2\text{O}_3$ solution = $1/6$ phenol

1000ml. $\text{Na}_2\text{S}_2\text{O}_3$ solution = 15.55gm of phenol

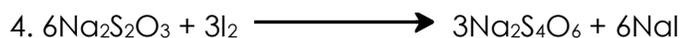
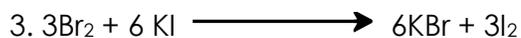
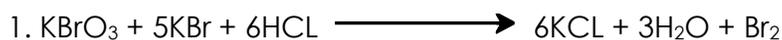
10 C ml. of 0.1N $\text{Na}_2\text{S}_2\text{O}_3$ soln = $15.55 \times 0.1 \times 10C / 1000$

EXPERIMENT: 11
ESTIMATION OF ANILINE

Aim: Estimate the amount of aniline present in the given solution.

Theory: Aniline is estimated by bromination method. It is method of quantitative precipitation of tribromoaniline method. The solution is acidified; the bromine liberates and reacts with aniline to form the tribromo derivative. Unconsumed bromine reacts with KI, displaces I₂ is titrated with standard Na₂S₂O₃ soln. The reading gives the quantity of bromine used.

Equation:



Process:

Part-I Standardization: Take 10ml. of brominating solution from a burette in a glass stoppered bottle and one test tube of water. Add 5ml. con. HCL and one test tube of 10% KI soln. Cork the bottle and shake well. Titrate immediately the liberated I₂ against 0.1N Na₂S₂O₃ soln. from the burette using starch as an indicator. End point will be from blue to colorless.

Part-II Estimation: Dilute the given solution to 250ml in a standard measuring flask with distilled water. Pipette out 25ml of it in a glass stoppered bottle. Add 5ml of conc. HCl and 25ml of brominating soln. from the burette. Cork the bottle, shake it well and keep for 10min. add one test tube of 10% KI soln., cork the bottle and shake well. Titrate immediately the liberated I₂ against 0.1N Na₂S₂O₃ soln. from the burette, using starch indicator. End point will be from blue to colorless.

Results:

(1) 25 ml. of diluted solution required (C) = ----- ml. of
brominating solution in terms of 0.1N Na₂S₂O₃ solution.

(2) Amount of aniline present = -----gm.

Calculations:

10ml. of brominating soln. required X ml. of 0.1N $\text{Na}_2\text{S}_2\text{O}_3$ solution
25ml. = $X \times 25 / 10$
= A ml. Quantity of
brominating soln. added = A ml. (In
terms of 0.1N $\text{Na}_2\text{S}_2\text{O}_3$ soln)

Quantity of brominating soln. unused = Y ml
(Back titration reading from the burette)

Quantity of brominating soln used up by 25ml. of diluted
Aniline solution = (A-Y) = C ml.

250 ml. diluted soln. used (in terms of 0.1N $\text{Na}_2\text{S}_2\text{O}_3$ soln)
brominating soln. = $10 \times C$ ml.

Since 6 $\text{Na}_2\text{S}_2\text{O}_3$ solution = 1 aniline

Then, 1 $\text{Na}_2\text{S}_2\text{O}_3$ solution = $1/6$ aniline

1000ml. $\text{Na}_2\text{S}_2\text{O}_3$ solution = 15.55gm of aniline

10 C ml. of 0.1N $\text{Na}_2\text{S}_2\text{O}_3$ solution = $15.55 \times 0.1 \times 10C / 1000$
gm. of Aniline

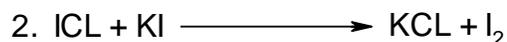
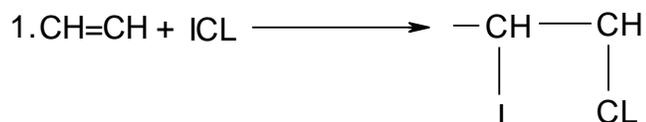
EXPERIMENT: 12

ESTIMATE THE AMOUNT OF OLEFIN

Aim: Estimate the amount of olefin (cinnamic acid) by wij's method.

Theory: When olefin is treated with an excess of wij's soln. i.e. with ICl, ICl gets absorbed due to addition of ICl to the double bonds. Unused ICl reacts with KI soln. and liberate an equivalent amt. of I₂ is titrated with standard Na₂S₂O₃ soln.

Equation:



Process: Add exactly 25ml. of wij's solution to the glass stoppered bottle that contains given olefin solution. Take 25ml. of wij's solution in another glass stoppered bottle for blank reading. Moisten the glass stoppers with 10% KI solution and stopper both the bottles. Keep them in dark for 30min. Then add one test tube of 10% KI solution and about four test tubes of water to solution in each bottle. Cork the bottles and shake them well. Titrate immediately the liberated I₂ against 0.1N Na₂S₂O₃ soln. from the burette, using starch indicator. End point will be from blue to colorless.

Results:

(1) Supplied olefin solution (i.e. consumed), (C) = ----- ml. of
wij's solution in terms of 0.1N $\text{Na}_2\text{S}_2\text{O}_3$ solution.

(2) Amount of olefin present = -----gm.

Calculations:

25 ml. of wij's soln. (blank reading) = X ml. of 0.1N $\text{Na}_2\text{S}_2\text{O}_3$ soln.

Supplied olefin soln (back titrated) = Y ml. of 0.1N $\text{Na}_2\text{S}_2\text{O}_3$ soln.

Amount of wij's soln. consumed, by olefin soln. = (X-Y) = C ml. of 0.1N $\text{Na}_2\text{S}_2\text{O}_3$ soln.

Since, $2 \text{Na}_2\text{S}_2\text{O}_3 = 2\text{I}$



1000 ml. of $\text{Na}_2\text{S}_2\text{O}_3 = 127$ Iodine

C ml. of 0.1 N $\text{Na}_2\text{S}_2\text{O}_3 = 127 \times C \times 0.1 / 1000$ gm of Iodine

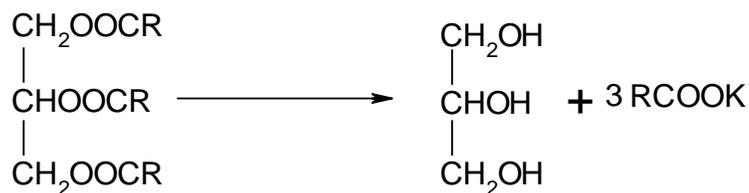
i.e. Wt. of the olefin = $127 \times C \times 0.1 / 1000$ gm of Iodine

EXPERIMENT: 13

ESTIMATE THE SAPONIFICATION VALUE OF OIL

Aim: Estimate the saponification value of oil in the given solution

Theory: Saponification value of oil is mg. of KOH required to saponify 1gm of oil. It is also called Koettstorfer number. Oil being tri-ester of glycerol, when treated with saturated solution of alcoholic KOH gets hydrolyzed or saponified into free glycerol and potassium salt of fatty acid.



Process: Part I Standardization: Take 10ml. of supplied 1N alc. KOH soln. in a standard measuring flask and dilute it to 100ml. with distilled water. Pipette out 10ml. of it in a conical flask and titrate it against 0.1N HCl soln. from the burette using phenolphthalein indicator. End point will be from pink to colorless.

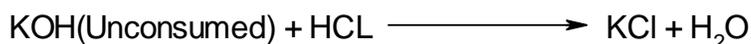
Part-II Estimation: Add 25ml of supplied (1N approximate) alcoholic KOH solution to the oil solution, given in a round bottom flask, fitted with a water condenser. Reflux the flask on a water bath for nearly 90 minutes, till the saponification is over. Cool the flask and dilute the contents of the flask to 250ml in standard measuring flask, with distilled water. Pipette out 25ml of it in a conical flask and titrate against 0.1N HCl soln. from the burette using phenolphthalein indicator. End point will be from pink to colorless.

Results:

(1) 25ml of dilute solution consumed C/10 ml i.e.ml of KOH in terms of 0.1 N HCl solution.

(2) Saponification value of the oil =mg.

Equation:



Calculations:

10 ml. of dilute KOH soln. = Xml.of 0.1N HCL soln.

100 ml. of dilute KOH soln. = 10 Xml.of 0.1N HCL soln.

Normality of KOH = $10 \times X \times 0.1 / 10 = A$

Amount of KOH added = 25ml of A N = $25 \times A / 0.1 = V$ ml.

This V ml corresponds to amount of KOH added in terms of 0.1N HCl solution.

Amount of KOH is unused in 25ml of dilute solution.(back titration reading)= y ml of 0.1N HCL solution.

250 ml = 10y ml of 0.1N HCL solution.

Amount of KOH used up for saponification of oil solution.

$$= (V-10y) = C \text{ ml}$$

Since, 1 KOH = 1 HCl

1000 ml of 1 N HCl = 56g of KOH

C ml of 0.1 N HCl = $56 \times C \times 0.1 / 1000$ g of KOH

$$= 56 \times C \times 0.1 \text{ m. g of KOH}$$

i.e. W g of the oil = $56 \times C \times 0.1$ m. g of KOH

Now the saponification value is amount of KOH required in milligrams for saponifying 1 gm of the oil. = $56 \times C \times 0.1 / W$ mg of KOH

(Where, W is the weight of the oil).

EXPERIMENT: 14

ESTIMATE THE AMOUNT OF HYDROGEN PEROXIDE

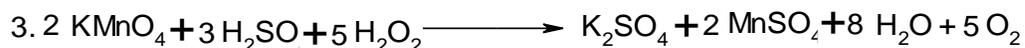
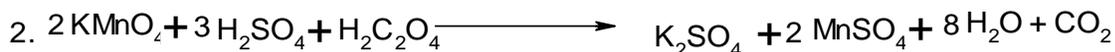
Aim: Estimate the amount of hydrogen peroxide.

Theory: H₂O₂ is an oxidizing agent. When reacts with KMnO₄ soln., it acts as an apparent reducing agent and gets oxidized to form water and oxygen. It oxidies as follow,



It can be estimated by titrating with standard KMnO₄ soln. in the presence of dilute H₂SO₄. Strength of H₂O₂ is expressed in terms of vol. of oxygen liberated.

Equation:



Process: Part I Standardization: Prepare 250ml. of 0.05N Na₂C₂O₄ soln.(0.838gm. of Na₂C₂O₄ dissolved in 250ml. of distilled water.). Pipette out 25ml. of Na₂C₂O₄ soln. in conical flask. Add 20-25 ml. of dilute H₂SO₄ to it shake it well. Heat the soln. on the wire gauze to about 70-80°C(heat carefully till vapour just coming out). Titrate hot soln. against 0.1N KMnO₄ soln. from the burette. End point will be pink color.

Part-II Estimation: Dilute the given soln. to 250ml in standard measuring flask, with distilled water. Pipette out 25ml of it in a conical flask and add about 20-25 ml. of dilute H_2SO_4 to it. Shake well. Titrate against 0.1N $KMnO_4$ soln. from the burette. End point will be pink.

10% Hydroxyaminehydrochloride

a. 25 ml. of 0.05N $Na_2C_2O_4$ required X ml. of $KMnO_4$

$$\text{Normality of } KMnO_4 = 25 * 0.05 / X = A \text{ N}$$

b. 25ml. of the diluted H_2O_2 soln. required Y ml. of A N $KMnO_4$ soln.

$$250 \text{ ml. of the diluted } H_2O_2 \text{ soln.} = 10 * Y \text{ ml.}$$

3. Now, $2H_2O_2 \longrightarrow 2H_2O + O_2$

68 gm of H_2O_2 = 32 gm. of O_2 i.e. 22.4 liters at S.T.P.

17 gm. of H_2O_2 = 8 gm. of O_2 i.e. 5.6 liters at S.T.P.

As 17 gm. of H_2O_2 is the equivalent weight of H_2O_2

1000 ml. of 1N H_2O_2 soln.

1 ml. of 1N H_2O_2 = 5.6 ml. of O_2

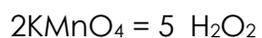
Strength of 1N H_2O_2 soln. = 5.6 vol.

4 Normality of diluted H_2O_2 soln. = $A * Y / 25 = B \text{ N}$

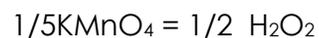
Normality of given H_2O_2 soln. = $(10 * B) \text{ N} = J \text{ N}$

Volume strength of given H_2O_2 soln. = $(J * 5.6)$

5. from equation,



gm. equivalent of KMnO_4 is



1000ml. of 1N KMnO_4 = 17gm. of H_2O_2

10Y ml. of A N KMnO_4 = $17 * 10Y * A / 1000$ gm. of H_2O_2

Results:

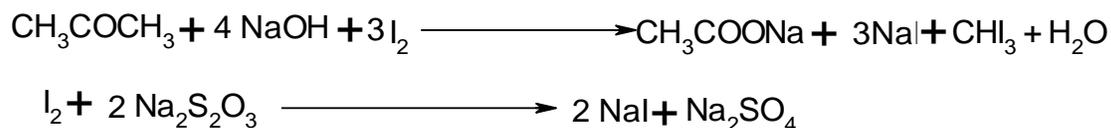
- 1) 25 ml. of dilute soln. required(Y) = ----- ml. of A N KMnO_4 soln.
- 2) Volume strength of H_2O_2 = ----- ml.
- 3) Amount of H_2O_2 present = ----- gm.

EXPERIMENT: 15
ESTIMATION OF ACETONE

Aim: Estimate the amount of acetone present in the given solution.

Theory: Acetone quantitatively reacts with I_2 in presence of NaOH and gets transfer into iodoform. Acetone is treated with known excess of I_2 soln. in presence of NaOH soln.; and the unreacted I_2 is titrated against standard $Na_2S_2O_3$ soln.

Equation:



Process:

Part-I Standardization: Take 10ml. of I_2 soln. in a conical flask and one test tube of water. Titrate immediately against 0.1N $Na_2S_2O_3$ soln. from the burette using starch as an indicator. End point will be from blue to colorless.

Part-II Estimation: Dilute the given solution to 100ml in a standard measuring flask with distilled water. Pipette out 10ml of it in a glass stoppered bottle. Add 5ml of 10% NaOH soln., shake to mix them well and exactly add 25ml. of I_2 soln. from the burette. Cork the bottle, shake it well and keep for 15min. add two test tube of dilute H_2SO_4 soln. and shake well. Titrate immediately the liberated I_2 against 0.1N $Na_2S_2O_3$ soln. from the burette, using starch indicator. End point will be from blue to colorless.

Calculations:

10ml. of I_2 soln. required Xml.of 0.1N $Na_2S_2O_3$ soln.

25ml. = $X * 25 / 10 = A$ ml.

Quantity of I_2 soln. added = A ml.

(In terms of 0.1N $Na_2S_2O_3$ soln)

Quantity of I_2 soln. unused = Y ml.

(Back titration reading from the burette)

Quantity of I_2 soln used up by 10ml. of diluted Acetone soln. = (A-Y) = C ml.

100 ml. diluted soln. used (in terms of 0.1N $Na_2S_2O_3$ soln) I_2 soln. = $10 * C$ ml.

Since 6 $Na_2S_2O_3$ soln = 1 acetone = 58 gm. of acetone

Then, 1 $Na_2S_2O_3$ soln = $1/6$ acetone = $58 / 6 = 9.667$ gm.of acetone.

1000ml. $Na_2S_2O_3$ soln = 9.667 gm.of acetone.

10 C ml. of 0.1N $Na_2S_2O_3$ soln = $9.667 * 0.1 * 10C / 1000$ gm. of Acetone.

Results:

(1). 10 ml. of diluted soln. required (C) = ----- ml. of I_2 soln. in terms of 0.1N $Na_2S_2O_3$ soln.

(2) Amount of acetone present = -----gm.

EXPERIMENT: 16

Preparation of Polystyrene & Polymethylmethacrylate

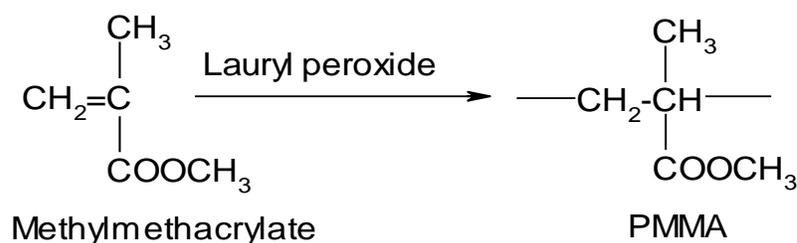
Aim: Preparation of polystyrene & polymethylmethacrylate by bulk polymerization method.

Requirements: Styrene monomer, Lauroyl peroxide, or benzoyl peroxide, Methylmethacrylate.

Theory: Styrene and Methylmethacrylate can be bulk polymerized by heating in presence of peroxide catalyst.

Bulk polymerization: A free radical catalyst or initiator is dissolved in the monomer which is then heated and stirred in a suitable vessel. The polymerization is exothermic and dissipation of heat through cooling may be required. As the reaction proceeds, the system becomes viscous making stirring difficult. The method is economical and the product is of high purity.

Equation:



Process:

(a) Polymerization of styrene: The exp. should preferably be carried out in a fume cupboard as the monomer vapour is toxic. The monomer should

preferably be freshly distilled, alternatively, make the monomer free from inhibitor by washing with it three-four times with 2% aq. NaOH, followed by two-three washes with distilled water. Add 3-4ml. styrene into a boiling tube, and add 0.02-0.03gm of Lauroyl peroxide. Allow the tube to stand in a beaker of boiling water, maintained at 100°C. Remove the test tube after one hour and allow it to cool at room temp. Pour the soln. into 200ml. of methanol containing in a beaker. Filter and wash with alcohol and dry in air.

(b) Polymerization of methylmethacrylate: Add a pinch (20-30 mg) of Lauroyl peroxide to 3-4ml of methylmethacrylate taken in a hard glass test tube and shake to dissolve. Using a clamp stand, secure the test tube in a water bath maintained at 60°C. As the polymerization progress, the liquid in the test tube slowly starts thickening and it about one hour changes to a transparent solid. Filter and wash with alcohol and dry in air.

Results:

Report the yield of polystyrene and polymethylmethacrylate.

EXPERIMENT: 17

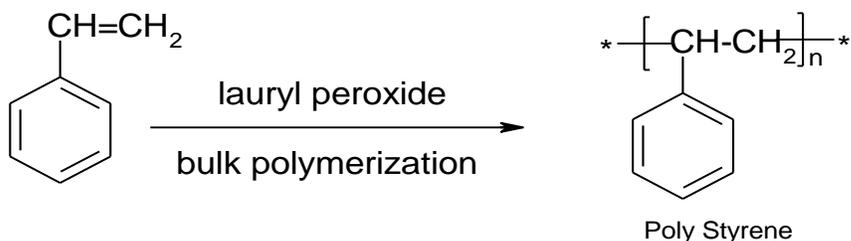
Preparation of Polystyrene

Aim: Preparation of polystyrene by suspension polymerization.

Requirements: Styrene monomer, Lauryl peroxide, or benzoyl peroxide.

Theory: In suspension polymerization a solution of the catalyst and the monomer is dispersed as fine droplets in an inert solvent usually water. To stabilize the suspension, water-soluble protective colloids such as polyvinyl alcohol, methyl cellulose, gelatin or starch are added and the mixture is kept stirring continuously. The problems of heat dissipation and viscosity increase are absent. The method gives a fairly high molecular weight product in the form of easily separable beads that can be filtered or centrifuged and water washed to remove the protective colloids. Make the monomer free from inhibitor by washing it three or four times with 2% sodium hydroxide, followed by three washes with distilled water.

Equation:



Process: In a three necked flask, equipped with a condenser and a stirrer, take 250 cc. of water. Add with stirring add 0.05 g of sodium lauryl sulphate and 0.75 g of gelatin as a suspending agent. Also add 0.75 g. of sodium sulphate. When the dissolution is completed, add 75 g of styrene in which 0.5 g of benzoyl peroxide is previously dissolved. Adjust the speed of the stirrer so that styrene is uniformly dispersed in water and the speed of the stirrer should be uniform. Heat the

reaction flask to 70-75°C on water bath or heating mantle. Throughout the process, the stirring should not be stopped.

Continue the reaction till the polymer beads become very hard, which indicates the completion of reaction. This may take about 4 hours. Then allow the reaction to cool to room temperature with stirring. Then filter the polymer beads, wash with methyl alcohol and dry at 50°C in vacuum oven.

Results:

Report the yield of polystyrene.

EXPERIMENT: 18

Preparation of Polystyrene

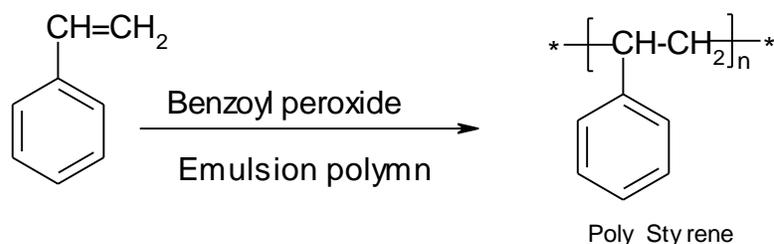
Aim: Preparation of polystyrene by emulsion polymerization.

Requirements: Styrene monomer, Lauroyl peroxide, or benzoyl peroxide.

Theory: In emulsion polymerization the practical size of the monomer is reduced to colloidal dimensions by more vigorous stirring and use of synthetic surfactants (anionic, cationic or non-ionic) in place of protective colloids used in suspension polymerization.

Usually water soluble catalysts such as persulphate or hydrogen peroxide are used; Thermal dissipation and viscosity problems are absent. Both the rate of polymerization and the molecular weight of the product formed are very high. The product, which is in the form of fine particles dispersed in water (called latex), can be used directly as adhesive or an emulsion paint, or it can be isolated by coagulating with an electrolyte.

Equation:



Process: Charge 100 ml. of water in a three necked flask and add to this 0.05 g. sodium lauryl sulphide with stirring and also add 0.05 g. potassium per sulphate. Start heating the contents on a water bath. Add 50 ml. of styrene through a separating funnel, drop wise over a period of about an hour. Raise the temperature to 90°C and keep it for four hours. Cool the resin and store it.

Results:

Report the yield of polystyrene.

EXPERIMENT: 19

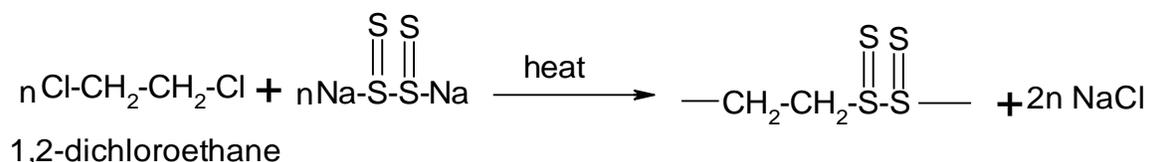
Preparation of Polysulfide rubber

Aim: To prepare Polysulfide rubber.

Requirements: Powder sulphur, NaOH, 1,2-dichloroethane.

Theory: It is a rubbery white substance and obtained by the treatment of sodium Polysulphide with 1,2-dichloroethane. Method of preparation of Thiokol rubbers involves through mixing in presence of a dispersing agent. A latex like product obtained is thoroughly washed with water.

Equation:



Process: In a 100ml. of beaker dissolve 2gm of NaOH in 50-60ml. of warm water. Boil the soln., and to this add in small amounts with constant stirring 4gm powder sulphur (if some sulphur remains undissolved filter the soln.). During the addition and stirring the yellow soln. turns deep red. Cool it to 60-70°C and add 10ml. of 1,2-dichloroethane with stirring. Stir for an additional period of 20min. while rubber polymer separates out as a lump. Pour out the liquid from beaker in the sink to obtain Thiokol rubber. Wash it with water under the tap. Dry in the fold of filter paper.

Results:

Report the yield of Polysulphide rubber.

EXPERIMENT: 20

DENSITY MEASUREMENT BY DISPLACEMENT METHOD

Aim: To find the density of the given plastic material having density more than water.

Requirements: Weighing balance, beaker, plastic specimen, nylon thread having negligible wt.

Theory: Density is the ratio of mass per unit volume at a specific temp. (23^o, 25^o & 27^oC).

Unit- gm/cm³, kg/m³.

Weight of specimen in air

Density = ----- * Density of water.

Loss in weight of specimen in water.

Process: Weighing balance is adjusted. Nylon thread is tied around the specimen and the specimen is suspended on the left hand side hook of the balance. Gradually weights are added or removed from the right side plate till the pointer of the balance is in the centre of scale. Note the weight. Fill a beaker with sufficient quantity of water. A wooden block is placed above the left side of pan without resting on the balance. The beaker with water is placed on it. The sample is attached to the left hand side hook and is gradually immersed in water. The specimen should not touch the side and bottom surface of the beaker. It should remain perfectly suspended in water. Weights are added and removed such that the pointer lies in the centre of the scale. Note down this weight.

Observations:

- 1) Weight of specimen in air (W_1) = -----
- 2) Weight of specimen in water (W_2) = -----
- 3) Density of water (H_2O) = 1 gm/cm^3

Calculations:

$$\text{Density of specimen} = \frac{\text{Weight of specimen of air}}{\text{Loss of wt. specimen in water}} * \text{density of water}$$

Loss in weight of specimen = Weight of specimen in air - Weight of specimen in water

$$= W_1 - W_2$$

$$= \frac{W_1}{W_1 - W_2} * \text{density of water}$$

Result:

The density of the given plastic specimen by displacement method = -----

EXPERIMENT: 21

DENSITY MEASUREMENT USING BUTYL ACETATE

Aim: To find the density of the given plastic material is having density less than water using butyl acetate.

Requirements: Weighing balance, beaker, plastic specimen, nylon thread having negligible wt.

Theory: Density is the ratio of mass per unit volume at a specific temp. (23^o, 25^o & 27^oC).

Unit- gm/cm³, kg/m³.

$$\rho = \frac{W_1}{W_1 - W_2} * \rho_1$$

Where;

ρ = Density of plastic specimen

W_1 = Wt. of specimen in air

ρ_1 = Density of butyl acetate

W_2 = Wt. of specimen in butyl acetate

Process: Weighing balance is adjusted. Nylon thread is tied around the specimen and the specimen is suspended on the left hand side hook of the balance. Gradually weights are added or remove from the right side plate till the pointer of the balance is in the centre of scale. Note the weight. Fill a beaker with sufficient quantity of butyl acetate. Place the beaker on a wooden stand above the left hand pan. Attach the sample to the left hand side hook and slowly immerse it in butyl acetate. Check that the specimen should not touch the side and bottom of the beaker. It should remain perfectly suspended in butyl acetate. Weight the specimen. Note down the reading.

Observations:

- 1) Weight of specimen in air (W_1) = -----
- 2) Weight of specimen in butyl acetate (W_2) = -----
- 3) Density of butyl acetate ρ_1 = -----
- 4) Density of specimen ρ = -----

Calculations:

$$\rho = \frac{W_1}{W_1 - W_2} * \rho_1$$

Result:

The density of the given plastic specimen = -----

EXPERIMENT: 22

DENSITY MEASUREMENT BY SINKER METHOD

Aim: To find out the density of the specimen lighter than water by Sinker Method.

Requirements: Weighing balance, beaker, plastic specimen, sinker, and nylon thread of negligible weight.

Theory: Density is the ratio of mass per unit volume at a specific temp. (23^o, 25^o & 27^oC).

Unit- gm/cm³, kg/m³.

$$\rho = \frac{W_1}{W_1 + W_2 - W_3} * \rho_1$$

Where;

ρ = Density of plastic specimen

W_1 = Wt. of specimen in air

ρ_1 = Density of water

W_2 = Wt. of sinker in water

W_3 = Wt. of specimen + sinker

Process: Weighing balance is adjusted. Nylon threads are tied around the specimen and weight it. Note the readings. Fill a beaker with sufficient quantity of water and place it on a wooden stand above the left hand pan. Tie a nylon thread around the sinker and suspend it fully in water. Check that the sinker should not touch the side and bottom of the beaker. Weight the sinker and note the reading. Tie the sinker and the specimen together with a nylon thread and weigh it after immersing in water. The specimen and the sinker should not touch the sides or the bottom of the beaker. Note down the readings.

Observations:

- 1) Weight of specimen in air (W_1) = -----
- 2) Weight of specimen in butyl acetate (W_2) = -----
- 3) Weight of sinker in water (W_3) = -----
- 4) Density of specimen $\rho =$ -----

Calculations:

$$\rho = \frac{W_1}{\frac{W_1 + W_2 - W_3}{\rho_1}}$$

Result:

The density of the given plastic specimen by sinker method = -----

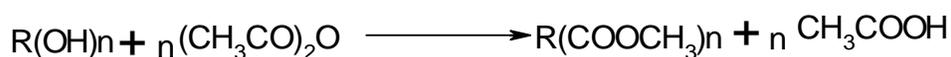
EXPERIMENT: 23

ESTIMATION OF ALCOHOLIC -OH GROUP

Aim: To determine % age of - OH group in the given sample of monosaccharide.

Apparatus : Iodine flask, pipette, burette, steam-bath, acetylating reagents, 0.5 M alcoholic NaOH soln. mixed indicator (1 part of 0.1% cresol red + 3 part of 0.1% Thymol blue).

Equation:



Process: Weight accurately 1 gms. Of the given sample of glucose. Transfer it into iodine flask. Add exactly 3 ml. of acetylating reagent. Moisture glass stopper with pyridine and seal it loosely. Heat the flask on a steam bath for 1 hrs. Add 5 ml. of water to the cap around the stopper and wall of the flask. Shake the content of the flask to ensure through mixing. Cool the flask under tap water and add 10 ml. of n-butanol soln. then add 2-3 drops of mixed indicator and titrate the content of the flask against std 0.5M alcoholic NaOH soln. from the burette till the end point reaches from green to gray violet. Carry out blank titration omitting sample.

Result:

%age of -OH group in the given sample of glucose = ----- %

Observations:

1) Wt. of glucose sample taken = ----- gms.

Calculations:

$$\% \text{ of } -\text{OH group} = \frac{(\text{Blank B.R.} - \text{sample B.R.}) * \text{Molarity of NaOH soln.} * 17.1 * 100}{W * 1000}$$

Where, W = Wt. of sample taken.