

GUJARAT TECHNOLOGICAL UNIVERSITY

BRIDGE COURSE

CHEMISTRY

B.E. 1st YEAR (w.e.f July 2014)

CONTENTS:

Sr. No.	Subject Content
1.	A Visit to Chemistry Laboratory
2.	Identification of Glass ware in Chemistry Laboratory
3.	List of Chemicals available in the Laboratory
4.	To prepare a sample of Colloid (Cream)
5.	To prepare a sample of suspension
6.	Identification of Elements in organic compounds
7.	Purification Methods
8.	Concepts of Normality, Molarity and Standardization
9.	Preparation of phenol formaldehyde resin
10.	Determination of Saponification value of oil
11.	Basics concepts of Chromatographic Technique

Bridge Course duration : 04 Weeks

Evaluation of Bridge course

Weekly group discussion, poster preparation, Quiz and activity etc. MCQ test at the end of the course .

Rubrics for the evaluation :

Sr. No.	Evaluation Criterion	Marks
01.	Involvement of student during activities.	10
02.	Communication and interaction of students among the groups.	10
03.	Presentation skills	10
04.	Critical Thinking	10
05.	Team work	10
	Total	50

Learning Resources :-

Course Material :-

- Presentations, Video and Course planning are provided.

Books:-

1. How things works by Louis A Bloomfeild, Wiley Publications
2. Chemistry by Raymond & Chang (Tata Macgrow Hill Pub.)
3. Vogels analytical chemistry (Organic and Inorganic)

Useful sites :-

1. <http://www.oecd.org/chemicalsafety/testing/goodlaboratorypracticeglp.htm>
2. <http://www.rsc.org/Education/EiC/issues/2008Mar/GoodLabPractice.asp>
3. <http://chemistry.about.com/od/labtechniques/>
4. <http://www.chem.wisc.edu/areas/organic/orglab/techniques.htm>

Suggested Activities:**Activity – 01****A Visit to Chemistry Laboratory**

Students will be oriented to the Chemistry Laboratory and in which the following observation is to be made:

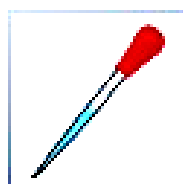
- 1 Ventilation provided in the laboratory along with fire extinguisher.
- 2 Placing of bench reagents, Stock solution, etc.
- 3 Placing of Glass-ware on the racks.
- 4 Placing of Chemicals (Solid and liquid) on the racks.
- 5 Display of Notice Board, Periodic Chart (Periodic Table), radical Table, etc.
- 6 Sink fitted with swan necked tap and proper plumbing system.
- 7 Gas connections attached to Bunsen burner.

Students will have to put on White Aprons during practical performance.

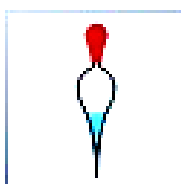
Remarks:

Further observations can be added to this activity.

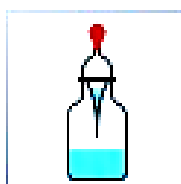
Activity – 02**Identification of Glass ware in Chemistry Laboratory**



Dropper



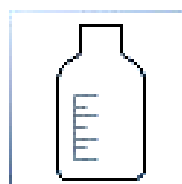
Dropper



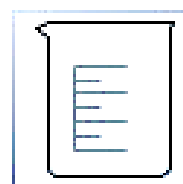
Dropping
Liquid



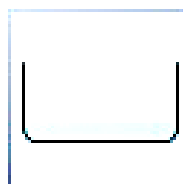
Gas Jar



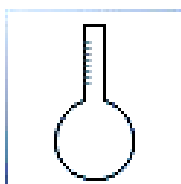
Gas Jar



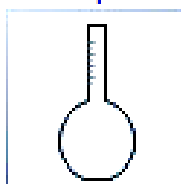
Beaker



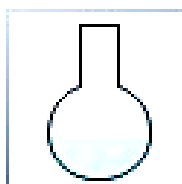
Trough



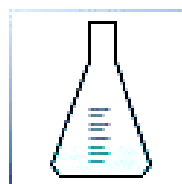
Flask



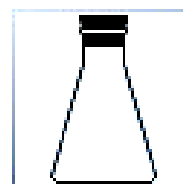
Flat
Bottomed



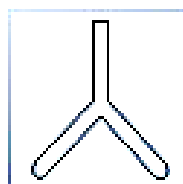
Flask



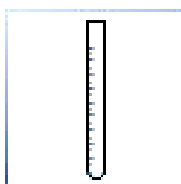
Conical
Flask



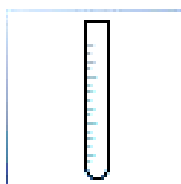
Conical
Flask 2



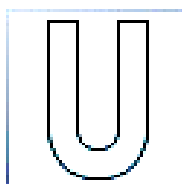
Y Tube



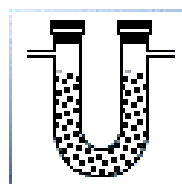
Small Test
Tube



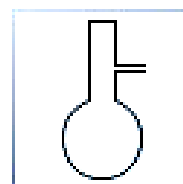
Test Tube



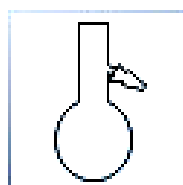
U Tube



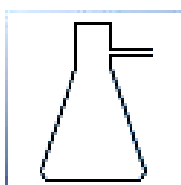
U Tube



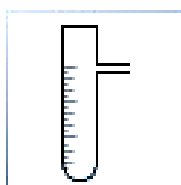
Pear-Shap...
Flask



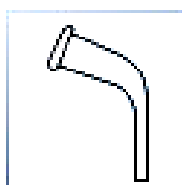
Pear-Shap...
Flask



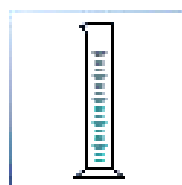
Suction
Flask



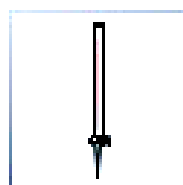
Canula



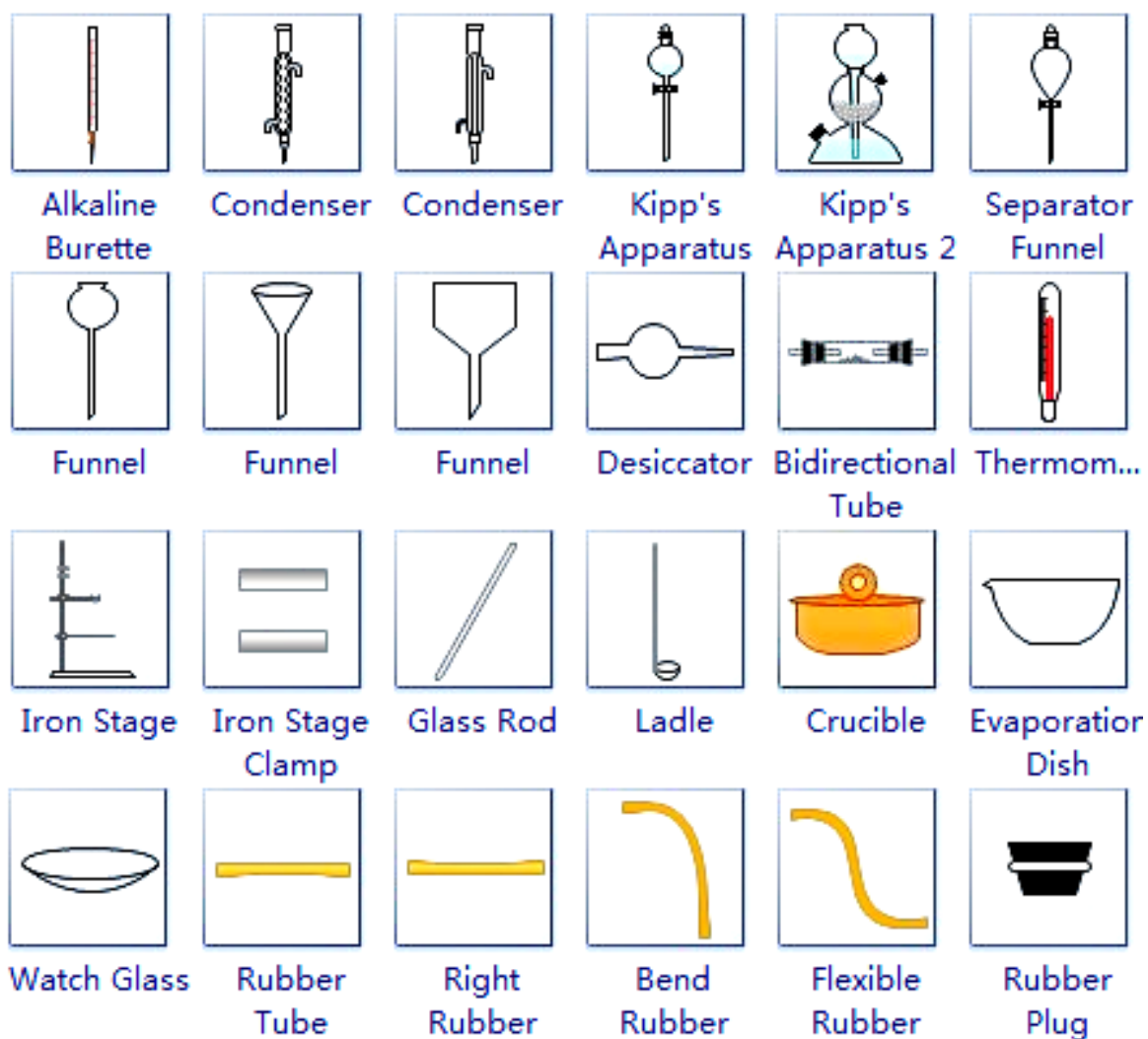
Liebig
Condenser



Measuring
Cylinder



Acid Burette



Bunsen Burner, Swan necked tap, laboratory balance and heating mentle.





Activity – 03

List of Chemicals available in the Laboratory, put ✓ for the chemical available or else ✗.

Name of the Chemical	(✓/✗)	Name of the Chemical	(✓/✗)
ACETALDEHYDE		<i>n</i> -HEXANE	
ACETAMIDE		NINHYDRIN SOL	
ACETANILIDE		NITROBENZENE	
ACETIC ACID		<i>o</i> -TOLUIDINE	
ACETIC ANHYDRIDE		OXALIC ACID	
ACETONE		<i>p</i> -AMINOPHENOL	
ALUMINIUM		<i>para</i> -AMINOPHENOL	
AMMONIUM CARBONATE		PARA-DAB	
AMMONIUM PERPURATE		PETROLIUM ETHER	
AMMONIUM SULPHATE		PHENOL	
AMMONIUM CHLORIDE		PHENOPHTHALENE	
ANTHRACENE		PHOSPHORIC ACID	
BaCl ₂		PHTHALIC ANHYDRIDE	
BENZALDEHYDE		PICRIC ACID	
BENZAMIDE		PINE OIL	
BENZENE		POT FERROCYNIDE	
BENZENE SULPHONYL CHLORIDE		POT. CHROMATE	
BENZOIC ACID		POT. DICHROMATE	
B-NAPHTHOL		POT. SULPHATE	
Br ₂ WATER		PYRIDINE	

BROMOTHYMOL		SALICYLIC ACID	
BUTYL ACETATE		SCHIFF`S REAGENT	
BUTYL ALCOHOL		SILVER NITRATE	
CaCl ₂		SnO ₂	
CAL. CARBONATE		SOD. CARBONATE	
CERIC AMMONIUM NITRATE		SOD. HYPOCHLORITE	
CHARCOAL		SOD. NITROPRUSSIDE	
KOH		SOD. SILICATE	
LEAD ACETATE		SOD.BICARBONATE	
LEAD CHROMATE		SODALIME	
LIME WATER		TANNIC ACID	
LITMUS SOL		TIN	
MALACHITE GREEN		TiO ₂	
METHANOL		TOLLENS REAGENT	
METHYL ORANGE		TOLUENE	
METHYL RED & BLUE		UREA	
MnCl ₂		ZINC	
<i>m</i> -NITROBENZENE		ZINC OXIDE	
MOHR`S SALT		ZnCl ₂	
Na ₂ S ₂ O ₃			
NaCl			

Activity 04

To prepare a sample of Colloid (Cream).

Apparatus: - Beaker, glass rod, china dish etc.

Chemicals: - Stearic acid, Potassium hydroxide, methanol, Piperment oil etc.

Procedure: -

- Dissolve 2 gm Potassium hydroxide in about 50 ml of water. Take 5 gm of Stearic acid and Melt it in a big china dish and add Potassium hydroxide solution to it. Stop heating the dish and stir it well.
- Dissolve 0.5 ml of Piperment oil in 3 ml of methanol and add this mixture to the china dish containing Potassium hydroxide and Stearic acid when cold.
- The cream obtained can be stored in to the bottle.

Result: - Weight of the Cream is _____ gm.

Explain surface chemistry along with dispersion, it's classification in detail.

Activity – 05

To prepare a sample of suspension.

Apparatus: - Pestle & Mortar, Beaker, measuring cylinder, test tube etc.

Chemicals: - Gum acacia, Kaoline light, Piperment oil etc.

Procedure: -In a clean dry mortar take 5 gm of gum acacia and 3 gm of kaoline light.

Now, we prepare solution of Piperment oil mixture :

- In one liter of water take 0.5 ml of Piperment oil with constant agitation. Take 90 ml of solution and start the procedure.
- In mortar take 4 to 5 ml of this peppermint solution by constant trituration.
- Now, slowly add this peppermint solution till paste is formed, transfer it into the clean beaker slowly rinse the mortar with the mixture and mix it into beaker. So, suspension is formed.

Conclusion :-Transfer about 10 ml of the content of beaker into test tube and observed it, tally with its property.

To write a note on suspension and its properties and applications.

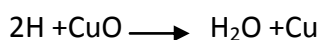
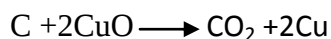
Activity – 06

Identification of elements in organic compound

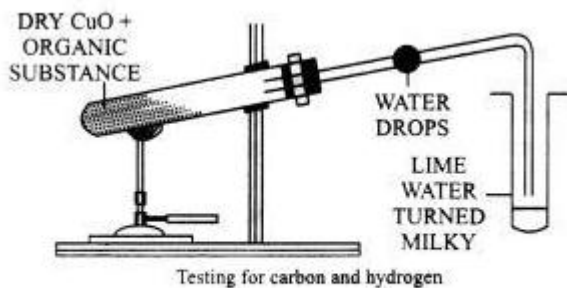
Element	Order of abundance
Carbon	Always present
Hydrogen	Nearly always present
Oxygen	Generally present
Nitrogen, halogens and sulphur	Less commonly present
Phosphorus and metals	Rarely present

1. Detection of Carbon and Hydrogen: If the compound under investigation is organic then there is no need. These elements are confirmed by the following common tests.

The organic substance is mixed with about three times its weight of dry copper oxide. The mixture is then placed in a hard glass test tube fitted with bent delivery tube. The other end of this tube is dipped into lime water in another test tube. On heating the mixture strongly the following reactions take place:



Thus, if carbon is present it is oxidized to carbon-dioxide and turns lime water milky. If hydrogen is also present, it will be oxidized to water which condenses in small droplets on the cooler walls of the test tube and inside the bulb, the formation of water can be further confirmed with anhydrous copper sulphate (white) which turns blue.



Note 1: If the substance is a gas or volatile liquid, then the vapours of the substance are passed over heated copper oxide contained in a hard glass combustion tube and the gases coming out of the tube are tested for water and CO₂ as described above.

Note 2: While testing for hydrogen, it is necessary that the apparatus, copper oxide and the substance are absolutely dry. Cupric oxide being hygroscopic in nature is strongly heated just before use.

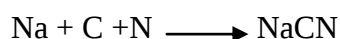
2. Detection of Oxygen: There is no direct test for detection of oxygen and its presence in organic compounds is often found by indirect methods.

(1) A method is to test for the presence of various oxygen containing groups such as carboxyl (—COOH), hydroxyl (—OH), nitro, aldehyde (—CHO); ketone (>CO); ester (—COOR) etc. If any of these groups is detected, the presence of oxygen is confirmed.

(2) The definite test for oxygen is to determine the percentage of all other elements present in the given compound. If the total of these percentages falls short of hundred, the remainder is the percentage of oxygen.

3. Detection of Nitrogen

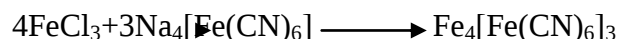
(a) **Lessaigne's test:** This is the test for nitrogen. Take a freshly cut piece of sodium in an ignition tube and cover it with a small quantity of the given substance. Hold the tube in tongs and heat it gently to melt the contents so that they may react and form sodium cyanide.



Now heat the tube strongly till the reaction ceases and the tube becomes red hot. Plunge it into about 10 mL of distilled water. Boil the contents and filter. The filtrate is called '**Sodium**

Extract' and contains nitrogen as sodium cyanide. Take a portion of this sodium extract and add two drops of caustic soda solution to make it alkaline. Then add 2 drops of freshly prepared ferrous sulphate solution and heat to boiling and cool. The sodium cyanide thus changes into sodium ferrocyanide.

To the cooled solution then add a little ferric chloride solution and excess of hydrochloric acid. A blue or green colouration due to formation of ferric ferrocyanide confirms the presence of nitrogen.



In case, sulphur is also present along with nitrogen; sodium sulphocyanide is formed.

This gives a blood red colouration with ferric chloride due to the formation of ferric sulphocyanide.



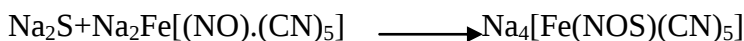
(b) **Soda lime test:** A small number of nitrogenous compounds respond to this test. The substance is mixed with double the amount of soda lime (NaOH + CaO) and heated in a test tube. If ammonia is evolved, the presence of nitrogen in the substance is proved. A negative result, however, does not prove the absence of nitrogen. Nitro and diazo compounds do not respond to this test.

(c) **Evolution of nitrogen test:** Substances, such as the diazo compounds, lose nitrogen at moderate temperatures and can not be tested by any of the above two tests. In such a case the substance is heated with cupric oxide in a tube filled with carbon dioxide and the evolved gas is led into a tube filled with potassium hydroxide solution. If nitrogen is present the evolved gases are not completely absorbed by potassium hydroxide and some gas collects over potassium hydroxide solution.

4. Detection of Sulphur

(a) **Lassaigne's test:** Prepare the sodium extract of the given organic compound as in the detection of nitrogen. Sulphur, if present, reacts with sodium to form sodium sulphide which dissolves in water. Sulphur in this extract may be tested as below:

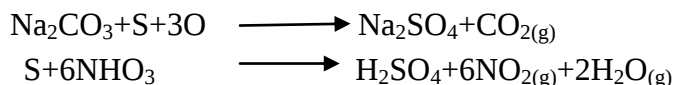
(i) To a portion of the sodium extract, add freshly prepared sodium nitroprusside solution. A deep violet colouration indicates the presence of sulphur.



(ii) Acidify a second portion of the extract with acetic acid and add lead acetate solution. A black precipitate of lead sulphide confirms the presence of sulphur.



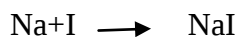
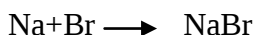
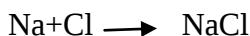
(b) **Oxidation test:** Easily volatile substances usually do not respond to Lassaigne's test. In such a case the compound is fused with a mixture of sodium carbonate and potassium nitrate, or heated with fuming nitric acid in a 'bomb tube' (see estimation of sulphur). Sulphur, if present, is oxidised to sulphate.



The heated mass is added to water, boiled, acidified with hydrochloric acid and then barium chloride solution is added to it. A white precipitate indicates the presence of sulphur.

5. Detection of Halogens

(a) **Lassaigne's test:** Prepare the sodium extract of the compound as in the detection of nitrogen. Halogens, if present, are converted to the corresponding sodium halides.



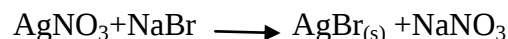
These salts are present in the sodium extract. A portion of the sodium extract is boiled with dilute nitric acid to decompose any cyanide or sulphide, and then cooled silver nitrate is added and following observations are recorded:

***Note:** If cyanide and sulphide are not removed before adding silver nitrate solution, these radicals will combine with silver nitrate and produce white and black precipitate of AgCN & Ag_2S respectively.

(i) A white precipitate readily soluble in ammonium hydroxide indicates **Chlorine**.



(ii) A yellowish precipitate soluble in a large excess of ammonium hydroxide indicates **Bromine**.



(iii) A yellow precipitate insoluble in ammonium hydroxide indicates **Iodine**.



(b) **Beilstein test:** A clean copper wire flattened at one end is heated in the oxidising flame of a Bunsen burner till it ceases to impart green colour to the flame. It is now taken out of the flame and a small quantity of the substance is put on the flattened end. The wire is reinserted in the flame. The halogen reacts with the copper to form a volatile copper halide which again imparts a blue or green colour to the flame.

This test, though very sensitive is not always reliable. It is also given by certain compounds which do not contain halogen, e.g., urea forms volatile cupric cyanide and gives this test.

6. Detection of Phosphorus: The compound is heated with fuming nitric acid under pressure, sodium peroxide or a mixture of sodium carbonate and sodium nitrate. The phosphorus is thus oxidized to phosphoric acid or sodium phosphate, which is then heated with nitric acid and ammonium molybdate. A yellow precipitate or colouration indicates the presence of phosphorus.

Activity-07

Purification Methods

- (1) Crystallization
- (2) Fractional crystallization.
- (3) Sublimation.
- (4) Distillation at atmospheric pressure.
- (5) Distillation under reduced pressure.
- (6) Steam distillation.
- (7) Fractional distillation with or without the use of a fractionating column.
- (8) Chemical methods of purification.

1. Crystallization: Most of the solids are purified by crystallization. The technique employed is the same as employed for inorganic solids, except that in addition to water, organic solvents like alcohol, ether, chloroform, benzene etc. are frequently used because the organic compounds are more readily soluble in organic solvents. This is based on the principle — **Similia Similibus**

Solventer” i.e., like dissolved by like. The process involves the following steps.

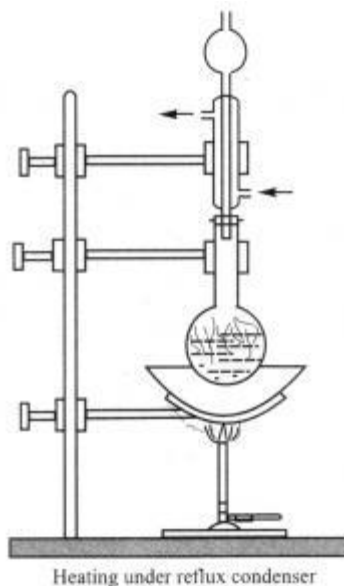
(i) Selection of the proper solvent: A proper solvent is that which (a) does not dissolve the impurities at all or dissolves them to a much larger extent so that either the impurity of the product remains in the mother liquor after separation of the solids, (b) the solvent selected should be such that it dissolves the maximum quantity of the solute when hot and throws out the maximum quantity when cooled.

The common solvents in use for this purpose are petroleum ether (323-333k); methyl alcohol (338K); acetone (329K); chloroform (334.2K); carbon tetrachloride (349—354K); ethyl acetate (350K); benzene (353.4k); toluene (383K) etc.

To select the proper solvent, small amounts (a few milligrams) of the substance are put into a number of small test tubes and treated with a small quantity of the common solvents. The suitable solvent will be that in which the substance dissolves on heating and from which it readily crystallizes out on cooling.

After selecting out the proper solvent, we must find out the amount of it to be used. Large excess of the solvent should be avoided otherwise a sufficient amount of the substance will remain dissolved in the mother liquor.

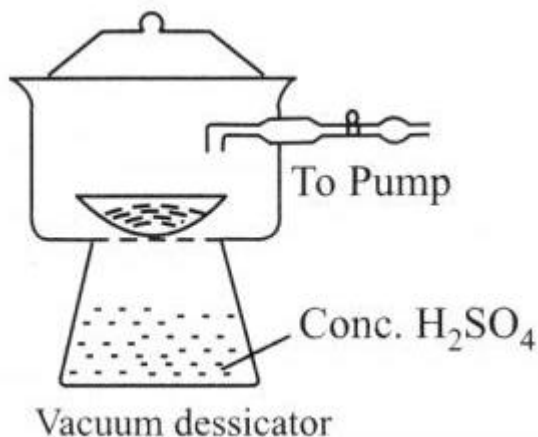
(ii) Preparation of the solution: First of all the crude material is well powdered and a suitable amount is taken in a round bottom flask. A small amount of solvent is added and the flask heated on a water bath using a reflux condenser. If the substance does not dissolve completely.



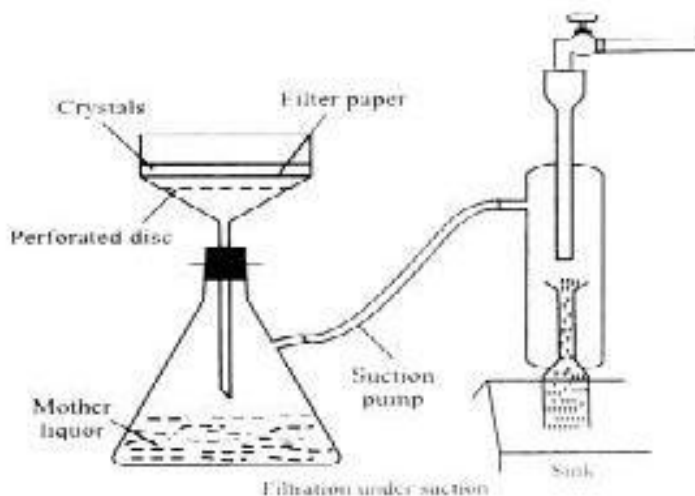
Preparation of the solution

then some more solvent is added. In case water is used as a solvent, the flask can be heated directly over the flame without using the reflux condenser. Sometimes only an air condenser is used.

(iii) Filtration: The hot solution prepared as above is then filtered through a funnel fitted with an ordinary filter paper. Occasionally hot water funnel is used to prevent undue cooling during filtration. Otherwise crystallization will start on the filter paper and choke it. It happens particularly when large volumes have to be filtered. The hot water funnel is an ordinary funnel surrounded by a circular double walled metal jacket with a side tube and filled with water, which is kept hot by a burner.

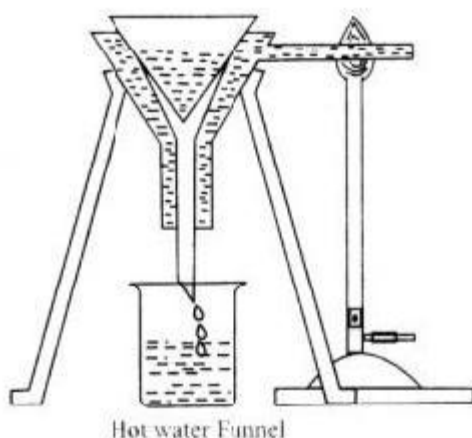


(iv) Crystallization: The hot filtrate obtained above is allowed to cool. For getting pure and smaller crystals the



filtrate should be cooled rapidly. If larger crystals are required, the cooling is done slowly. But such crystals are not very pure as they may entrap minute amounts of impurities during their formation. The cooling should be continued until complete separation of the crystals has taken place. The soluble impurities remain in the mother liquor and do not come out with crystals.

(v) Separation and drying of crystals: The crystals together with the mother liquor are transferred to a buchner funnel fitted on a suction flask which is connected to a water suction pump. The funnel is previously fitted with a filter paper. The suction pump is started. The mother liquor passes down into the filtration flask quickly due to reduced pressure inside the flask. The crystals remain on the filter paper. They are washed two or three times with a small quantity of pure solvent to remove any adhering impurities. The suction is continued so as to dry the crystals as far as possible.



Separation and drying of crystals

For further drying the crystals are first pressed between two pads of filter paper and then dried in air or in a temperature controlled oven depending upon the nature of the substance. Sometimes the crystals are dried in a vacuum desiccator containing concentrated sulphuric acid or anhydrous calcium chloride. Sometimes it is convenient to make use of a centrifugal machine. In industries the centrifugal machine is very commonly used. If crystals obtained are somewhat coloured due to the presence of trace of impurities, they are again dissolved in the minimum quantity of the solvent, a little of animal charcoal is added, the suspension is boiled for a few minutes, filtered and crystallized as before. Crystallization is repeated until the solid has the same melting point on two successive operations.

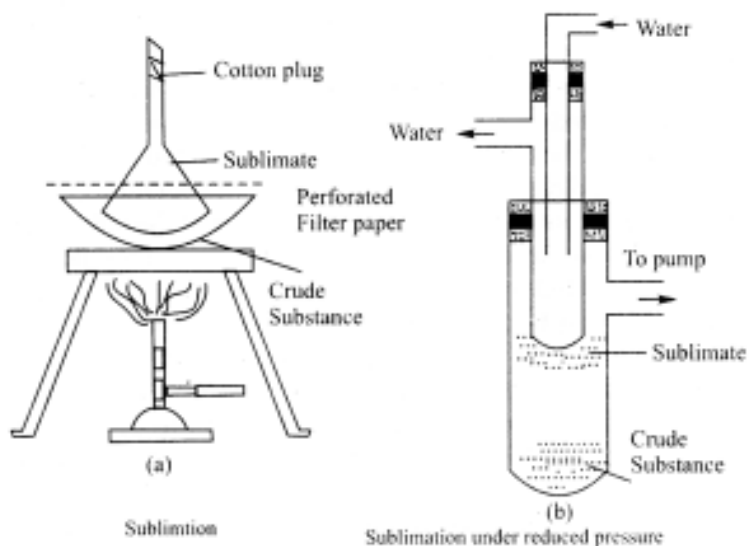
2. Fractional Crystallization: The method of simple crystallization is suitable for the purification of a single substance, contaminated with small amount of impurities. But when two (or more) substances (both of which are soluble in the solvent used), are to be separated, the technique of fractional crystallization is employed. The hot solution of the two or more substances in a suitable solvent is cooled slowly.

The constituent which has a lower solubility crystallizes out first at a certain temperature. It should be filtered at this temperature. The other constituent will crystallize out on further cooling. The

process is to be repeated several times for complete separation. The separation becomes more and more difficult when the solubility of the constituents are nearly equal.

3. Sublimation: Certain substances when heated, first directly from the solid to the vapour state without melting.

The vapour when cooled, give back the solid substance. This process is known as Sublimation which is very helpful in separating a volatile solid from a non-volatile solid. It is, however, of limited importance as only a few substances such as naphthalene, camphor, benzoic acid etc. can be purified by this method.

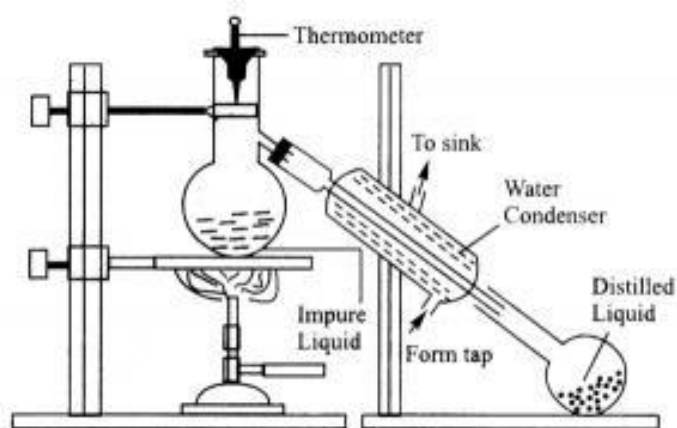


The method consists in placing the impure material in a dish or big watch glass and covering this with an inverted funnel, the stem of which has been plugged with cotton. The substance is kept covered by a perforated filter paper which checks the sublimate from falling back into the dish. The dish or the water glass is heated very slowly on a sand bath with a free flame.

The upper edges of the funnel may be kept cooled by covering it with layers of wet filter papers. The solid vaporizes and condenses on the walls of the funnel, leaving the non-volatile impurities in the dish.

The above procedure of sublimation is a crude one. Several more refined apparatus are available for this purpose. The one designed by Bruhl is very efficient. For substances which decompose under ordinary conditions of heating, vacuum sublimation is used. The substance in this case is heated under vacuum.

4. Distillation at Atmospheric Pressure: The process of distillation is employed for the purification of liquids from non-volatile impurities.



The impure liquid is boiled in a flask and the vapours thus obtained are collected and condensed in another vessel called receiver. The non-volatile impurities are left behind in the first flask. The apparatus used for simple distillation is shown above.

The impure liquid is taken in a round bottom distillation flask connected to a water condenser. (An air condenser can be used if the boiling point of the liquid is above 423K). The flask is

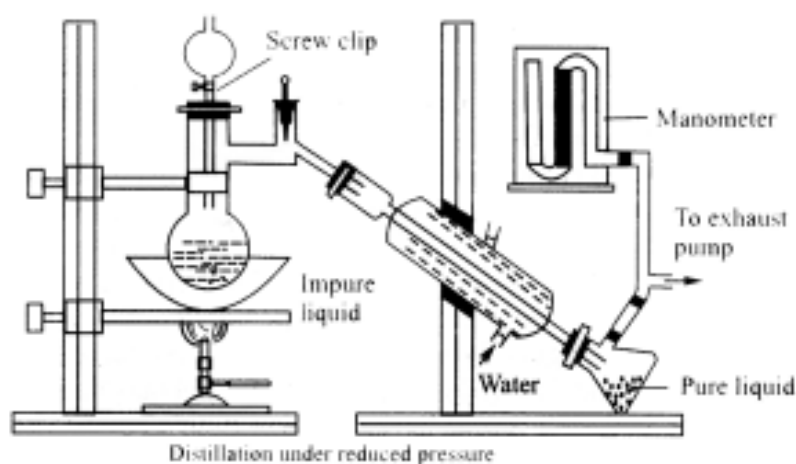
heated on a water bath or sand bath depending upon the boiling point of the liquid. The vapours of the substance rise and come into the condenser tube where they liquify and are collected in a receiver placed at the lower end of the condenser.

It is better to protect the receiver from the flame especially in case of low boiling point liquids. The impurities remain behind in the distillation flask. It is desirable to add a few glass beads, pumice stone or porcelain pieces to avoid bumping or super heating of the liquid in the distillation flask.

5. Distillation under Reduced Pressure: The process of distillation is suitable only for those liquids which boil without decomposition at atmospheric pressure.

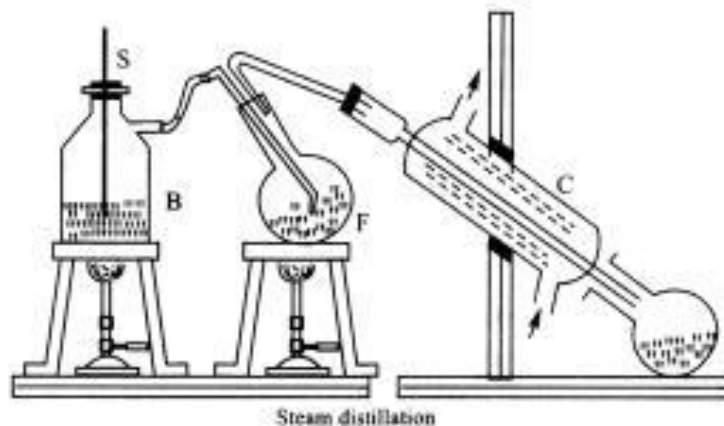
Organic liquids which decompose before their boiling point, distillation is carried out under reduced pressure when the liquid boils at a lower temperature. A liquid boils at a temperature when its vapour pressure becomes equal to the atmospheric pressure.

The apparatus used for this purpose is shown in the figure. To avoid increased chances of bumping and superheating a Claisen flask fitted with a capillary tube is used. The capillary is kept immersed in the liquid and by using a pressure tubing screw clip arrangement air is regulated through it, which eliminates bumping, or superheating.



The pressure inside the apparatus is reduced by using a filter pump, or for lower pressures, a vacuum pump. A mercury manometer is also used to indicate the pressure. Thus glycerin, which has a normal b.p. of 663K; can be distilled at 553K without decomposition at 12 mm pressure.

6. Steam Distillation: Many substances that are insoluble in water and are volatile in steam are purified by distillation in a current of steam and this process is known as **Steam Distillation**. In this method thenon-volatile impurities are left in the distillation flask. The substance to be purified is placed in a large round bottom flask, clamped at an angle, so as to prevent the solution being thrown into the condenser. The steam is generated in the steam generator and is bubbled through the impure substance by means of a tube.



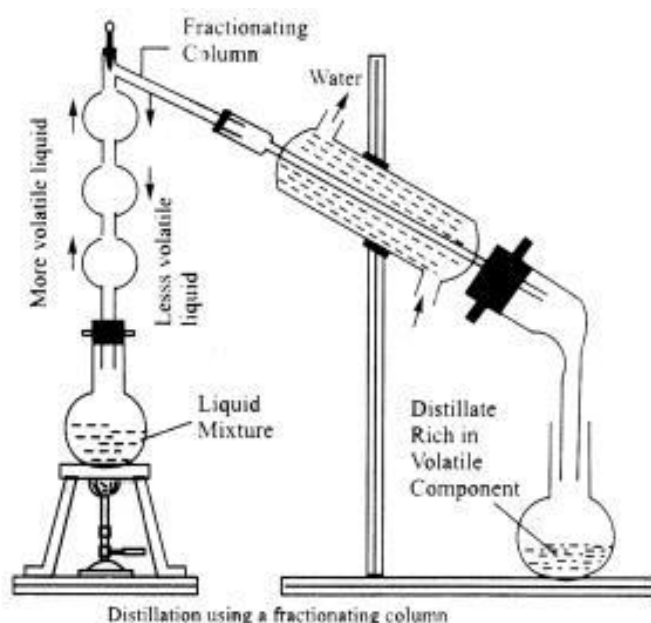
The flask containing the substance is also heated gently on a sand bath in order to avoid too much condensation of steam in it. The liquid in the flask soon begins to boil and mixed vapours of the compound and steam pass over into the condenser and the condensed liquid is received in the receiver.

The distillation should be continued for about 15 minutes after oily drops or solid particles cease to appear in the condenser. In case of a soluble acid the distillation should be stopped when the distillate no longer gives an acid reaction to litmus.

The distillate in the receiver consists of water and the purified substance. In case the substance in an oily liquid of aniline, it is separated from water by means of a separating funnel. If it is an insoluble solid it is separated by filtration. If the substance is soluble in acid, the solution is exactly neutralized with Na_2CO_3 and the solution evaporated to dryness.

From the pure sodium salt the acid is recovered by distilling with sulphuric acid. Essential oils, petroleum, turpentine oil, para separating funnel dichlorobenzene etc. are purified by steam distillation.

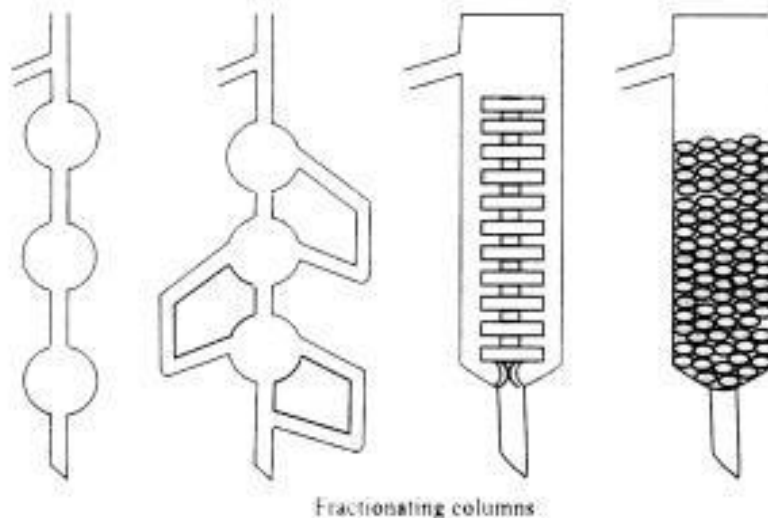
7. Fractional Distillation: A mixture of two or more volatile liquids can be separated by fractional distillation. If the boiling points differ by more than 40°C , the operation can be carried out with the help of ordinary distillation apparatus as discussed in distillation at atmospheric pressure. The more volatile over first and is collected in a receiver. Now temperature is again raised and the first receiver is disconnected and a new receiver is attached. Thus the distillate is collected in fractions and hence this process is known as **Fractional Distillation**.



But when the boiling points of the constituents are near, such as acetone (b.p. 329 K) and methyl alcohol (b.p. 338 K), they will not separate with the simple distillation apparatus. The apparatus is slightly modified. Instead of using a flask with a side tube, a simple round flask is used and the same is fitted with a suitable type of fractionating column.

The condenser is attached to the side tube of the fractionating column. Several designs of fractionating columns are in use. The real purpose of a fractionating column is to increase the cooling surface and to provide obstruction to the passage of ascending vapour or descending liquid.

In industrial processes, much longer and efficient columns are used. As the mixture containing the liquids A and B boiling say at 323 K and 333K respectively, is heated in this apparatus,



the vapours consisting more of A than B rise up. On coming into contact with the large cooling surface of the fractionating column, the vapours of B condense more than those A because B is less volatile. As the condensate consisting mostly of the less volatile component B flows down the fractionating column, it meets the fresh hot ascending vapours.

During this process, the ascending vapours are deprived of the less volatile component B which condenses and in exchange the down coming condensate loses its more volatile A which joins the upward going vapours is now much enriched in A.

This exchange of vapours is repeated throughout the length of the column; with the result that by the time the vapours reach the top of the column, they consist mainly of A while the down coming liquid mostly of B. In this way an almost complete separation is achieved. If one fractionation is not sufficient, then it may be repeated for complete separation.

8. Chemical Methods of Purification: The physical methods of purifying and separating organic compounds, described in the above sections are of general application and of great value in separating substances that are chemically similar. On the other hand many chemical methods are also employed in case of mixture of substances that are chemically different. The exact details of the method employed have been discussed with the individual compounds. Only examples of a few cases have been given below:

(i) In purification of petroleum and coal tar products for their acidic, basic and neutral components, sulphuric acid is used to separate the basic component and caustic soda solution to separate the acid component.

(ii) Separation of acetic acid from pyroligneous acid as its calcium salt and decomposing the latter with concentrated hydrochloric acid.

(iii) Preparation of pure methyl alcohol free from acetone by conversion of alcohol into methyl oxalate, which is then decomposed by boiling with caustic soda solution.

(iv) Separation of primary, secondary and tertiary amines by Hinsberg or Hoffmann methods using diethyl oxalate or benzene sulphonyl chloride for their separation.

Activity-08

Concepts of Normality and Molarity and standardization.

It is necessary to know how many moles of solute are present in one liter of solution, especially when these solutions are involved in chemical reactions.

Molarity and normality describe the numbers (moles) of reactants or products dissolved in one liter of solution.

Molarity(M) = moles of solute contained in one liter of solution.

The molecular weight of glucose sugar is 180 g/mole. If 360 g of glucose is dissolved in enough water to make one liter of solution, the concentration of glucose is 2.00M.

Normality(N) = moles of reactive units per liter (equivalents per liter)

Where molarity describes the moles of a complete substance per liter of solution, normality describes only the moles of reactive species per liter of solution. Normality is always a multiple of molarity. It describes the “equivalent” moles of reactants involved in chemical reactions.

Normality in Acid-Base Reactions: In an acid-base reaction, normality is a measure of the protons (H^+) or hydroxides (OH^-) that react with one another.

Consider a 1 M solution of sulfuric acid, H_2SO_4 . Since 2 protons are available to react on each molecule of H_2SO_4 , the normality is 2 N.

The same is true for bases containing more than one hydroxide ion. An 0.2 M solution of $Ca(OH)_2$ is actually 0.4 N with respect to OH^- ions.

Experiment: To standardize the given solution of NaOH & HCl solution by using 0.1 N succinic acid solution.

Apparatus used: Burette, pipette, beaker, conical flask, weight box, chemical balance etc.

Chemicals : NaOH (x N) (Sodium hydroxide) , HCl solution (x N), Succinic acid , Phenol phthelin indicator.

Procedure :

PART (1)

To weight accurately 1.475g succinic acid powder by counterpoise method.

Transfer the powder in to a 250 ml beaker.

Add about 150 ml distilled water in beaker, dissolve the powder using glass rod. Transfer this solution in 250 ml measuring flask & make the quantity 250 ml by adding required quantity of distilled water. This is 0.1 N succinic acid solutions.

PART (2)

Now, Standardization of standard solution of 0.1 N succinic acid $\text{HOOC}-(\text{CH}_2)_2-\text{COOH}$

Wash all the accessories with water & then use with solution as per we use.

Fill the burette with NaOH of x N solution.

Take 10 ml solution of succinic acid by a pipette in conical flask.

Add three drops of reagent phenolphthalein.

Titrate the solution again with NaOH solution it becomes light pink to colorless. Repeat the titration to get at least three concordant or constant reading.

Calculate exact normality or gm/ liter of NaOH .

PART (3)

Finally ,Standardization of HCl by using standard solution of NaOH.

Fill the burette with NaOH solution.

Take 10ml solution of HCl of x N by pipette in a conical flask. Add 3 drops indicator phenolphthalein.

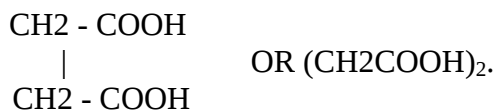
Titrate the solution again with NaOH till it becomes light pink to colorless.

Calculate exact normality & gram 1 liter.

OBSERVATION & CALCULATION

PART (1)

Formula of Succinic acid:-



Molecular weight of succinic acid :- $4 (\text{C}) + 6 (\text{H}) + 4 (\text{O}) = \text{_____ gm/mol}$

Equivalent weight of $(\text{CH}_2\text{COOH})_2$:- Molecular weight of $(\text{CH}_2\text{COOH})_2$ No. of H^+ ions charge
Amount of required for 1L of 0.1N succinic acid solution :- Equivalent wt. of $\text{CH}_2(\text{COOH})_2 =$
Normality x 100 Volume of $(\text{CH}_2\text{COOH})_2$

PART (2)

Burette : xN NaOH

Titration flask : 0.1N $\text{CH}_2(\text{COO})_2$ + phenolphthalein

Color change: colorless to light Pink

Observation Table :-

S.R. No	I (ml)	II (ml)	III (ml)	Average
Initial Reading				
Final Reading				
Difference				

Calculation :- $N_1 V_1 (\text{NaOH}) = N_2 V_2 (\text{CH}_2 \text{COOH})_2$
 Normality of NaOH = $N_2 V_2 / N_1$
 Gram / liter of NaOH = Normality x equivalent weight

PART (3)

Burette : xN NaOH

Titration flask : xN HCl + phenolphthalein

Color change: colorless to light Pink

Chemical equation :- $\text{NaOH} + \text{HCl} \rightarrow \text{NaCl} + \text{H}_2\text{O}$

Observation Table :-

S.R. No	I (ml)	II (ml)	III (ml)	Constant
Initial Reading				
Final Reading				
Difference				

Calculation :-

$N_1 V_1 (\text{NaOH}) = N_2 V_2 (\text{HCl})$

Normality of HCl = $N_1 V_1 / V_2$

Gram / liter of NaOH = Normality x equivalent weight

Activity-09

Preparation of phenol formaldehyde resin.

Aim: To prepare phenol formaldehyde resin (Novalak).

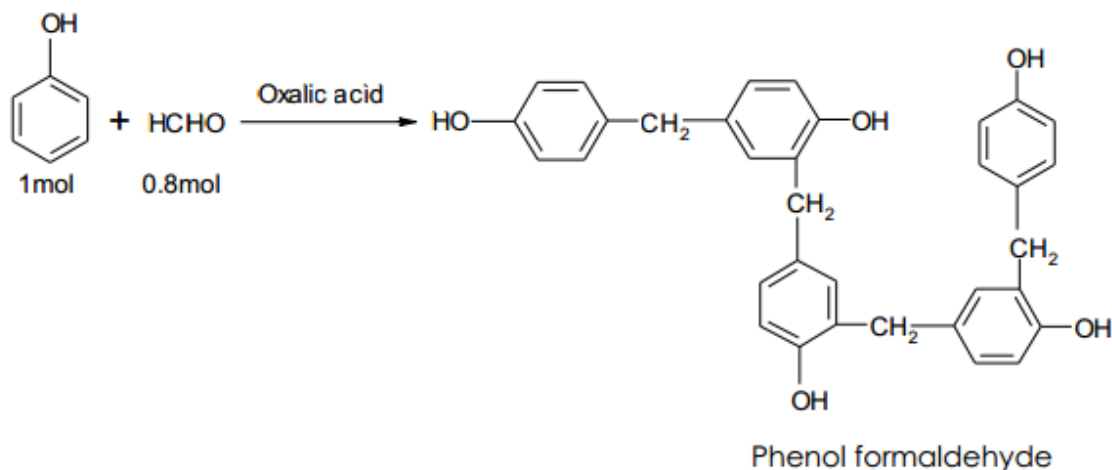
Requirements: Phenol, formaline, oxalic acid, Round bottom flask, Reflux condenser.

Process: In a 250ml Round bottom flask, take 25gm phenol and 16 ml of formaline and 0.375 gm Oxalic acid. 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 with very difficulty from the surface of the solution. At that time stop heating and pour immediately the content of flask in previously weighed test tube.

(1) Weight of phenol = _____ grams

(2) Volume of formaline = _____ ml.

Equation:



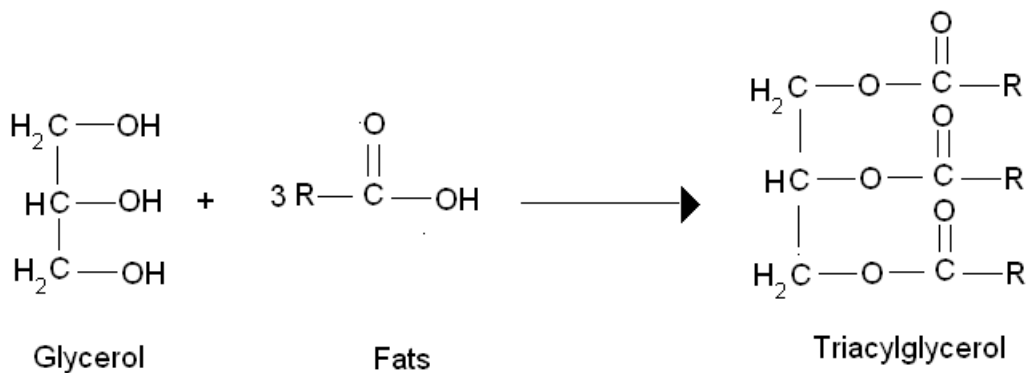
Result: Weight of phenol formaldehyde resin formed = _____ grams

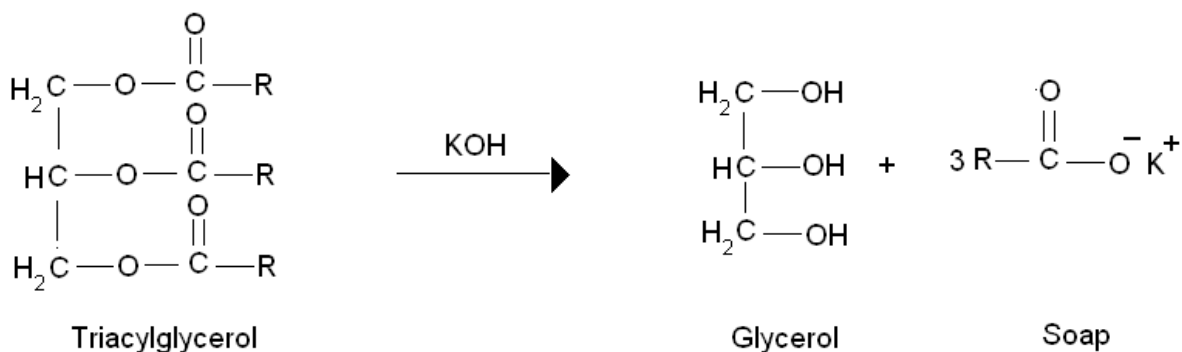
Activity 10

Determination of Saponification value of oil

Saponification is the hydrolysis of oils and fats under basic conditions to produce glycerol and the salt of the corresponding fatty acid. Saponification literally means 'soap making'.

The Saponification number is the number of milligrams of potassium hydroxide required to neutralize the fatty acids resulting from the complete hydrolysis of 1g of oil.





Procedure:

Materials Required:

- | | |
|---|-------------------------|
| 1) Oils/Fats [coconut oil, sunflower oil] | 2) Conical Flask |
| 3) 100ml beaker | 4) Weigh Balance |
| 5) Dropper | 6) Reflux condenser |
| 7) Boiling Water bath | 8) Glass pipette (25ml) |
| 9) Burette | |

Reagents Required:

- | | |
|------------------------------------|-------------------------------|
| 1) Alcoholic KOH(95% ethanol, v/v) | 2) Potassium hydroxide [0.5N] |
| 3) Fat solvent | 4) Hydrochloric acid[0.5N] |
| 5) Phenolphthalein indicator | |

Procedure:

- 1) Weigh 1g of oil in a dry beaker and dissolve in about 3ml of the ethanol /ether mixture.
- 2) Quantitatively transfer the contents of the beaker three times with a further 7ml of the solvent.
- 3) Add 25ml of 0.5N alcoholic KOH and mix well, attach this to a reflux condenser.
- 4) Set up another reflux condenser as the blank with all other reagents present except the sample oil.
- 5) Place both the flasks in a boiling water bath for 30 minutes .
- 6) Cool the flasks to room temperature .
- 7) Now add phenolphthalein indicator to both the flasks and titrate with 0.5N HCl .
- 8) Note down the endpoint of blank and test .
- 9) The difference between the blank and test reading gives the number of millilitres of 0.5N KOH required to saponify 1g of fat.
- 10) Calculate the saponification value using the formula :

Observation Table
(for test and blank)

S.R. No	I (ml)	II (ml)	III (ml)	Constant

Initial Reading				
Final Reading				
Difference				

Saponification value = mg of KOH consumed by 1g of fat.

Weight of KOH = Normality of KOH x Equivalent weight x volume of KOH in litres

Volume of KOH consumed by 1g fat = [Blank – test]ml

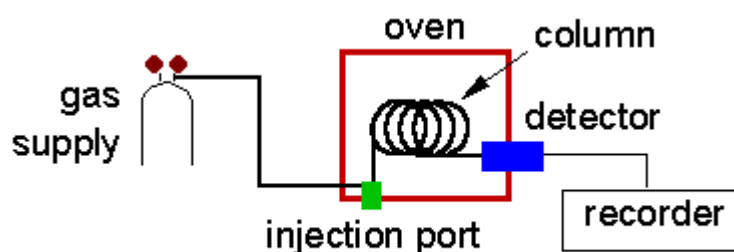
Activity-11

Basics concepts of Chromatographic Technique

Chromatography is a physical method of separation that distributes components to separate between two phases, one stationary (stationary phase), the other (the mobile phase) moving in a definite direction. A method of separating and analyzing mixtures of chemicals.

The separation, especially of closely related compounds, by allowing a [solution](#) or [mixture](#) to seep through an adsorbent (such as clay, gel, or paper) so each [compound](#) becomes adsorbed into a separate, often colored, [layer](#).

Gas Chromatography (G.C.) This technique can be used to separate mixtures of volatile organic compounds. A gas chromatograph consists of a flowing mobile phase, an injection port, a separation column containing the stationary phase, a detector, and a data recording system.



Liquid chromatography (L.C.) This technique is useful for separating mixtures of ions or molecules that are dissolved in a solvent.

Thin-Layer chromatography (TLC) This technique is useful for separating mixtures of organic compounds. Because of the simplicity and rapidity of TLC, it is often used to monitor the progress of organic reactions and to check the purity of products.

Key words used in chromatographic techniques:

The **eluate** is the mobile phase leaving the column.

The **eluent** is the solvent that carries the analyte.

An **eluotropic series** is a list of solvents ranked according to their eluting power.

The **solute** refers to the sample components in partition chromatography.

The **solvent** refers to any substance capable of solubilizing another substance, and especially the liquid mobile phase in liquid chromatography

An **immobilized phase** is a **stationary phase** that is immobilized on the support particles, or on the inner wall of the column tubing. The stationary phase is the substance fixed in place for the chromatography procedure. Examples include the silica layer in thin layer chromatography.

The **mobile phase** is the phase that moves in a definite direction.

In the case of HPLC the mobile phase consists of a non-polar solvent(s) such as hexane in normal phase or polar solvents in reverse phase chromatography and the sample being separated. The mobile phase moves through the chromatography column (the stationary phase) where the sample interacts with the stationary phase and is separated.

The **retention time** is the characteristic time it takes for a particular analyte to pass through the system (from the column inlet to the detector) under set conditions.

Preparative chromatography is used to purify sufficient quantities of a substance for further use, rather than analysis.