



GUJARAT TECHNOLOGICAL UNIVERSITY

ACTIVITY BOOK

(CHEMISTRY BRIDGE)

BE FIRST SEMESTER-2013

**INITIATIVE BY- Directorate of Technical Education,
Govt. of Gujarat**

Coordinator- Prof. M. R. Patel Principal, VGEC, Chandkheda, Gandhinagar

Subject Convenor - K Gurjar, Asst Professor, Department of Chemical Engg.
VGEC, Chandkheda

Subject Experts- Dr. Shashi V Ranga, Asst. Professor, Department of Chemical Engg
LDEC, Ahmedabad
Purvi B Shukla, Asst. Professor, Department of Chemical Engg
LDEC, Ahmedabad

Objectives:

- Identification of chemical compounds.
- To handle various lab instruments.
- To prepare solution of desired concentration.
- To co-relate fundamentals/basics in real world.
- Identify various glass-wares.
- Adjustment in college atmosphere and able to work in team.

Outcomes: At the end of course, Students will able to

- Use various lab equipments and glass-wares.
- Work in team and adjust in new atmosphere
- Understand industrial applications of various compounds
- Learn basics of analytical chemistry

Evaluation Pattern-

- Weekly group discussion, poster preparation, Quiz and activity etc.
- MCQ test at the end of the course (50 marks, 1 Hrs duration)

References-

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

Bridge Course-(Chemistry)

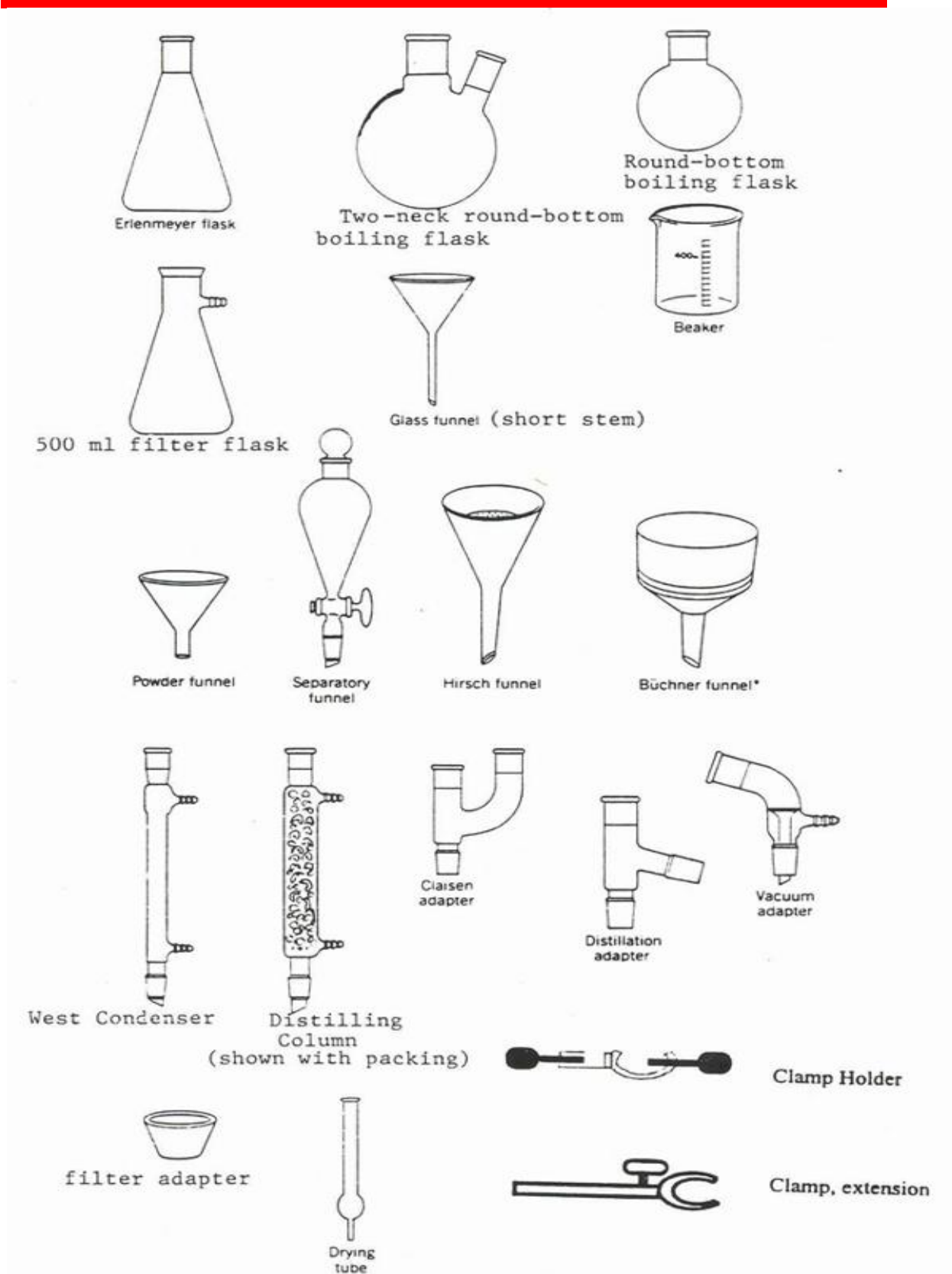
B.E Semester 1

Duration: 6 Weeks (4 Hrs/Week)

Sr. No.	Title of Activity	
1	A Visit to chemistry Laboratory	
2	Identify the following equipments/glass-wares in your chemistry laboratory	
3	Identification of elements in organic compounds	
4	Crystallization/ sublimation methods for purification	
5	Preparation of standard solution and its standardization.	
6	Measurement of pH by pH-meter	
7	Measurement of hardness of water by EDTA method. (complexometric titration)	
8	Paper/ Column Chromatography method	
9.	Identification of metals in alloys	
10	Preparation of phenol-formaldehyde resin	
11.	Study of an electrochemical cell	
12.	List of chemicals	

Note: Students may perform any 10 activities.

Activity-01 (A Visit to chemistry Laboratory)



Activity-02

Identify the following equipments/glass-wares in your chemistry laboratory-





Activity

Write the name of other equipments those are not listed in this book-

- 1
- 2
- 3
- 4
- 5
- 6
- 7
- 8
- 9
- 10

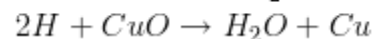
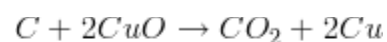
Activity-03

Identification of elements in organic compound

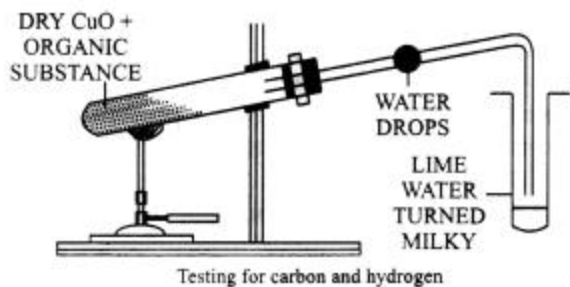
Element	Order of abundance
Carbon	Always present
Hydrogen	Nearly always present
Oxygen	Generally present
Nitrogen, halogens and sulphur	Less commonly present
Phosphorus and metals	Rarely present

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 CO_2 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 ($-\text{COOH}$), hydroxyl ($-\text{OH}$), nitro, aldehyde ($-\text{CHO}$); ketone ($>\text{CO}$); ester ($-\text{COOR}$) 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.

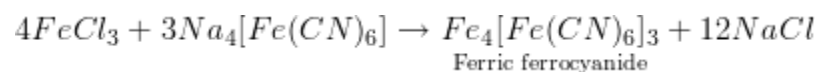
3. Detection of Nitrogen

(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 a tongs and heat it gently to melt the contents so that they may react and form sodium cyanide.

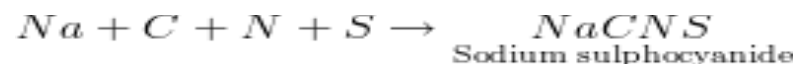


Now heat the tube strongly till the reaction ceases and the tube becomes red hot. Plunge it into about 10 mL of distilled water. Boil the contents and filter. The filtrate is called '**Sodium Extract**' and contains nitrogen as sodium cyanide. Take a portion of this sodium extract and add two drops of 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.

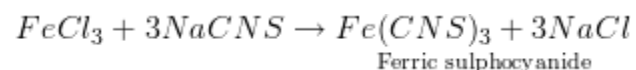
To the cooled solution then add a little ferric chloride solution and excess of hydrochloric acid. A blue or green colouration due to formation of ferric ferrocyanide confirms the presence of nitrogen.



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.



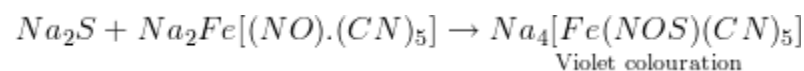
(b) **Soda lime test:** A small number of nitrogeneous compounds respond to this test. The substance is mixed with double the amount of soda lime (NaOH + CaO) and heated in a test tube. If ammonia is evolved, the presence of nitrogen in the substance is proved. A negative result, however, does not prove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

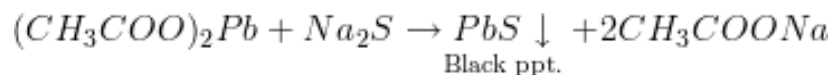
4. Detection of Sulphur

(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:

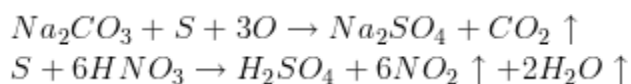
(i) To a portion of the sodium extract, add freshly prepared sodium nitroprusside solution. A deep violet colouration indicates the presence of sulphur.



(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.



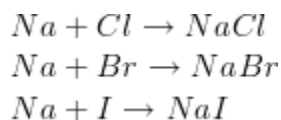
(b) **Oxidation test:** Easily volatile substances usually do not respond to Lassaigen's test. In such a case the compound is fused with a mixture of sodium carbonate and potassium nitrate, or heated with fuming nitric acid in a 'bomb tube' (see estimation of sulphur). Sulphur, if present, is oxidised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

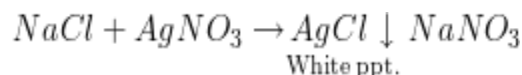
(a) **Lassaigne's test:** Prepare the sodium extract of the compound as in the detection of nitrogen. Halogens, if present, are converted to the corresponding sodium halides.



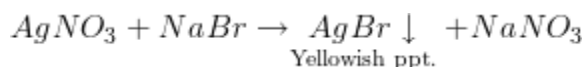
These salts are present in the sodium extract. A portion of the sodium extract is boiled with dilute nitric acid to decompose any cyanide or sulphide, and then cooled silver nitrate is added and following observations are recorded:

***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.

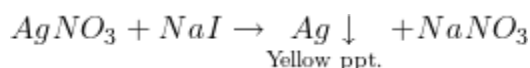
(i) A white precipitate readily soluble in ammonium hydroxide indicates **Chlorine**.



(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 (a) fuming nitric acid under pressure (b) sodium peroxide or (c) a mixture of sodium carbonate and sodium nitrate. The phosphorus is thus oxidised 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-04

Methods of Purification

- (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.

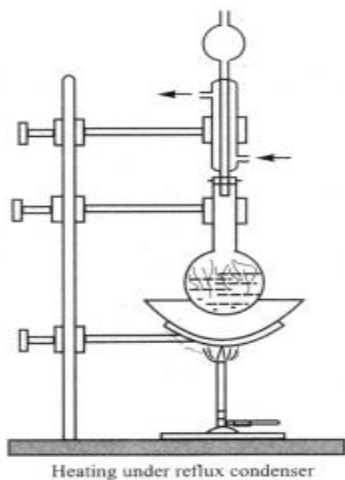
(i) Selection of the proper solvent: A proper solvent is that which (a) does not dissolve the impurities at all or dissolves them to a much larger extent so that either the impurity of the product remains in the mother liquor after separation of the solids, (b) the solvent selected should be such that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

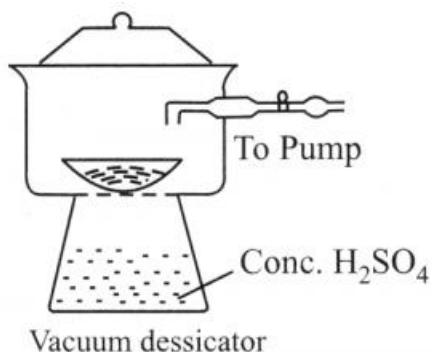
(ii) Preparation of the solution: First of all the crude material is well powdered and a suitable amount is taken in a round bottom flask. A small amount of solvent is added and the flask heated on a water bath using a reflux condenser. If the substance does not dissolve completely.



Preparation of the solution

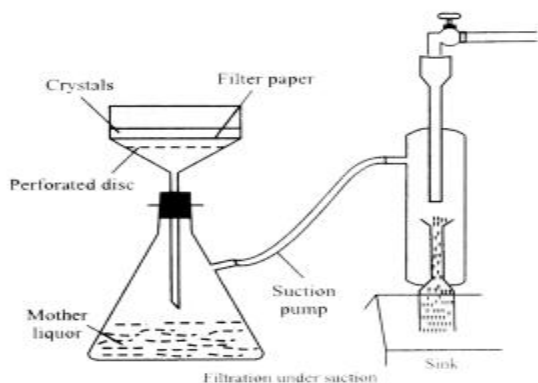
then some more solvent is added. In case water is used as a solvent, the flask can be heated directly over the flame without using the reflux condenser. Sometimes only an air condenser is used.

(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.



Filtration

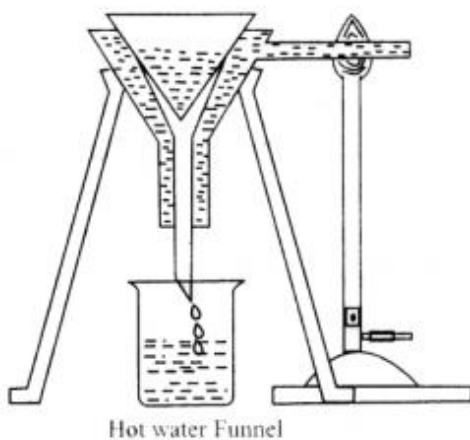
(iv) Crystallization: The hot filtrate obtained above is allowed to cool. For getting pure and smaller crystals the



Crystallization

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.

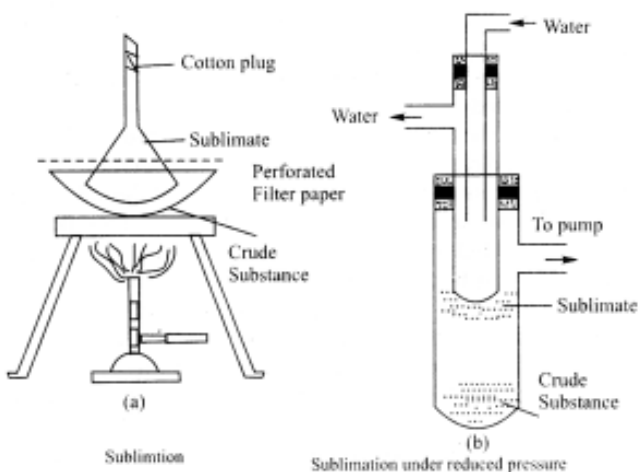
2. Fractional Crystallization: The method of simple crystallization is suitable for the purification of a single substance, contaminated with small amount of impurities. But when two (or more) substances (both of which are soluble in the solvent used), are to be separated, the technique of fractional 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 watch glass is heated very slowly on a sand bath with a free flame.

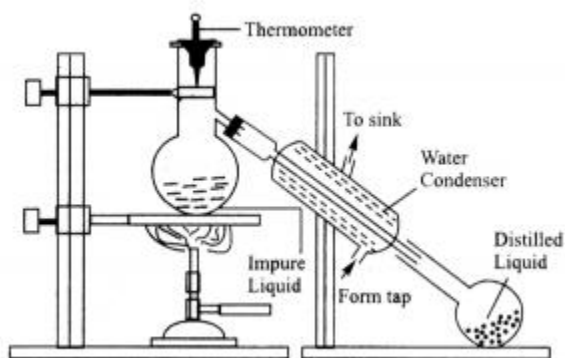


Sublimation

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.



Distillation at atmospheric pressure

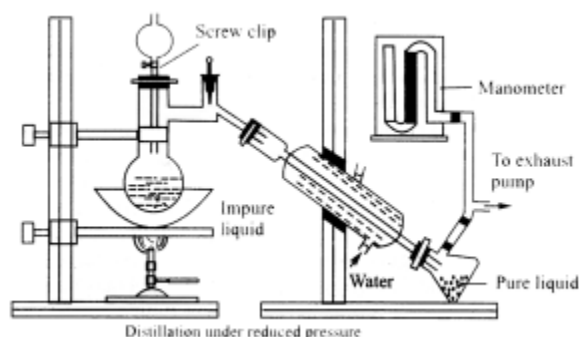
The impure liquid is boiled in a flask and the vapours thus obtained are collected and condensed in another vessel called receiver. The non-volatile impurities are left behind in the first flask. The apparatus used for simple distillation is shown above.

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. For those



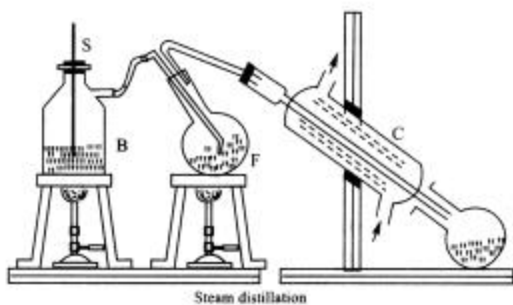
Distillation under reduced pressure

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 the non-volatile



Steam distillation

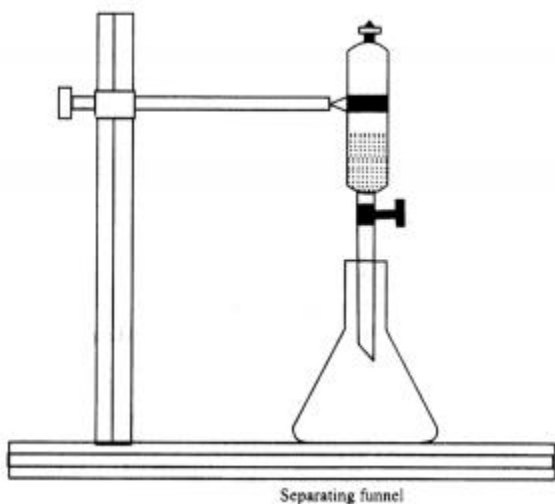
impurities are left in the distillation flask. The apparatus is shown in figure given above. The substance to be purified is placed in a large round bottom flask, clamped at an angle, so as to prevent the solution being thrown into the condenser. The steam is generated in the steam generator and is bubbled 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.

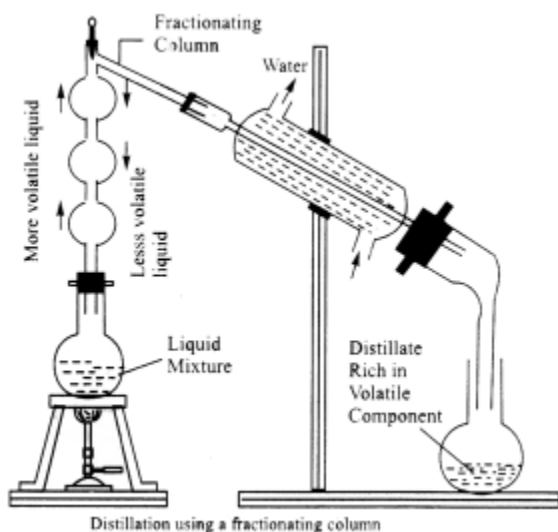
The distillate in the receiver consists of water and the purified substance. In case the substance is an oily liquid of aniline, it is separated from water by means of a separating funnel. If it is an insoluble solid it is separated by filtration. If the substance is soluble in acid, the solution is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



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**.

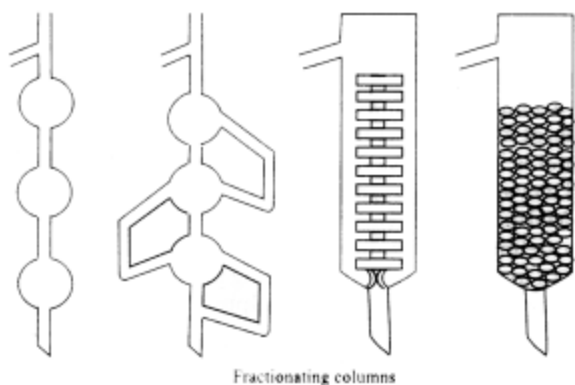


Fractional distillation

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,



Fractional distillation

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 of 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-05

Aim: 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) , HCl solution (x N), Succinic acid powder , Phenol phthelin indicator.

Procedure :

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 (II)

Standardization of standard solution of 0.1 N succinic acid ($\text{CH}_2(\text{COOH})_2$).

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 ($\text{CH}_2(\text{COOH})_2$) or succinic acid 0.1 N 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 (Sodium hydroxide.)

Part (3):-

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.

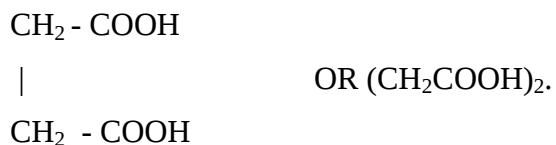
Titrate the solution again with NaOH till it becomes light pink to colorless.

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 $(\text{CH}_2\text{COOH})_2$:-

$$= \frac{\text{Molecular weight of } (\text{CH}_2\text{COOH})_2}{\text{No. of H}^+ \text{ ions charge}}$$

Amount of required for 1L of 0.1N succinic acid solution :-

$$= \frac{\text{Equivalent wt. of } (\text{CH}_2\text{COOH})_2 * \text{Normality} * 100}{\text{Volume of } (\text{CH}_2\text{COOH})_2}$$

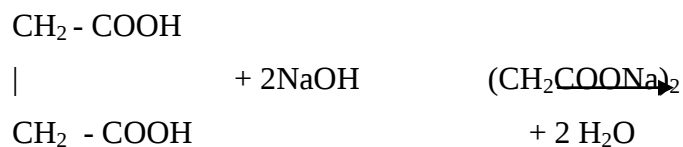
PART (2)

Burette : xN NaOH

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

Color change: colorless to light Pink

Chemical equation :-



Observation Table :-

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

Calculation :-

$$N_1 V_1 (\text{NaOH}) = N_2 V_2 (\text{CH}_2 \text{COOH})_2$$

$$\text{Normality of NaOH} = \frac{N_2 V_2}{N_1}$$

$$\text{Gram / liter of NaOH} = \text{Normality} * \text{equivalent weight}$$

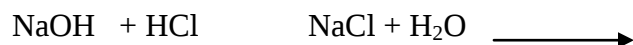
PART (3)

Burette : xN NaOH

Titration flask : xN HCl + phenolphthalein

Color change: colorless to light Pink

Chemical equation : -



Observation Table :-

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

Calculation :-

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

$$\text{Normality of HCl } N_2 = \frac{N_1 V_1}{V_2}$$

Gram / liter of NaOH = Normality * equivalent weight

Activity – 06

AIM: - Measurement of P^H value of given Sample by P^H Paper & PH meter.

Requirements: - 0.1N NaOH, 0.1 N HCl, Distilled water, PH meter, PH paper.

Chemicals: - 0.1N NaOH, 0.1N HCl, distilled water, tap water.

PART (I)

Calibrate P^H meter by buffer Solution having P^H positive calibration Knob.

Measure P^H of different Solution by P^H meter.

Note down reading in PH meter.

PART (II)

Measure P^H of sample of P^H paper by different Solution.

Note down reading in observation table.

Result :-

PH meter gives the accurate result then PH paper.

S.R. No.	Name Of Solution	P ^H by P ^H paper	P ^H by P ^H meter

Activity– 07

AIM: - To determine the total hardness of given sample of water using 0.01M EDTA Solution.

Apparatus: - burette, pipette, conical flask, beaker etc.

Chemical: - 0.01 M EDTA solution, ammonia buffer, erichrom black-T (EBT), indicator, distilled water, tap water.

Procedure:-

Fill the burette with EDTA solution take 25 water sample, in conical flask of P^H 10 + 0.1 (mixture of NH₄Cl + NH₄OH).

Now add 2 drops of EBT Indicator which gives wine red color now titrate with 0.01N EDTA solution till wine recolor convert in to sky blue.

Note down reading repeat the process till constant reading is obtained suppose x ml.

1 ml 1 M EDTA = 100 mg hardness as constant.

1 ml 0.1M EDTA = 1mg hardness as CaCO₃

X ml 0.1M EDTA = x ml hard in 25ml water

Hardness in mg /L = 40 * x

Total hardness = 40 * x

Result :-

25 ml water sample reading = _____ ml of 0.01M EDTA solution.

Total hardness in mg/L as CaCO₃ in water = _____ mg /L

Note: - results are expressed as CaCO_3 because it is primary standard and verify of cation & remain in water.

OBSERVATION & CALCULATION

Burette: - 0.01 M EDTA solution.

Pipette: - 25 ml water + 1 ml ammonia buffer.

Indicator: - 5 drop EBT.

Color change: - wine red to Sky blue.

Chemical Reaction:-

$\text{M C (divalent polyvalent cation)} + \text{EBT} = \text{EBTM (vine red)}$

$\text{EBTM} + \text{EDTA} = \text{EDTAM} + \text{EBT (Sky blue)}$.

Observation Table:-

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

Calculation:-

Molecular weight of $\text{CaCO}_3 = 40 + 12 + 48$

Now, 1000 ml 1 M EDTA 100g hardness as CaCO_3

1000 ml 1 M EDTA = 100 gm hardness as Constant.

Activity- 08

AIM: - To find out R_f value of red & blue links by using paper Chromatography.

Procedure :- Take filter paper in form of a rectangle Draw a base line about 1.5 to 2 cm on paper put spot of blue & Red links on base line. Dip paper in solvent distance travelled by substance & solvent solvent Calculate.

Observation & Calculation

Observation -

Red – ink Observation:-

$\text{RF} = \frac{\text{Distance travelled by Substance}}{\text{Distance travelled by Solvent}}$

Blue – ink Observation:-

RF = Distance travelled by Substance

Distance travelled by Solvent

Result :- R₁ value is : Red = 0.2, Blue = 0.1707

Activity- 09

Introduction

An alloy is a homogeneous mixture of two or more metals or a metal and non-metal.

They are generally harder than their components with reduced malleability and ductility. Alloys are prepared to enhance certain characteristics of the constituent metals, as per requirement.

In this project, we shall qualitatively analyze the chemical composition of two alloys:-

Brass and bronze

Aim of the Experiment

General objective:

This project is being carried out with a view to increase the appreciation of alloy-analysis as an important branch of chemistry. The hands-on laboratory experience gained is highly beneficial in understanding the general procedure of qualitative analysis of an unknown sample.

Specific objective:

In this activity, we shall be analyzing the constituents of Brass and Bronze.

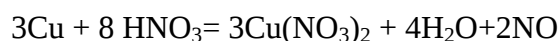
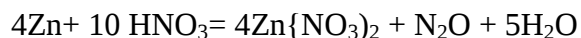
MATERIALS REQUIRED

- 1) BRASS AND BRONZE PIECES
- 2) CHINA DISHES
- 3) FILTRATION APPARATUS
- 4) NITRIC ACID
- 5) HYDROGEN SULPHIDE GAS
- 7) AMMONIUM CHLORIDE
- 8) POTASSIUM FERROCYANIDE
- 9) AMMONIUM SULPHIDE
- 10) DIL HYDROCHLORIC ACID

Theory

A. Brass

Brass contains Cu and Zn . Both dissolve in nitric acid.



Further analysis is carried out for respective ions.

Cu dissolves in H_2S to give black ppt. of CuS . It is filtered to get the solution of Zinc Sulphide. It precipitates out in the form of ZnCl_2 in an ammoniacal soln. of Ammonium chloride. The precipitate is dissolved in dilute HCl and then treated with Potassium ferrocyanide to get a bluish-white ppt. of $\text{Zn}_2[\text{Fe}(\text{CN})_6]$.

Bronze

Bronze contains Cu and Sn. Their nitrates are obtained by dissolving the sample in conc. Nitric acid. The nitrates are precipitated as sulphides by passing H_2S through their solution in dil. HCl . The CuS is insoluble in yellow ammonium sulphide, while SnS is soluble. The ppt. is separated by filtration.

The ppt. is dissolved in conc. HNO_3 and then Ammonium hydroxide solution is passed through it. Blue colouration confirms the presence of Cu.

The filtrate is treated with conc. HCl followed by Zinc dust to obtain SnCl_2 . Then HgCl_2 solution is added. Formation of slate-coloured ppt. indicates the presence of Sn.

Detail of Procedure/Observations Brass,:

1. A small piece of brass was placed in a china dish and dissolved in minimum quantity of 50% conc nitric acid.
2. The soln. was heated to obtain a dry residue. The residue was dissolved in Dilute HCl . H_2S gas was passed and a black ppt. was observed. The soln. was filtered and the ppt. was dissolved in NH_4OH soln. A blue coloration observed indicates the presence of Cu.
4. The filtrate was tested for presence of Zn.

Ammonium hydroxide and chloride solutions were added and then H_2S gas was passed. A dull grey ppt. was separated and dissolved in dil. Nitric acid followed by addition of Potassium ferrocyanide. A bluish white ppt. confirms the presence of Zn.

Bronze:

1. The sample was dissolved in 50% HNO_3 and then heated to obtain nitrates.
2. The nitrates were dissolved in dil. Nitric acid and then precipitated as sulphides by passing H_2S gas.
3. The precipitates were treated with yellow amm.sulphide when a part of it dissolves. The solution was filtered.
4. The ppt. was tested for Cu as in the case of brass.
5. The filtrate was treated with conc. HCl followed by Fe dust.

6. Then HgCl_2 soln. was added. Formation of a slate- coloured ppt. confirmed the presence of Sn.

Conclusion-

Brass contains Copper and Zinc

Bronze contains Copper and Tin.

Activity-10

PREPARATION OF PHENOL FORMALDEHYDE RESIN

Aim: To prepare phenol formaldehyde resin (Novalak).

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

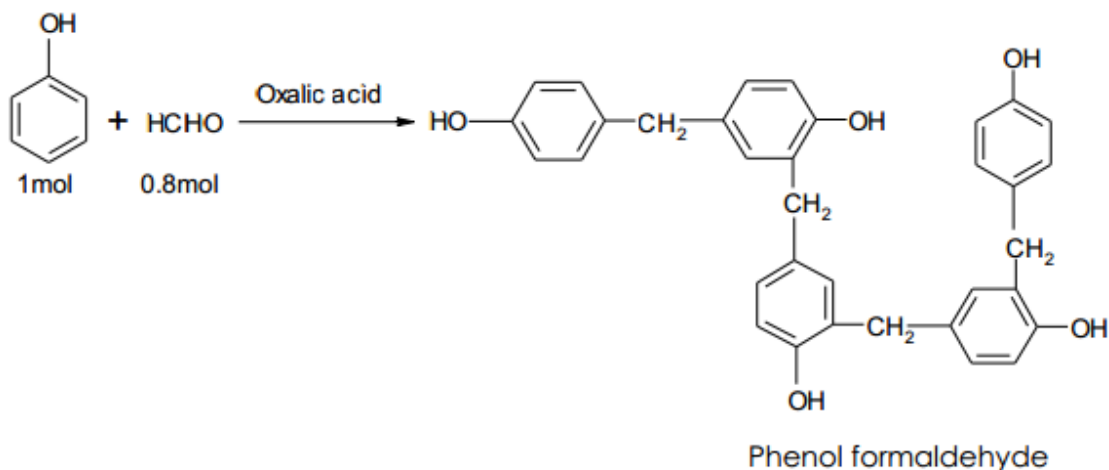
Process: In a 250ml Round bottom flask, take 25gm phenol and 16 ml of formaline and 0.375 gm Oxalic acid. The liquor is warmed continuously under reflux by means of a low Bunsen flame applied by hand directly to the flask. As soon as bubble start to rise, showing that the exothermic reaction has begun the burner is taken away and the liquor is allowed to boil on its own. Heat the flask to $60-80^\circ\text{C}$ for at least two to three hours. A time will reach when the bubble will escape with 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:



Activity- 11

To study the variation of cell potential in $\text{Zn}/\text{Zn}^{2+} // \text{Cu}^{2+}/\text{Cu}$ cell with change in conc. Of electrolytes at room temp.

Materials and Equipment

To do this experiment we will need the following materials and equipment:

Two beakers.

Zinc and Copper plate.

Filter paper.

Voltmeter.

Connecting wires.

Card board.

KNO_3 solution.

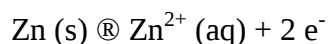
1 M, 0.1M, 0.01 M solution of :-

a. CuSO_4

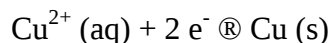
b. ZnSO_4

Daniel Cell

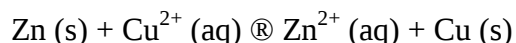
When an external circuit is connected, the chemical equation for the zinc side (anode) half cell is:



For the copper sulphate side (cathode) half cell:

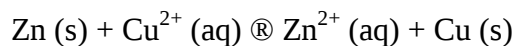


Therefore, the overall reaction of the Daniel cell is:



Introduction

It is an arrangement to convert the chemical energy of the redox reaction into electric energy.



Features of Daniel Cell:-

Zinc rod at which oxidation occurs is called the anode while the copper rod at which the reduction takes place is called cathode.

The overall reaction occurring in electrochemical cell is due to two half-cell reaction, one occurring in each beaker.

The half-cell reaction occurring at anode is called oxidation -half cell reaction while the occurring at cathode is called reduction.

The two half-cell reactions always take place simultaneously i.e. . . Half cell reaction cannot take place immediately.

Since electrons are produced at zinc electrode, it is rich in electrons and pulls these electrons into the external circuit and hence acts as negative pole. The copper electrode on the other hand is deficient in electrons and thus pulls the electrons from the external circuit and act as positive pole.

The electrons flow from negative pole to positive pole in the external circuit. However, conventionally the current is said to flow in opposite direction i.e. from positive pole to negative pole in the external circuit.

The concentration of copper sulphate solution decreases with passage of time as the cell operates, consequently the current fall with passage of time.

Salt Bridge :-

It consists of a tube filled with semi-solid paste obtained by adding gelatine or agar to the solution of strong electrolyte such as NaCl , NH_4NO_3 , KNO_3 etc, which does not change chemically during the process.

v Function of salt bridge:-

1. To complete the electrical circuit by allowing the solution to flow from one solution to another without mixing the two solutions.
2. To maintain electrical neutrality of solution in two half-cells.

EMF of Cells:-

When a current flows through two points a potential difference generated by a cell when the cell draws no current is called EMF.

Procedure

- I. Take two beakers and pour the required chemicals in respective beaker and mark them for identification.
- II. Take two square to slide in and connecting wire to their screw.
- III. Connect negative of the voltmeter to the anode and its positive to the cathode
- IV. Take filter paper long enough to dip into both the solution. Dip the filter paper in KNO_3 solution and put it as a salt bridge.
- V. Put on the electrode voltmeter set up. Note the reading quickly and then put off the electrode voltmeter set up.
- VIII. For measuring variations with change in concentration of electrolyte, use the electrolytes of different molarity and then do step 5 again.

Observations:-

v Electrode Potential of Zinc =.....V

v Electrode Potential of Copper=.....V

v Variation with Concentration:-

Molarity of CuSO ₄ (M)	Molarity of ZnSO ₄ (M)	Voltmeter Reading (V)
1.		
2.		
3.		
4.		

Conclusions:-

The EMF varies non-linearly with change in concentration of reactants.

Increase in concentration of ions in anode half-cell decreases EMF and vice-verse.

Activity-12

List of chemicals- Find these in your laboratory

KHSO₄	KOH
2,4-DNP	LEAD ACETATE
ACETALDEHYDE	LEAD CHROMATE
ACETAMIDE	LIME WATER
ACETANILIDE	LITMUS SOL
ACETIC ACID	MALACHITE GREEN
ACETIC ANHYDRIDE	METHANOL
ACETONE	METHYL ORANGE
ALUMINIUM	METHYL RED & BLUE

AMMONIUM CARBONATE	MnCl ₂
AMMONIUM CHLORIDE	<i>m</i> -NITROBENZENE
AMMONIUM PERPURATE	MOHR`S SALT
AMMONIUM SULPHATE	Na ₂ S ₂ O ₃
ANILINE	NaCl
ANTHRACENE	Na-METAL
BaCl ₂	NaNO ₂
BENZALDEHYDE	NaNO ₃
BENZAMIDE	NAPHALENE
BENZENE	NH ₃ LIQUIRE
BENZENE SULPHONYL CHLORIDE	<i>n</i> -HEXANE
BENZOIC ACID	NINHYDRIN SOL
B-NAPHTHOL	NITROBENZENE
Br ₂ WATER	<i>o</i> -TOLUIDINE
BROMOTHYMOL	OXALIC ACID
BUTYL ACETATE	<i>p</i> -AMINOPHENOL
BUTYL ALCOHOL	<i>para</i> -AMINOPHENOL
CaCl ₂	PARA-DAB
CAL. CARBONATE	PETROLIUM ETHER
CERIC AMMONIUM NITRATE	PHENOL
CHARCOAL	PHENOPHTHALENE
CHLOROFORM	PHOSPHORIC ACID
COBALT NITRATE	PHTHALIC ANHYDRIDE
COPPER	PICRIC ACID
COPPER SULPHATE	PINE OIL
DIETHYL ETHERE	POT FERROCYNIDE
EDTA	POT. CHROMATE

ERICHROMBLACK-T	POT. DICHROMATE
ETHYL ACETATE	POT. SULPHATE
FeCl₂	PYRIDINE
FeCl₃	SALICYLIC ACID
FEHLING SON A & B	SCHIFF`S REAGENT
FERROUS SULPHATE	SILVER NITRATE
FORMALDEHYDE	SnO₂
FORMIC ACID	SOD. CARBONATE
FURFURAL	SOD. HYPOCHLORITE
GLYCEROL	SOD. NITROPRUSSIDE
H₂SO₄	SOD. SILICATE
HCl	SOD.BICARBONATE
HgSO₄	SODALIME
HNO₃	TANNIC ACID
IODINE	TIN
IRON FILLING	TiO₂
K₂SO₄	TOLLENS REAGENT
KCl	TOLUENE
KCNS	UREA
KI	ZINC
KMnO₄	ZINC OXIDE
	ZnCl₂

