

## Purification of Organic Compounds

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A pure substance contains only one kind of molecules while an impure substance is a mixture of different kind of molecules. Thus to purify an impure substance is to separate the desired molecules from the mixture and therefore, the purification is a separation. The differences in the properties of different molecules are the basis of various separations. Products of organic reactions are seldom pure; they may be contaminated with either the starting material or side-products. To obtain the satisfactory physical constants, the substance must be pure. And to carry out the purification, various techniques have been employed depending on the physical state of the compound, which are discussed in this chapter.

### 2.1 CRYSTALLIZATION

Crystallization is a simple, effective and very important technique to separate and purify solids. It is based on the fact that all organic compounds are more soluble in hot than in cold solvents, so that solid gets dissolved on heating and is obtained back on cooling.

#### 2.1.1 Steps Involved in Crystallization

The following steps are involved in the purification by crystallization:

- (a) Selection of a solvent
- (b) Dissolution of the sample
- (c) Decolourisation of the solution
- (d) Hot filtration
- (e) Cooling for crystallization
- (f) Cold filtration
- (g) Washing the crystals
- (h) Drying the crystals
- (i) Checking the purity

**Table 2.1.** Common solvents for Crystallisation

S.No	Solvent	b.p. (°C)	Remarks
1.	Diethylether*	35	Inflammable
2.	Petroleum ether	Boiling fractions 40-60 60-80 80-100 100-120	Inflammable
3.	Dichloromethane**	41	Non-inflammable & toxic
4.	Acetone	56	Inflammable
5.	Chloroform**	61	Non-inflammable & toxic
6.	Methanol**	64.5	Non-inflammable & toxic
7.	Carbon tetrachloride**	77	Non-inflammable & toxic
8.	Ethanol	78	Inflammable
9.	Ethyl acetate	78	Inflammable
10.	Benzene***	80	Inflammable, highly Toxic and Carcinogenic
11.	Cyclohexane	81	Inflammable
12.	Acetic acid	118	Not very inflammable and pungent vapours

\* Its use should be avoided wherever possible because of its high inflammability and also its tendency to creep up the walls of the containing vessel and hence results in the deposition of solid material by complete evaporation rather than the crystallization.

\*\* Its vapours are toxic and therefore inhalation should be avoided and the recrystallization should be done in fuming cupboard.

\*\*\* Toluene is much less toxic than benzene and so wherever possible it is used preferentially.

Use of carbon disulphide, b.p. 46°C, should be avoided as it has a very low flash point and its vapours form explosive mixture with air.

continued in 0.5 ml portion each time, if the substance is not dissolved completely. The mixture is heated after each addition. If even after adding 3 ml of solvent, the compound does not dissolve on heating, the solvent is unsuitable due to the low solubility of the compound in it. If an almost clear solution is obtained, the tube is cooled by immersing it in cold water and if the solution is not clear, it is filtered and then the tube is allowed to cool. If crystallization does not start rapidly, it may be due to the lack of nuclei for crystal growth and therefore the scratching of the tube below the surface of the solution is done with the help of a glass rod. Crystals often form rapidly after scratching as fine scratches on the walls form the sites for crystal growth. If crystals do not separate even after scratching another

coloured impurities get dissolved in the boiling solvent and adsorbed by the crystals, giving an impure product. Such type of impurities are preferentially adsorbed by the activated charcoal so can be removed by boiling the coloured solution with a small amount of activated charcoal for 5-10 minutes followed by hot filtration. Now the filtrate is usually almost colourless and therefore pure crystals are obtained.

Not only the coloured impurities can be removed by the activated charcoal but also the resinous or finely divided matter can also be removed from the solution. Sometimes the solution is not clear due to the presence of resinous or very finely divided insoluble impurities, which cannot be removed by simple filtration. These impurities can also be removed by heating the solution with activated charcoal. The activated charcoal takes away these from the solution and makes it clear.

Only a small amount of activated charcoal must be used (1-2% by weight of the sample) otherwise some of the compound which is to be purified may also get adsorbed. The same procedure could be repeated with some more fresh charcoal (1-2%), if this amount is not sufficient enough. It should also be noted that the activated charcoal should be added only after the solution is cooled otherwise the solution would boil over when it is added. After this addition, the solution can be reheated to the boiling point.

Animal charcoal contains large proportion of calcium salts and should not be used with acidic solutions and also the impurities present in charcoal itself may get dissolved in hot solvent and separate out with the crystals of the compound. These problems can be sorted out by boiling animal charcoal under reflux with dilute hydrochloric acid (1:1) for 3 hours. Then the mixture is diluted with hot distilled water and filtered through a Buchner funnel. It is washed repeatedly with hot distilled water till all the acid has been removed, drained and finally dried by heating in an evaporating basin in an electric oven.

#### ***(d) Hot filtration***

Now the desired compound is in the solution in the hot solvent along with insoluble impurities and decolourising carbon, if used. They can be separated by gravity filtration. Vacuum filtration cannot be used as the reduced pressure in the suction flask will boil the filtrate and the solid will get deposited on the walls of the flask.

These processes (swirling and pouring) are repeated till whole of the solid material has been transferred into the funnel. The suction is continued till most of the liquid is filtered.

The above procedure (filtering the bulk of mother liquor followed by the entire compound) is quicker than bringing the solid material into suspension and then filter it. And also, a gentle suction is more effective and rapid filtration than powerful suction as the finer particles of the solid may drag into the pores of the filter paper in the latter case and hence results in the slower filtration.

### ***(g) Washing the crystals***

Crystals are now washed with some fresh cold solvent to remove soluble impurities from the surface of the crystals, otherwise crystals will be contaminated with these impurities when solvent get evaporated.

If the crystals are relatively soluble in the solvent, then a minimum amount of cold solvent must be used. While if they are not very soluble in solvent, washing can be done by large amount of solvent (no need to cool it). If the crystals are not in the form of solid cake, washing can be done by releasing the vacuum and then pouring of the wash liquid over the crystals and again applying the vacuum, while if the solid cake is formed, the solvent is added to the crystals in the funnel and the solid is carefully broken down and suspended by the help of a spatula. Care should be taken during the suspension that the entire solid product gets suspended without the tear or dislodge of the filter paper. Alternatively, the solid cake can be transferred to a breaker and then washing is done by breaking the cake in the solvent and the product is again collected by suction filtration.

When a non-volatile solvent e.g. acetic acid is used for recrystallization, the washing of the crystals is done with a more volatile solvent, in which crystals are not soluble to wash off non-volatile solvent so as to speed up the drying process.

### ***(h) Drying the crystals***

The crystals obtained by suction filtration can simply be dried by spreading it on the thick pad of filter paper with the help of a spatula. Another similar pad is placed on the top and then the solid is pressed. These sheet pads are changed by the fresh pads occasionally as the mother liquor wets them.

- (iv) By addition of solid carbon dioxide. On addition of a few lumps of it, cold spots are produced in the solution which facilitate the crystallization.
- (v) In some cases, when all the above methods fail, the crystallization is carried out by keeping the solution in a refrigerator or freezer for long periods of time.

### 2.1.3 Semimicro Microrecrystallizations

Sometimes very small quantity of a compound may be needed to recrystallize e.g. the compounds obtained by natural products, small-scale expensive preparations or by-products of reactions.

#### *(a) Semimicro recrystallization*

In this recrystallization the apparatuses used are shown in Figure 1.10 (b-c). The compound to be recrystallized is dissolved in semi-micro test tubes or centrifuge tubes. Heating should preferably be done in a water or oil bath to regulate the heat. While for inflammable solvents, solution is prepared under reflux conditions by using semimicro glass apparatus. The solutions are allowed to cool and separated crystals are filtered using a small Hirsch funnel as shown in Figure 1.10 (b).

Filtration nail may also be used. It is a thin glass rod flattened at one end, which is then fitted into a small glass funnel as shown in Figure 1.10 (c). The nail head is covered with a circle of filter paper of appropriate size. The size of the funnel and filtration nail may be varied depending upon the amount of crystals to be filtered.

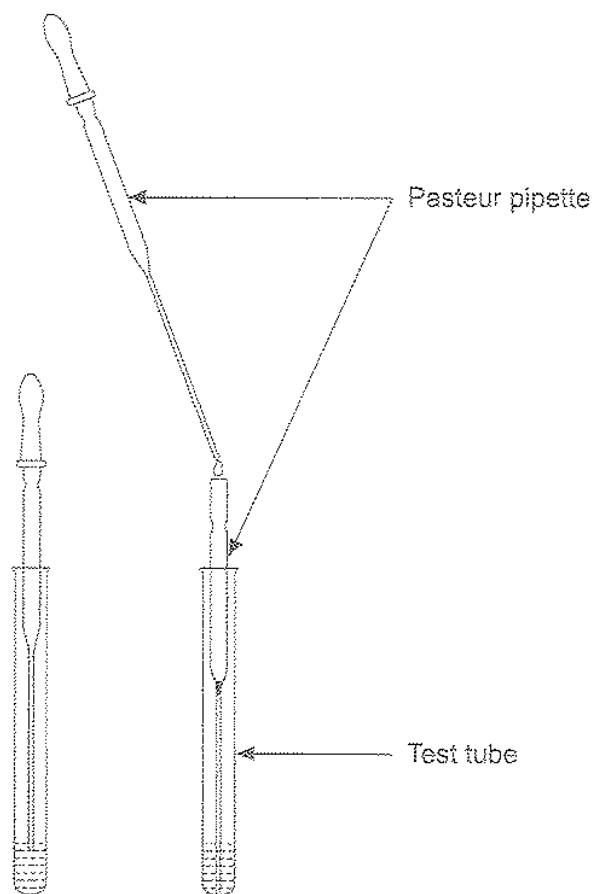
#### *(b) Micro-scale recrystallization*

When micro quantity of the compound is to be recrystallized, usual semi-micro techniques of recrystallization give great loss of the crystals.

An easy and cheap way to recrystallize the compound on micro scale is as follows:

A test tube filled with only about 1/8th by the solution should be chosen and the sample is added to it. Solvent is added and the test tube is warmed in a water bath or on a sand bath. As the amount of compound is very small, heating should be done for a brief period only. Relatively large size of test tube helps in condensation of solvent vapours by providing large surface area.

The solution is now filtered into another test tube, smaller than the previous one, by using a Pasteur pipette with a small piece of cotton just at the point where the pipette starts to narrow. Keep it on the tip of the second test tube as shown in Figure 2.2. Second Pasteur pipette is used to transfer the hot solution from the test tube to the filter pipette. The solution passes down through the pipette to the second test tube, which can be warmed carefully in water bath or sand bath to keep the solution warm. After filtration, the second test tube is kept aside for cooling.

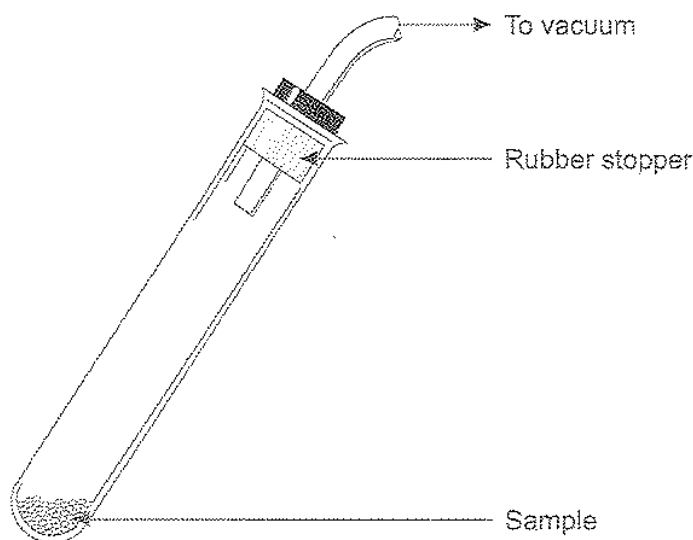


**Fig. 2.2** Filtration in a micro-scale recrystallization

When crystallization is completed, the solvent can be removed by using another Pasteur pipette. It withdraws solvent by pushing its tip in the bottom of the test tube. Washing can be done by using cold solvent.

The solvent can also be removed by centrifugation. After centrifugation, crystals are settled down and the solvent can easily be decanted. Washed with the solvent and removed it by centrifugation followed by decantation.

The crystals are dried by connecting it to the vacuum as shown in the Figure 2.3.



**Fig. 2.3** Drying crystals in a test tube

Dried crystals are collected over a piece of filter paper by inverting the test tube and tapping it with a glass rod.

#### **2.1.4 Exercises in Recrystallization**

A few examples are given to understand the recrystallization technique.

##### **(a) *Benzoic Acid from distilled water (a non-inflammable solvent)***

Some other compounds which can be recrystallized from water are acetanilide, salicylic acid etc.

Weigh 1 g of crude Benzoic acid into a 100 ml conical flask. Add about 20 ml of hot distilled water. Heat the mixture on sand bath or electric hot plate. Add more of hot water in small portions with continuous stirring and heating until the entire solid has dissolved. If the solution is colourless, filter the hot solution through a fluted filter paper supported on a stemless funnel. During filtration, keep the remaining solution hot with the help of a hot plate or filter the solution by using a hot water funnel (Figure 1.8). If the solution is coloured, allow it to cool slightly and then add about 0.1 g of decolourising carbon and boil the mixture for few minutes to remove the coloured impurities and then carry out the filtration as described earlier.

Collect the filtrate in a 100 ml conical flask, heat it again to dissolve unwanted crystals of the benzoic acid and then cover it through watch glass and allow to cool to room temperature of its own. After an hour when complete crystals separate out, filter them with suction using a small Buchner funnel. Wash the crystals twice with 5 ml portions of cold water to remove the mother liquor adhering to it. Press the crystals in the funnel with spatula to remove the mother liquor as much as possible. Remove the funnel from the filtration set and invert it on a pad of 3-4 filter papers resting upon another pad of newspaper and let them dry in air. For rapid drying, the crystals may be kept in oven on a watch glass. Note the yield and m.pt. of the recrystallized sample. The crystallization may be repeated if the recrystallized sample is not pure i.e. if compound melts over a range. Melting point of pure benzoic acid is 121°C.

**(b) Naphthalene from alcohol (an inflammable solvent)**

Some other compounds which can be recrystallized with an inflammable solvent are:

- (a) m-dinitrobenzene from alcohol
- (b) tribromoaniline from alcohol

Weigh 1 g of commercial naphthalene into a 50 ml round bottomed flask. Add 5 ml of rectified spirit and 2-3 pieces of pumice stone in it to avoid bumping. Fit a reflux condenser (Figure 1.25). Heat the mixture in a water bath or electric hot plate to boil the solvent. Add 2-3 ml of the solvent, boil carefully after each addition until whole naphthalene apart from insoluble impurities, has dissolved. If it is coloured, use 0.1 g of animal charcoal as described in the above procedure. Filter the hot solution through a fluted filter paper. Care should be taken that no flames must be there in the vicinity. Collect the filtrate in conical flask, heat it and cover it by a watch glass. When the complete crystals are separate out, filter them through a small Buchner funnel. Wash the crystals twice with 5 ml portions of cold rectified spirit. Press the crystals through spatula and dry them as described in the above procedure. Record the yield and melting point of the recrystallized compound. The melting point of pure naphthalene is 80°C.

**(c) Sulphanilic acid from water**

Crude sulphanilic acid is usually almost black in colour. It is recrystallized from distilled water and decolourized by using animal charcoal.



Weigh 1 g of crude sulphanilic acid in a conical flask and add 20 ml of distilled water. Heat to dissolve almost whole of the compound by using sand bath or hot plate. Cool it slightly and add 0.1 g of animal charcoal to the solution and continue boiling for few minutes. If the filtered solution is coloured, boil it with a further 0.1 g of animal charcoal. Filter it as in the above procedures and collect crystals of sulphanilic acid. Record\* the yield.

**(d) *Para-aminoazobenzene from solvent pair***

Aminoazobenzene ( $C_6H_5-N=N-C_6H_4-NH_2$ ) is highly soluble in methylated spirit and insoluble in water. So for recrystallization this solvent pair is used. Dissolve the crude sample in boiling methylated spirit using water bath. Remove the conical flask from water bath and then add water drop by drop until the solution becomes just cloudy. Put the solution on a water bath until it becomes clear and then remove it from water bath immediately. Filter if necessary; allow it to cool to room temperature slowly. Filter the crystals and note the yield and melting point. The melting point of pure para-aminoazobenzene is 126°C.

## 2.2 FRACTIONAL CRYSTALLIZATION

Fractional crystallization is employed to separate a mixture of two or more substances in their pure states, provided they differ in solubilities in a particular solvent. The solvent to be used can be found out by trial and if there is no appreciable difference in their solubilities, the process of separation is difficult.

In this method, the mixture is treated with a minimum quantity of the hot suitable solvent to dissolve almost all of the more soluble part leaving the less soluble behind. The solution also contains a small amount of less soluble part. Now, the solution is filtered while hot and the more soluble substance present in the solution, is obtained on cooling. The solid, hence obtained, consists of the major portion of more soluble substance and a very small portion of less soluble substance. So, the solid is dissolved once again in the minimum amount of the solvent and recovered by crystallization. This whole procedure of making solution and crystallization is repeated a number of times until the pure component (more soluble) is obtained. Purity of the substance is checked by melting point determination.

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\* Melting point cannot be recorded as it decomposes on heating and therefore, it has no melting point.

The residue obtained after the first filtration consists of mainly the less soluble part along with a very small amount of the more soluble part. This (less soluble part) can also be obtained in the pure form by working up in the similar manner, the purity of which is also checked by melting point determination.

### 2.2.1 Exercise on Fractional Crystallization

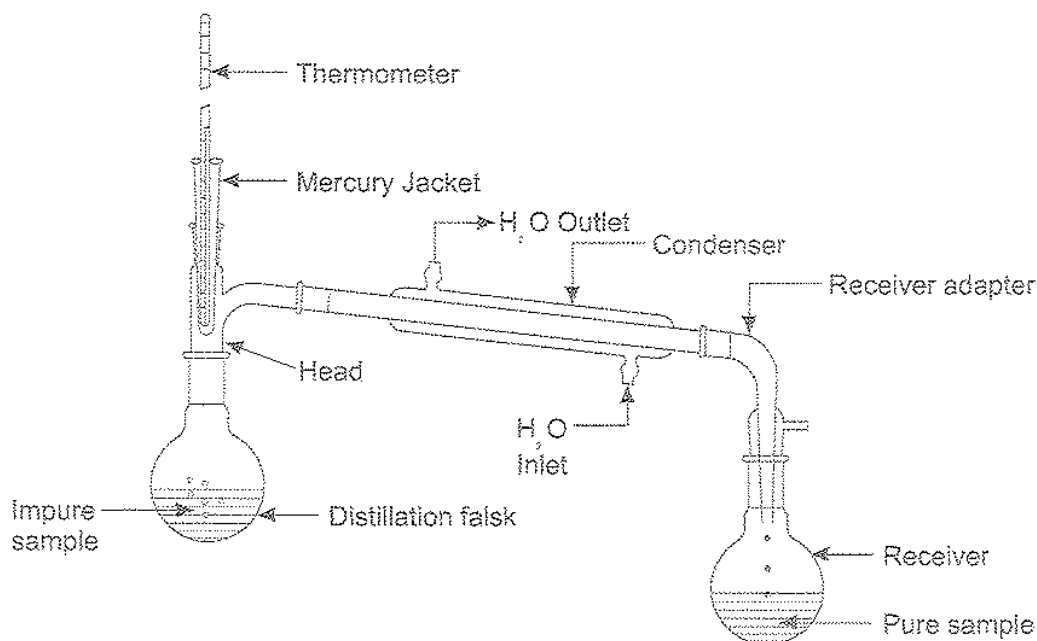
#### (a) Separation of benzoic and cinnamic acid

The two acids differ in their solubilities in warm water (40°C). Cinnamic acid is insoluble in water at this temperature and benzoic acid is soluble. And, therefore, these two acids can be separated by fractional crystallization using warm water (40°C) as a solvent. A mixture of 2 g each of the above acids is placed in a 100 ml beaker and 50 ml of warm water (40°C) is added to it. The mixture is stirred for 5 minutes on a hot water bath. The temperature of the mixture is to be maintained at 40°C. The contents of the mixture are allowed to settle down and the supernatant liquid is filtered while hot using a hot water funnel. The filtrate is collected in another beaker. The residue left in the first beaker, is treated similarly with warm water six times and then the final residue is recrystallized from boiling water. The product cinnamic acid hence obtained, is checked for purity by melting point determination (i.e. 133°C). All the filtrates are combined in 400 ml beaker and is concentrated down to 50 ml. On cooling, benzoic acid is crystallized out, which is further purified by recrystallization from hot water. The purity of benzoic acid is also checked by melting point determination (i.e., 121°C).

## 2.3 DISTILLATION

If a sample of an organic liquid contains impurities, it may be purified by distillation. This method may also be used to determine the boiling point (4.3.1). If impurities are non-volatile, on simple distillation, they are left behind in the distillation flask and when the impurities are volatile, fractional distillation method is used to purify the given liquid.

In this section, simple distillation is discussed. The method is used when the pure liquid and impurities have widely different boiling points (b. pt.) (The b.pt. difference between the two must be at least 50°C). Typical apparatus used for simple distillation is shown in Figure 2.4.



**Fig. 2.4** Apparatus used for simple distillation

A distillation flask of suitable size is fitted to a water condenser, to which an adapter is fitted so as to collect the distilled liquid into a receiver. Thermometer is fitted in the neck of the distillation flask by means of a well-bored cork. The bulb of the thermometer should be in centre and just below the level of the side tube otherwise the accurate boiling point will not be obtained.

The following points must be taken into account, while carrying out the distillation process:

- (1) The size of the distillation flask should be such that it will be one half or at the most two-thirds filled by the liquid. If the flask used is too large, then superheating and sometimes, decomposition may occur.
- (2) Few pieces of unglazed porous porcelain or carborundum must be added in the distillation flask to provide a surface on which bubbles of the vapours can be formed and hence results in the gentle boiling. In the absence of these boiling stones, the liquid often reaches a temperature above its b.pt. and leads to the violent eruptions—'bumping'.
- (3) The complete distillation apparatus should be tightly fitted as it may be used for inflammable liquids.

- (4) All the clamps used should be lined with cork as otherwise glass apparatus may have cracks on applying excessive pressure on the clamp.
- (5) In water condenser, the inlet of water should be from the lower end and outlet of water should be above the jacket so that the condenser is full of water.
- (6) A water condenser is replaced by an air condenser [a glass tube with no jacket or emptying the water from condenser jacket] if the boiling point of liquid is above 150°C.
- (7) For low boiling inflammable liquids, heating should be done by a water bath while for liquids having b.pt. more than 80°C, sand bath or direct heating on a wire-gauze can be used. Electrically heated mantles can also be used for all the liquids.

The liquid is poured in the distillation flask with the help of a funnel having stem which extends below the side arm, pumice stones\* are added to it and a thermometer is placed in the neck of the flask. The flask is heated by a suitable means, described above. The temperature will first rise rapidly until it is near boiling point of the liquid, then it will rise slowly and then will become constant. This constant temperature is the boiling point of the liquid. So heating can be fast until boiling commences, then the flame must be decreased so that the one or two drops of the liquid are collected quickly. The distillation should not be conducted too slowly as otherwise due to the lack of a constant supply of vapour on the thermometer bulb will result in an irregular boiling point while it should not be too fast so that it causes superheating by heating directly a part of the vapour as well as liquid. If the liquid is pure, most of it will be collected at a constant temperature, i.e. boiling point of the liquid, while if two components are to be separated, the first component will pass over at a constant temperature, then before second component starts coming, an intermediate stage will come in which there is rise in the temperature, it is collected in another receiver and finally the second component will come over again at a constant temperature which is collected in the third receiver. It must be noted that never overheat the flask to distil the last drop of the remaining liquid – a small residue must be left in the flask.

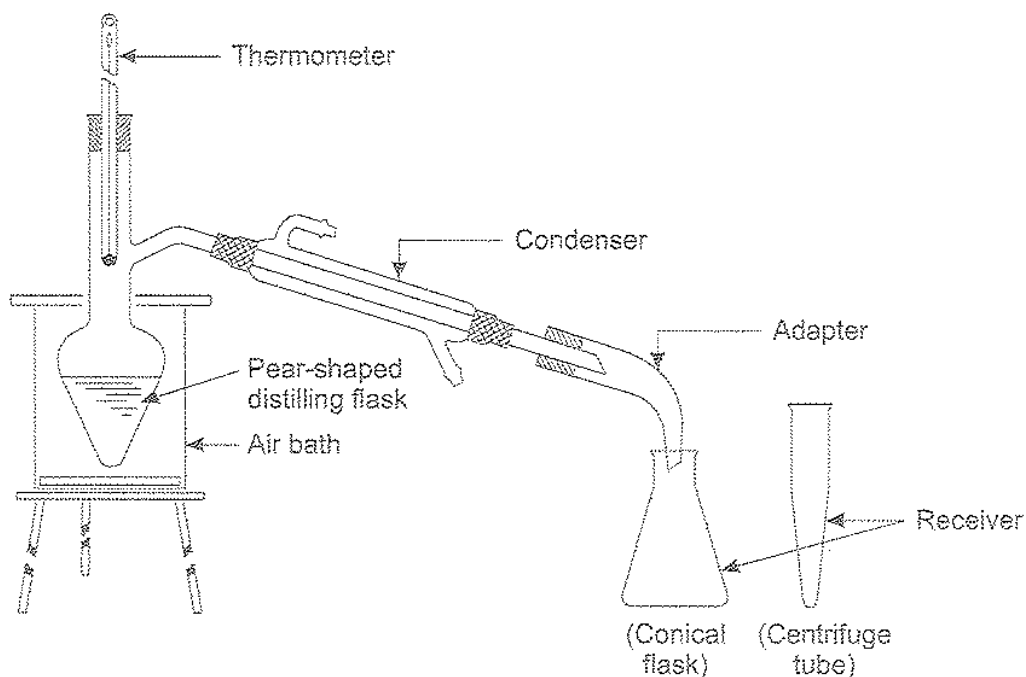
For small quantity liquids, pear-shaped flask may be used. The apparatus is shown in Figure 2.5. A 10 or 25 ml pear-shaped flask having a

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\* Should never be added to the hot liquid.

long side arm is fitted with a condenser, which in turn is fitted with a receiver. The flask is heated in an air bath and distillate is collected in an appropriate size receiver.

If instead of attaining a constant temperature, there is a steady rise in temperature, simple distillation cannot be used to purify the sample. And for the purification, fractional distillation must be used.



**Fig. 2.5** Apparatus for micro-scale distillation

## 2.4 FRACTIONAL DISTILLATION

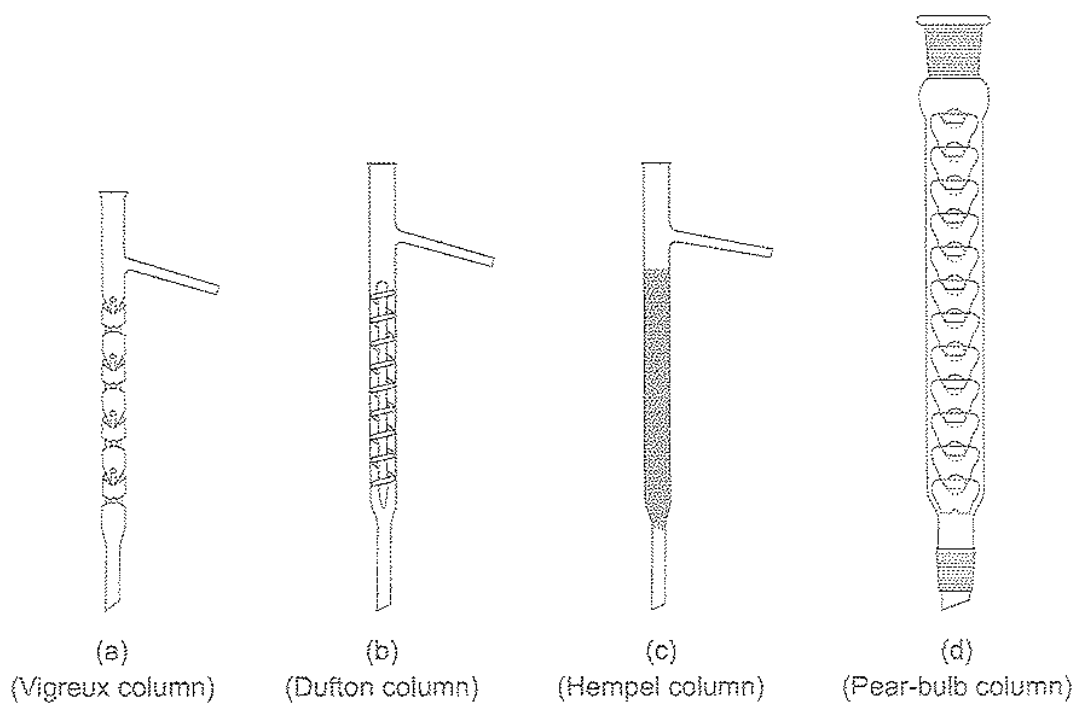
This method is used to separate the liquids whose boiling points difference is less than  $50^{\circ}\text{C}$ . For this type of distillation, the fractionating column is vertically inserted between the flask containing the liquid and condenser.

A fractionating column consists of a long vertical tube through which first the vapour goes up and then is partially condensed which comes down to the flask. This condensed liquid (having higher boiling point component) when flows down through the column gets in contact with the ascending vapours and results in the interchange of heat.

To reach the equilibrium within liquid-vapour system, the vapours get enriched with the more volatile component at the expense of the liquid. And therefore, ascending vapours become richer and more volatile i.e. the lowest boiling component while the descending condensate becomes

richer in the highest boiling component. Efficiency of a fractionating column is determined by the extent of separation. Fractionating columns, which may be used in the laboratory, are shown in Figure 2.6 (a-d).

Figure 2.6(a) shows the Vigreux column having moderate efficiency and the most widely used column. It is made by a glass tube with a series of downward slope indentations such that points of each pair of indentations must almost touch each other and form a spiral of glass inside the tube so that the vapour cannot pass directly through the whole length of the column without meeting an indentation.



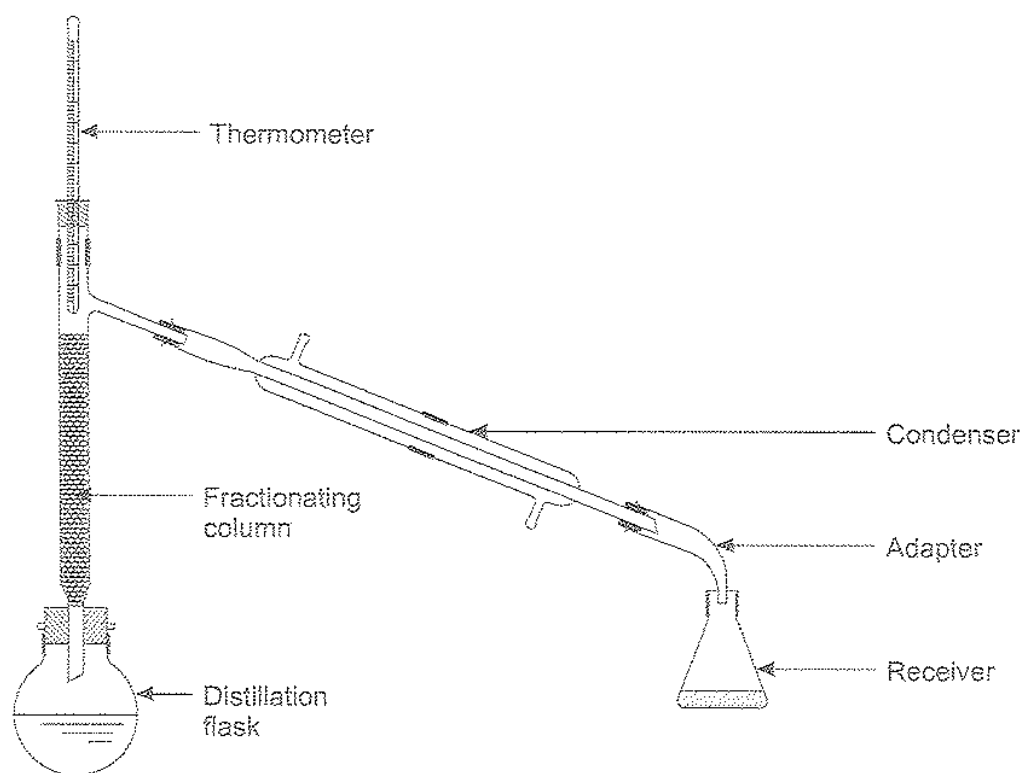
**Fig. 2.6 (a-d)** Types of fractionating column

While Figure 2.6 (b) shows all-glass Dufton column having efficiency greater than Vigreux column. It is suitable for general use. A glass spiral is fitted tightly in a plain tube around a central tube by grinding the edges of the spiral into the tube. This column has an advantage that a low volume of liquid is retained in the column.

Figure 2.6 (c) shows Hempel column whose efficiency is also greater than the Vigreux column. It is a simple glass tube packed with a suitable packing e.g.  $3/16'' \times 3/16''$  glass or porcelain rings. It has very high efficiency.

Figure 2.6 (d) shows pear-bulb column, the increase in cooling surface is provided by these pear-shaped bulbs and the efficiency of this column can be increased by increasing the number of bulbs in the column.

The apparatus employed for the fractional distillation is shown in the Figure 2.7. The impure liquid is placed in a round bottomed flask of suitable size, one-third to one-half, pumice stones are added and then the column is fitted absolutely vertical in the position followed by fixing of a water condenser to the side arm and the distillate is collected in the receiver. The thermometer is placed in the position such that its bulb should be just below the level of the side arm. The mixture is heated with a low flame. Care should be taken that the flame should be devoid of draughts so that uniform heat is supplied (use of an air bath, Figure 1.43 gives better results). Initially heating of the liquid should be slow so as to avoid the choking of the column [on fast heating, extra condensation takes place while the column is warming up and so column may be choked with the liquid]. When once the distillation has started, the flame should be adjusted that about one drop of the liquid passes in 2-3 seconds so that an efficient fractionation can be obtained. First, the low boiling point

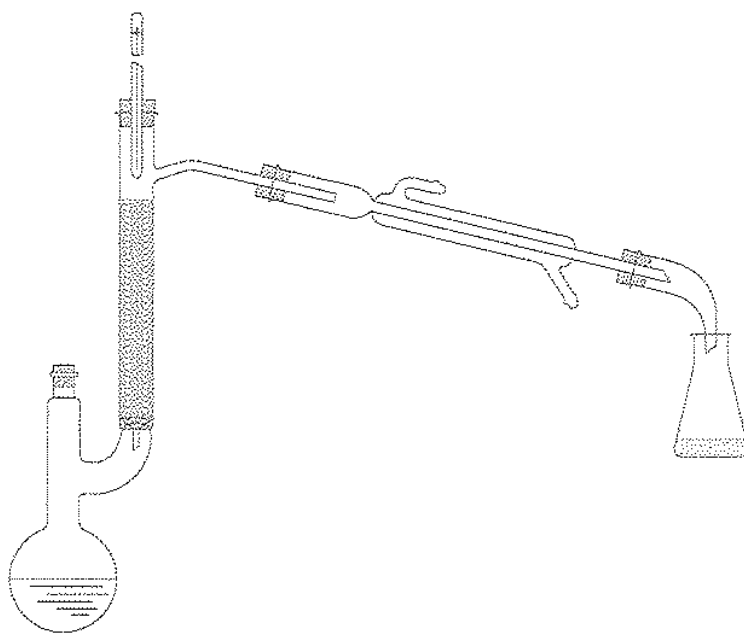


**Fig. 2.7** Apparatus for fractional distillation

component will be passed over so when it has passed, distillation should cease. Then heating is slowly increased and a second fraction starts distilling over with a sharp rise in the boiling point. A relative large intermediate fraction may be obtained, if the distillation set-up is inefficient. The distillation should be conducted slowly so as to obtain pure fractions, otherwise the fractionation has to be repeated.

If the boiling point of any of the component exceeds  $100^{\circ}\text{C}$  lagging of the column is necessary, as it will avoid the excessive cooling. Lagging is done by wrapping asbestos cloth or cotton wool around it. Excessive cooling may also be avoided by surrounding the column with a vacuum jacket or an electrically heated jacket.

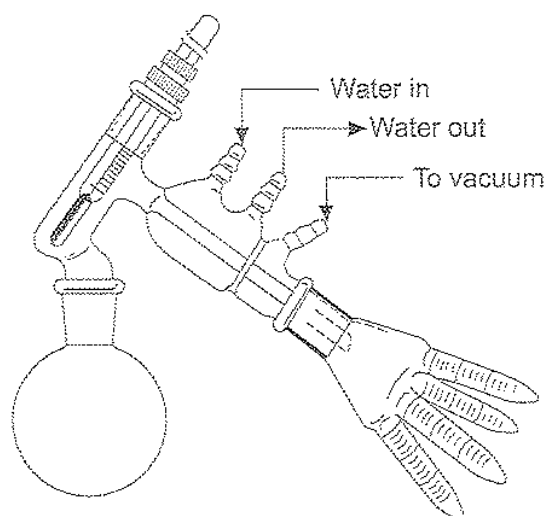
For small-scale work, the apparatus generally used is shown in Figure 2.8. It consists of a round bottomed flask with a long fractionating sidearm packed with  $3/16" \times 3/16"$  glass or porcelain rings.



**Fig. 2.8** Apparatus for micro-scale fractional distillation

A special receiver may be used to collect the fractions of different boiling ranges of a mixture without interrupting the distillation process, which is shown in Figure 2.9.





**Fig. 2.9** Micro-scale distillation apparatus with a rotating fractional collector

### 2.4.1 Exercise in Fractional Distillation

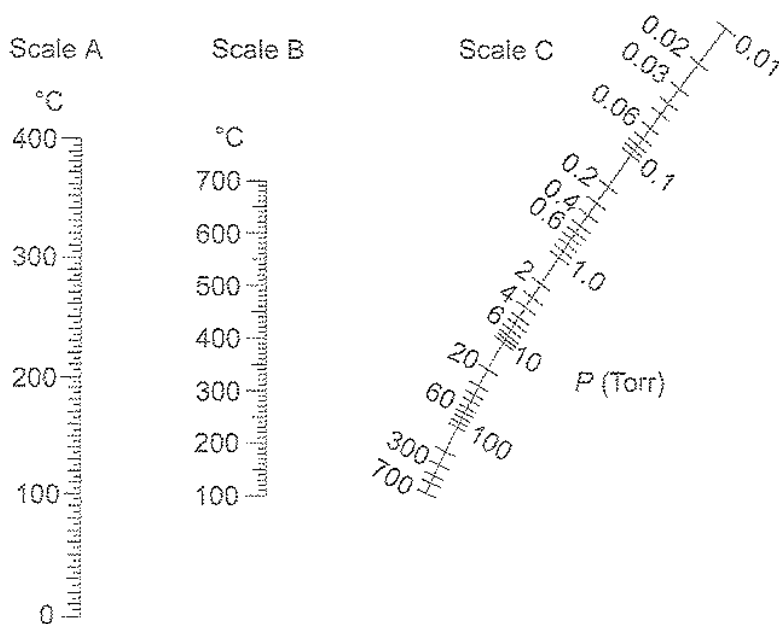
#### (a) *Fractional distillation of a mixture of benzene and toluene*

The boiling point of benzene is  $80^{\circ}\text{C}$  while that of toluene is  $110^{\circ}\text{C}$  and as the b.pt. difference is  $30^{\circ}\text{C}$  (less than  $50^{\circ}\text{C}$ ) and so the mixture can be separated by fractional distillation. It is carried out by using the apparatus shown in Figure 2.7, and here the lagging of the column is not required. For about 40 ml of a mixture containing equal volumes of benzene and toluene, about one and a half hour is needed if distillation is carried out slowly. The flame should have no draughts during the process. Fractions of boiling points (i)  $80\text{-}85^{\circ}\text{C}$ , (ii)  $85\text{-}107^{\circ}\text{C}$  and (iii)  $107\text{-}111^{\circ}\text{C}$  are collected in different receivers. The approximate volumes of each fraction are 19, 2 and 17 ml respectively.

## 2.5 DISTILLATION IN VACUUM

Some organic compounds decompose before their b.pt. are reached and so they cannot be distilled under atmospheric pressure. In such cases, the compound is distilled under reduced pressure so the boiling point is reduced and hence can be distilled without decomposition e.g. aniline has a boiling point  $184^{\circ}\text{C}$  at 760 mm Hg while it falls to  $151^{\circ}\text{C}$  at 300 mm Hg and to  $77^{\circ}\text{C}$  at 155 mm Hg.

Generally, as the external pressure is reduced by a factor of two, the boiling point of a compound will decrease by about  $20\text{-}30^{\circ}\text{C}$ . A nomograph (shown in Fig. 2.10) is useful for estimating the boiling point as a function of pressure. It estimates not only the expected reduced pressure boiling points from the normal boiling points but also the normal boiling points



**Fig. 2.10** A Nomograph for estimating the boiling point as a function of pressure

from observed reduced pressure boiling points. It is applicable for non-associated liquids only. For associated liquids, the variation of boiling point with pressure is 10-20% less than that for non-associated liquids.

The nomograph relates the normal boiling point of a substance (Scale B) to boiling points at reduced pressures (Scales A and C). A line joining points on two scales will intersect the third scale at some point. So, if the values of A and C are known (boiling point A at reduced pressure C), B (normal boiling point) can be estimated and if the values of B are known, boiling point A at a reduced pressure C can be estimated.

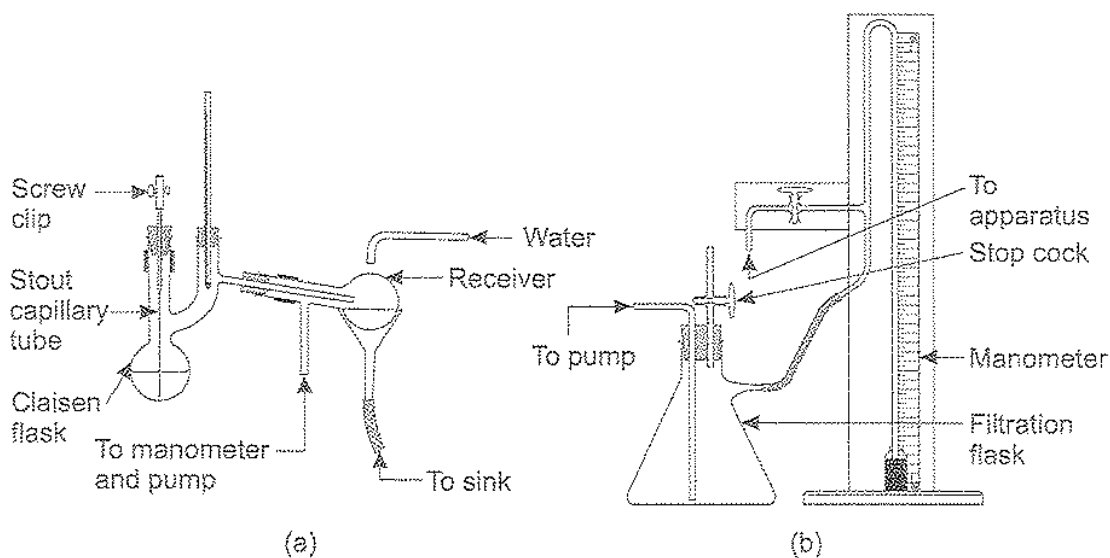
Table 2.2 shows the approximate boiling points of some of the substances at reduced pressure.

Apparatus employed for such type of distillation is shown in the Figure 2.11 (a-b).

A specially designed flask, known as Claisen flask is used [As a liquid can bump violently when superheated even under normal distillation process so the possibility of bumping is greatly increased when distillation is carried out under reduced pressure, so to reduce the risk of superheating and hence danger of bumping, Claisen flask is used]. Claisen flask has two necks. The right-hand neck carries a thermometer with the bulb just below the level of the side arm, while the left hand one is carrying a stout capillary tube, whose upper end is closed by a short piece of pressure tubing and a screw clip to regulate the amount of air passing through the capillary. The capillary used here should not be prepared by ordinary glass "quill" tube, as

**Table 2.2** Boiling points (°C) at reduced pressures

S.No.	Name of the Compound	Boiling point in °C at reduced pressure in mmHg [bp <sub>red pressure (mmHg)</sub> ]
1.	Water	bp <sub>5</sub> 1°/bp <sub>20</sub> 22°/bp <sub>25</sub> 26°/bp <sub>760</sub> 100°
2.	DMF	bp <sub>10</sub> 40°/bp <sub>39</sub> 76°/bp <sub>760</sub> 153°
3.	DMSO	bp <sub>0.37</sub> 20°/bp <sub>5.11</sub> 56.6°/bp <sub>17.83</sub> 83°/bp <sub>760</sub> 189°
4.	Chlorobenzene	bp <sub>5</sub> 10°/bp <sub>25</sub> 39°/bp <sub>50</sub> 54°/bp <sub>760</sub> 132°
5.	Formamide	bp <sub>10</sub> 109.5°/bp <sub>66</sub> 147°/bp <sub>200</sub> 175.5°/bp <sub>760</sub> 210.5°
6.	Glycerol	bp <sub>10</sub> 167°/bp <sub>20</sub> 182°/bp <sub>30</sub> 192°/bp <sub>760</sub> 290°
7.	Quinoline	bp <sub>17</sub> 114°/bp <sub>40</sub> 136.7°/bp <sub>100</sub> 163.2°/bp <sub>760</sub> 237.7°
8.	Ethyl salicylate	bp <sub>10</sub> 105°/bp <sub>25</sub> 124°/bp <sub>50</sub> 139°/bp <sub>760</sub> 234°
9.	Diglyme (Dimethyl ether of diethylene glycol)	bp <sub>3</sub> 20°/bp <sub>35</sub> 75°/bp <sub>200</sub> 116°/bp <sub>760</sub> 162°

**Fig. 2.11 (a-b)** Apparatus for vacuum distillation

by this method, capillary obtained will be very fragile and probably may snap during the course of the distillation. It should always be prepared by drawing out a piece of thick-walled capillary tube of 3-5 mm external bore and then the length of the fine drawn-out capillary is adjusted so that it reaches to within 1-2 mm of the bottom of the flask. Then, the side arm of the Claisen flask is fitted to an ordinary distillation flask, acting as a receiver, in such a way that it is well into the bulb of the receiver for the complete condensation of the vapours. Well-fitting and well-bored rubber stoppers and heavy-walled rubber tubing should be used throughout the apparatus. Cooling of the receiver is essential for complete condensation of

the vapours and so for liquids having boiling points below 140-150°C, the bulb of the receiver is cooled with a stream of water and the waste water is collected by funnel below, as shown in Figure 2.11 (a) and then carried to the sink with the help of the rubber tubing while for higher-boiling liquids, cooling is usually carried out by dipping the bulb of the receiver in cold water.

The distilling flask (receiver) is then fitted to filter flask, acting as a reservoir to equalize the pressure, by a pressure tubing, which is then connected to a manometer and a water pump as shown in the Figure 2.11 (b). The glass tube, which connects the filter flask to the pump, should touch the bottom of the flask so that any water, which may flow back due to the unequal pressure of water, may be sucked back as the water pressure retains. The larger flow back of water may be checked by opening the stop-cock (connected to the filtration flask) until the original water pressure is restored.

The reduction of pressure is carried out by using a water pump or oil pump. In water pump, the vapour pressure of water at that temperature is the lowest pressure that can be achieved e.g. in winter, the temperature of water is 6-8°C and the vapour pressure of water at this temperature is 6-8 mm, while in summer, when the temperature of water may be 22°C, the vapour pressure of water is 22 mm and therefore, in a water pump, the pressure varies from 7 to 22 mm. For lower pressures, oil pump should be used.

To start the distillation, first the liquid is placed in a Claisen flask (it should not be more than half-filled) and few pumice stones are added to it, the complete apparatus for distillation is fitted as shown in Figure 2.11 (a-b). Reading of barometric pressure is noted down. Now, the flask is heated by an air bath (Figure 1.43) or by a water or an oil bath, by immersing two-thirds of the flask into the bath. The water is supplied to the condenser and then water pump is turned on to attain its maximum capacity, keeping screw clip almost fully closed.\* A fine steady stream of air bubbles is passed through the solution by adjusting the screw clip. This will minimise the bumping as the introduction of air prevents the delay in the appearance of the vapour phase and thus superheating. And the volume of air passed in the form of bubbles is so small that there is almost negligible

---

\*If the flask contains traces of volatile solvents, warm the flask slightly and pass a comparatively large volume of air through liquid, so that the volatile solvents get removed down the water pump, otherwise the pressure obtained will have value above the capacity of the pump which can lead to either non-functioning of the pump or leakage in the apparatus. After the removal of all traces of volatile solvent, the screw clip is either almost completely closed or adjusted.

effect of partial pressure on the boiling point when the mercury level in the manometer is constant, the reading is noted. To obtain the value of pressure in the system, the above value is subtracted from atmospheric pressure. The apparatus set-up is considered to be satisfactory if the pressure obtained does not differ by more than 10 mm from the expected value [the temperature of the tap water], while if the pressure is unsatisfactory, the apparatus must be checked for leaks that all glass joints are firmly in position and proper fitting of all the pressure tubings over glass tubings. After achieving the satisfactory pressure, heating of flask is carried out by using a water bath, oil-bath or air bath. When water-bath or air-bath is used, the temperature of the bath should be 20-25°C above the boiling point of the liquid at that pressure, while if an air-bath is used, the temperature is raised slowly until the liquid starts to distil and heating is maintained so that the liquid distils at the rate of 1-2 drops per second. During the distillation, reading of temperature and pressure should be recorded by the thermometer and the manometer respectively. During the distillation, the boiling point of the pure compound should not be raised more than one to two degree, even if the temperature of bath is increased in the end to take off the last drop of the liquid. Sometimes, the liquid distil out first is having the boiling point lower than expected and so in such cases, the heating is continued until the expected temperature is reached and then the liquid is collected in another receiver. To remove the receiver, first the flame is removed and the Claisen flask is allowed to cool slightly by lowering the bath or raising the flask, then vacuum is released gradually by opening the stopcock of the filter flask and screw clip of the filtration flask, the latter will help in preventing the flow of liquid in the capillary.

Now receiver is replaced, again all joints are tightly joined and the whole procedure mentioned above is repeated. When the pressure is having constant value, the heating is started again.

The boiling point at a given pressure may be estimated approximately for the working pressure of a water pump (10-25 mm) by assuming that one mm difference in pressure corresponds to one-degree difference in boiling point. And the more accurate method is by using the expression:

$$\Delta t = 0.00012 (760 - P) (T + 273)$$

where,  $\Delta t$  = correction in the °C to be applied to the observed boiling point  $T$  and barometric pressure is  $P$ .

For water, alcohols, acids and other associated liquids, the expression used is:

$$\Delta t = 0.00010 (760 - P) (T + 273)$$

## 2.6 FRACTIONAL DISTILLATION IN VACUUM

If more than one fraction distils, the whole process of simple vacuum distillation has to be stopped after collecting each fraction, as described above and this is the main disadvantage of the simple vacuum distillation. This can be overcome by using a 'Pig' shown in the Figure 2.12 in place of a receiver flask. So various fractions can be collected without disturbing the distillation.

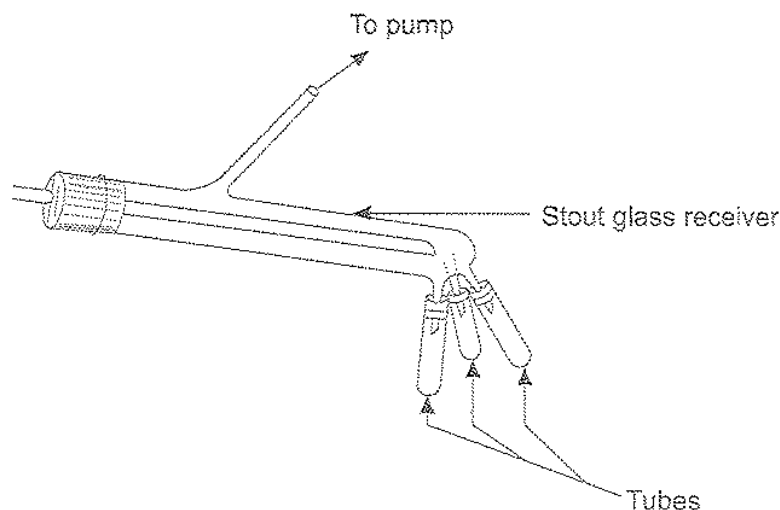


Fig. 2.12 Pig

The 'Pig' contains a glass receiver, which is fitted in place of a receiver flask. It has an outlet tube at the top for the connection to the manometer and pump, and three outlet tubes to which receivers are fitted. So when distillation starts first fraction is collected in one tube and when the second fraction starts coming, then the tube is rotated slightly to bring another outlet in the lowest position so as to collect second fraction in it, similarly third fraction can be collected in third outlet tube. However, this method is unsuitable as if a good vacuum is obtained in the apparatus, rotation of the tube is difficult as then it will be firmly attached while if the rotation is easy around the cork, it means that leakage is there at this point and so varying pressure is recorded by the manometer. The ground glass fitting shown in Figure 2.13 can overcome the leakage problem.

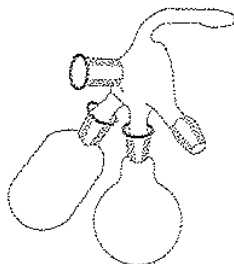
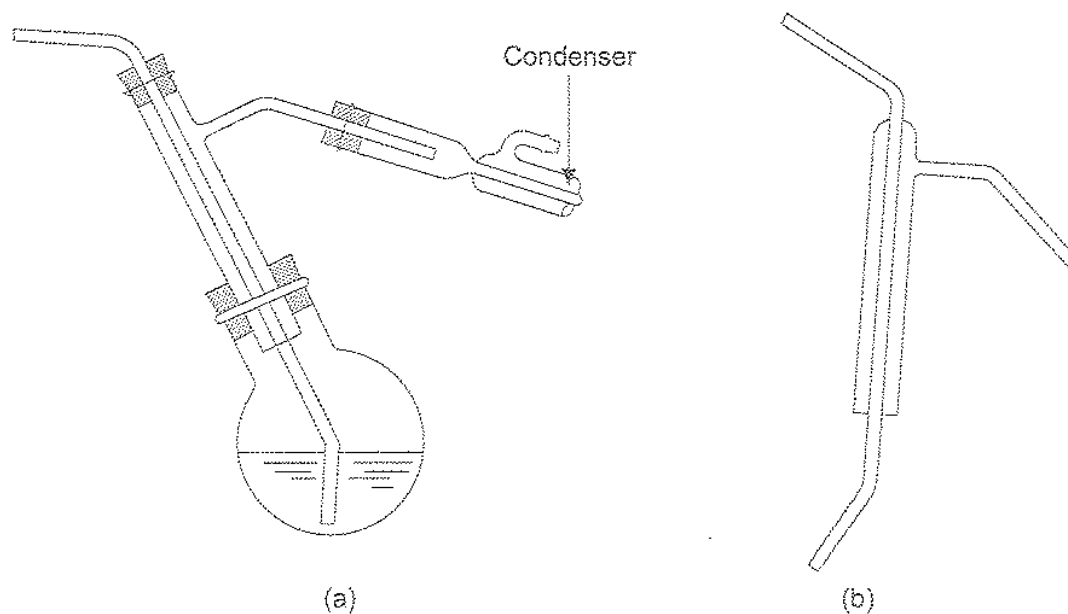


Fig. 2.13 Fractional collector

The isolation of the pure organic compound from the steam-distillate depends on the physical state and its solubility in water. The methods of isolation employed are:

- (a) If the compound is water insoluble liquid, it will form a separate layer so can be separated by separating funnel.
- (b) If compound is water insoluble solid, it will crystallize out in the aqueous distillate, so can be separated by filtration.
- (c) If water-soluble organic compound, then the compound can be isolated by solvent extraction [See Sec. 2.15].

To minimize the probability of the splashing of material over into the condenser, the distillation head shown in Figure 2.17 (a) may be used while the probability of leakage problems may also be reduced by using the all-glass head as shown in Figure 2.17(b).



**Fig. 2.17 (a-b)** Types of distillation head

When the steam distillation is carried out for small quantities, the apparatus, which may be used, is shown in the Figure 2.18. The substance to be distilled is placed in the inner tube, which is dipped in water of the outer flask.

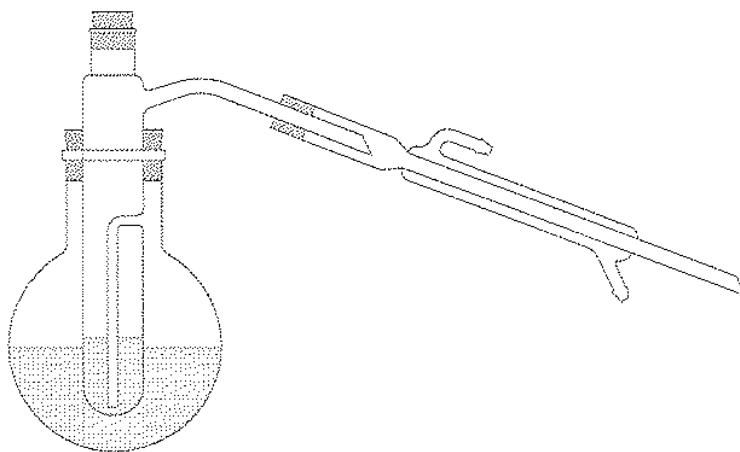
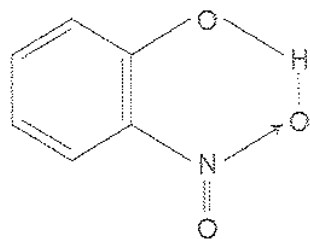


Fig. 2.18 Apparatus for micro scale steam distillation

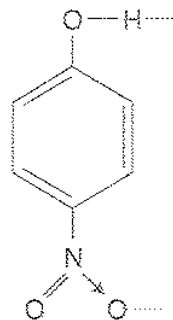
### 2.7.1 Exercises on Steam Distillation

#### (a) Separation of *o*- and *p*-nitro phenol

These two can be separated by steam distillation as the ortho compound contains an intermolecular hydrogen bonding, between oxygen of nitro group and hydrogen of OH group in the ring, while in case of para intermolecular hydrogen bonding is present. This results in high volatility of *o*-isomer in comparison to *p*-isomer.



(Intramolecular H-bonding)



(Intermolecular H-bonding)

These nitro phenols can be prepared by nitration of phenol. So the mixture obtained by the nitration is steam distilled as discussed above until no more *o*-nitro phenol passes over to the receiver flask. If it gets condensed in the condenser, then the flow of water in the condenser is turned off for a few moments. Distillate is cooled in the ice water for complete solidification of *o*-nitro phenol. It is filtered at the pump and drained thoroughly. The yield of the dry *o*-nitro phenol is 7g, if the

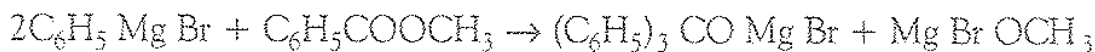


nitration is carried out with 20 g of phenol. The m.pt. of pure o-nitro phenol is 46°C.

The p-nitro phenol is obtained by cooling the residue of the flask in ice for 20 minutes. The crude p-nitro phenol is filtered and its purification is carried out by boiling it with 200 ml of 2% HCl along with 1 g of decolourized charcoal for 5-10 minutes. It is filtered through a hot water funnel and filtrate is allowed to cool overnight. The pure p-nitro phenol crystals are filtered and dried. The yield of p-nitro phenol (m.pt. 112°C) is 4 g.

**(b) Purification of triphenyl carbinol from the unreacted starting material and the by-products**

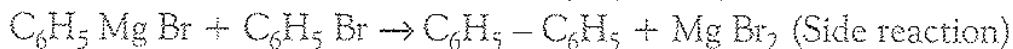
Triphenyl carbinol, a tertiary alcohol can be prepared by a Grignard reaction, shown below:



(Phenyl  
Magnesium  
Bromide)                      (Methyl benzoate)



(Triphenyl Carbinol)



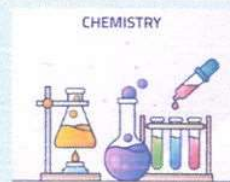
(biphenyl)

The residue obtained during this grignard reaction contains triphenyl carbinol, unreacted starting materials and biphenyl, as side product, out of which the former is non-volatile and the latter two are volatile, so can be separated by steam distillation.

To the residue [starting with 6 ml of bromobenzene] 30 ml of water is added and the mixture is placed for steam distillation as discussed above. Steam distillation is carried out until no more oil [unreacted starting material and biphenyl] passes over to receiver. On cooling the residue of the flask, solid separates out, which is filtered at the pump and dried. The triphenyl carbinol so obtained is colourless but crude and so it is crystallized from methylated spirit or benzene. The yield of pure triphenyl carbinol (m.pt. = 162°C) is 4.2 g.



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**Add On Course (Purification of Organic Compound)**

**TIME TABLE (3 Nov.- 25. Nov. 2021)**  
**(2021-22)(UG)**

<b>Class</b>	<b>Time</b>	<b>Friday(03/11/2021)</b>	<b>Saturday(04/11/2021)</b>
B.Sc.-III	11.20 am-12.08pm	Theory (SDJ)	Theory (NDN)
	12.08pm-12.56pm	Theory(NDN)	Theory(NMG)
	3.00 pm-6.00pm	Practical(SDJ)	Practical(NMG)

<b>Class</b>	<b>Time</b>	<b>Friday(10/11/2021)</b>	<b>Saturday(11/11/2021)</b>
B.Sc.-III	11.20 am-12.08pm	Theory (VSK)	Theory (NMG)
	12.08pm-12.56pm	Theory(PSP)	Theory(SDJ)
	3.00 pm-6.00pm	Practical(VSK)	Practical(NAG)

<b>Class</b>	<b>Time</b>	<b>Friday(17/11/2021)</b>	<b>Saturday(18/11/2021)</b>
B.Sc.-III	11.20 am-12.08pm	Theory (NDN)	Theory (NBP)
	12.08pm-12.56pm	Theory(NBP)	Theory(SSM)

	3.00 pm-6.00pm	Practical(NDN)	Practical(ABD)
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Class	Time	Friday(24/11/2021)	Saturday(25/11/2021)
B.Sc.-III	11.20 am-12.08pm	Theory (NDN)	Theory (VSK)
	12.08pm-12.56pm	Theory(SSM)	Theory(ABD)
	3.00 pm-6.00pm	Practical(PSP)	Practical(NMG)

SDJ - Dr. S. D. Jadhav

NDN - Dr. N. D. Nikam

NMG - Mr. N. M. Gosavi

VSK - Dr. V. S. Koshti

PSP - Dr. P. S. Patil

NAG - Miss. N. A. Ghadge

ABD - Miss. A.B. Deshmukh

NBP - Miss. N. B. Pawar

SSM - Miss. S. S. Mane

**Course  
Coordinator**

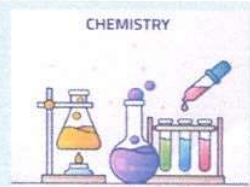
  
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**D. P. Bhosale College, Koregaon**

**Department of Chemistry**



(2021-22)

**Notice**

**Date :28/10/2021**

All the Students of B.Sc.-III (Chemistry) are here by informed that Department of Chemistry going to organize your Add On Course (Purification of Organic Compounds) has been Scheduled from 03/11/2021 to 25/11/2021 Kindly, remain present at prescribed time in lecture hall.

**Course  
Coordinator**

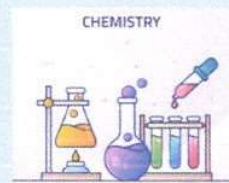
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**Department of Chemistry**




**Add On Course (Purification of Organic Compound)  
2021-22 (UG)  
Registration**

Sr. No.	Roll No.	Full Name
1	22561	Adsul Akash Uttam
2	22562	Ambekar Ganesh vijay
3	22563	Bandgar Shubhangi Dadasaheb
4	22564	Barge Adarsh Ramchndra
5	22565	Barge Rutuja Ramchadra
6	22566	Bhosale Sayalee Vikas
7	22567	Bhosale Shubham Shankar
8	22568	Chavan Akshay Rajendra
9	22569	Chavan Ketaki Vishal
10	22570	Dalavi Vivek Vishwanath
11	22571	Dhembare Priti Dipak
12	22572	Gaikwad Shivani Santosh
13	22573	Gavali Ankita Prsrharam
14	22574	Ghadage Prajwal Vaishwanath
15	22575	Ghadge Arpita Anandrao
16	22576	Ghadge Sonali Nandkumar
17	22577	Gharge Vaishnavi Nitin
18	22578	Ghorpade Swagat Chandrakant
19	22579	Ghorpade Vaishnavi Dilip
20	22580	Ingle Rohit Bajarang
21	22581	Jadhav Akanksha Arjun
22	22582	Jadhav Prachiti Shankar
23	22583	Jadhav Pradnya Sanjay
24	22584	Jadhav Sujata Santosh
25	22585	Jagadale Nikita Sambhaji
26	22586	Jagadale Prasad Dipak
27	22587	Jagtap Punam Ashok

28	22588	Jagtap Sakshi Vijay
29	22589	Jare Rutuja Dinesh
30	22590	Kadam Avadhut Anand
31	22591	Kenjale Rushikesh Anil
32	22592	Khatal Sanket Bhagvan
33	22593	Kudle Vaishnavi Rajesh
34	22594	Kumbhar Pratik Sandip
35	22595	Mahamuni Shraddha Vasant
36	22596	Manade Bapu Sunil
37	22597	Mane Ankita Chandrakant
38	22598	Mohite Shubham Shivaji
39	22599	More Vaibhav Shivaji
40	22600	More Vaishnavi Sandip
41	22601	Mulani Sameer Najeer
42	22602	Nade Bhagyashri Dattatray
43	22603	Naykude Nikita Gajanan
44	22604	Netke Akanksha Rajendra
45	22605	Nikam Ashwini Sunil
46	22606	Pawar Ankita Ashok
47	22607	Pawar Arati Ajay
48	22608	Pawar Nikhil Shankar
49	22609	Phadatare Onkar Namdev
50	22610	Phadtare Ankita Vitthal
51	22611	Pisal Suraj Pandurang
52	22612	Rasal Priti Appa
53	22613	Rathod Kanchan Pandurang
54	22614	Salunkhe Shubham Kalyan
55	22615	Sawant Aditi Adhik
56	22616	Shinde Anisha Sunil
57	22617	Shinde Harshada Vitthal
58	22618	Shinde Monali Sampat
59	22619	Shinde Urmila Manaji
60	22620	Shirke Dhanadhri Rajendrakumar
61	22621	Shirtode Arati Laxman
62	22622	Sonawale Kiran Shankar
63	22623	Tate Ashwini Vijay
64	22624	Tate Pranali Prabhakar
65	22625	Thorat Saurabh Sanjay
66	22626	Toraskar Sushant Dadaso

67	22627	Veer Akash Ashok
68	22628	Vibhute Snehal Jagannath
69	22629	Shinde Ganesh Bhimasen
70	22630	Kenjale Omkar Anandrao

**Course  
Coordinator**

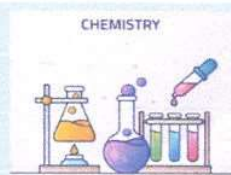
  
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**Department of Chemistry**



**Add On Course**  
**Question Paper (Purification of organic compound)**  
**2021-2022**

**Day & Date: 05/02/2022**  
**Time – 12:30pm to 01:00**

**Marks- 20**

- The IUPAC name of picric acid is
  - 2,4,6-trinitrophenol
  - 2,4,6-trinitrobenzoic acid
  - 4-nitrophenol
  - None of these
- 2.58 ml of N/5  $H_2SO_4$  are used to neutralize ammonia given by 1 g of organic compound. Percentage of nitrogen in the compound is
  - 34.3
  - 82.7
  - 16.2
  - 21.6
- IUPAC name of  $(CH_3)_2CH - CHO$  is:
  - 1-methyl-2 propanal
  - 2, 2-dimethyl propanal
  - None of these
  - 2-methyl propanal
- Which of the following relations gives the value of  $n =$ 
  - Molecular Mass / Atomic Mass
  - Molecular Mass / Empirical Mass
  - None of these
  - Empirical Mass / Molecular Mass
- 0.2595g of an organic substance in a quantitative analysis yielded 0.35 g of the barium sulphate. The percentage of sulphur in the substance is
  - 18.52g
  - 182.2 g
  17. 5 g
  - 175.2g
- f a compound on analysis was found to contain  $C = 18.5\%$ ,  $H = 1.55\%$ ,  $Cl = 55.04\%$  and  $O = 24.81\%$ , then its empirical formula is



- (a)  $\text{CHClO}$
- (b)  $\text{CClOH}$
- (c)  $\text{CHOCl}$
- (d)  $\text{ClCHO}$

7. 16 mg of a compound on vaporisation in a Victor Meyer's apparatus displaces 44.8 ml of air measured at S.T.P. The molecular weight of the compound is

- (a) 116
- (b) 232
- (c) 58
- (d) 44.8

8. The vapour density of the methyl ester of an organic monocarboxylic acid is 37. What is the molecular weight of the acid

- (a) 46
- (b) 60
- (c) 70
- (d) 74

9. If 0.228 g of silver salt of dibasic acid gave a residue of 0.162g of silver on ignition then molecular weight of the acid is

- (a) 70
- (b) 80
- (c) 90
- (d) 10

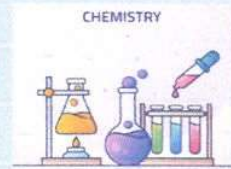
10. In Victor Mayer's method 0.2 gm of an organic substance displaced 56 ml of air at STP the molecular weight of the compound

- (a) 56
- (b) 112
- (c) 80
- (d) 28



Rayat Shikshan Sanstha's,

**D. P. Bhosale College, Koregaon**  
**Department of Chemistry**



**Add On Course**  
**Model Answer Paper (Purification of organic compound)**  
**2021-2022**

**Day & Date: 05/02/2022**

**Marks-20**

**Time – 12:30pm to 01:00pm**

1. The IUPAC name of picric acid is

- (a) 2,4,6-trinitrophenol
- (b) 2,4,6-trinitrobenzoic acid
- (c) 4-nitrophenol
- (d) None of these

2. 58 ml of N/5  $\text{H}_2\text{SO}_4$  are used to neutralize ammonia given by 1 g of organic compound. Percentage of nitrogen in the compound is

- (a) 34.3
- (b) 82.7
- (c) 16.2
- (d) 21.6

3. IUPAC name of  $(\text{CH}_3)_2\text{CH} - \text{CHO}$  is:

- (a) 1-methyl-2 propanal
- (b) 2, 2-dimethyl propanal
- (c) None of these
- (d) 2-methyl propanal

4. Which of the following relations gives the value of  $n$  =

- (a) Molecular Mass / Atomic Mass
- (b) Molecular Mass / Empirical Mass
- (c) None of these
- (d) Empirical Mass / Molecular Mass

5. 0.2595g of an organic substance in a quantitative analysis yielded 0.35 g of the barium sulphate. The percentage of sulphur in the substance is

- (a) 18.52g
- (b) 182.2 g

- (c) 17.5 g
- (d) 175.2g

6.f a compound on analysis was found to contain  $C = 18.5\%$ ,  $H = 1.55\%$ ,  $Cl = 55.04\%$  and  $O = 24.81\%$ , then its empirical formula is

- (a)  $CHClO$
- (b)  $CClOH$
- (c)  $CHOCI$
- (d)  $ClCHO$

7.16 mg of a compound on vaporisation in a Victor Meyer's apparatus displaces 44.8 ml of air measured at S.T.P. The molecular weight of the compounds is

- (a) 116
- (b) 232
- (c) 58
- (d) 44.8

8. The vapour density of the methyl ester of an organic monocarboxylic acid is 37. What is the molecular weight of the acid

- (a) 46
- (b) 60
- (c) 70
- (d) 74

9. If 0.228 g of silver salt of dibasic acid gave a residue of 0.162g of silver on ignition then molecular weight of the acid is

- (a) 70
- (b) 80
- (c) 90
- (d) 10

10. In Victor Mayer's method 0.2 gm of an organic substance displaced 56 ml of air at STP the molecular weight of the compound

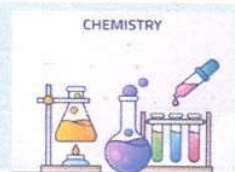
- (a) 56
- (b) 112
- (c) 80
- (d) 28



Rayat Shikshan Sanstha's,

**D. P. Bhosale College, Koregaon**

**Department of Chemistry**



**Add On Course (Purification of Organic Compound)  
2021-22 (UG) Result Analysis**

Sr. No.	Roll No.	Full Name	Marks	Grade
1	22561	Adsul Akash Uttam	16	B+
2	22562	Ambekar Ganesh vijay	18	A
3	22563	Bandgar Shubhangi Dadasaheb	20	A+
4	22564	Barge Adarsh Ramchndra	20	A+
5	22565	Barge Rutuja Ramchadra	16	B+
6	22566	Bhosale Sayalee Vikas	18	A
7	22567	Bhosale Shubham Shankar	16	B+
8	22568	Chavan Akshay Rajendra	16	B+
9	22569	Chavan Ketaki Vishal	14	B
10	22570	Dalavi Vivek Vishwanath	18	A
11	22571	Dhembare Priti Dipak	18	A
12	22572	Gaikwad Shivani Santosh	20	A+
13	22573	Gavali Ankita Prsharam	14	B
14	22574	Ghadage Prajwal Vaishwanath	16	B+
15	22575	Ghadge Arpita Anandrao	18	A
16	22576	Ghadge Sonali Nandkumar	20	A+
17	22577	Gharge Vaishnavi Nitin	14	B
18	22578	Ghorpade Swagat Chandrakant	16	B+
19	22579	Ghorpade Vaishnavi Dilip	18	A
20	22580	Ingle Rohit Bajarang	20	A+
21	22581	Jadhav Akanksha Arjun	14	B
22	22582	Jadhav Prachiti Shankar	18	A
23	22583	Jadhav Pradnya Sanjay	20	A+
24	22584	Jadhav Sujata Santosh	18	A
25	22585	Jagadale Nikita Sambhaji	16	B+
26	22586	Jagadale Prasad Dipak	16	B+
27	22587	Jagtap Punam Ashok	14	B
28	22588	Jagtap Sakshi Vijay	18	A
29	22589	Jare Rutuja Dinesh	18	A

30	22590	Kadam Avadhut Anand	20	A+
31	22591	Kenjale Rushikesh Anil	14	B
32	22592	Khatal Sanket Bhagvan	16	B+
33	22593	Kudle Vaishnavi Rajesh	18	A
34	22594	Kumbhar Pratik Sandip	20	A+
35	22595	Mahamuni Shraddha Vasant	14	B
36	22596	Manade Bapu Sunil	18	A
37	22597	Mane Ankita Chandrakant	16	B+
38	22598	Mohite Shubham Shivaji	16	B+
39	22599	More Vaibhav Shivaji	14	B
40	22600	More Vaishnavi Sandip	18	A
41	22601	Mulani Sameer Najeer	18	A
42	22602	Nade Bhagyashri Dattatray	20	A+
43	22603	Naykude Nikita Gajanan	14	B
44	22604	Netke Akanksha Rajendra	16	B+
45	22605	Nikam Ashwini Sunil	18	A
46	22606	Pawar Ankita Ashok	20	A+
47	22607	Pawar Arati Ajay	14	B
48	22608	Pawar Nikhil Shankar	18	A
49	22609	Phadatare Onkar Namdev	16	B+
50	22610	Phadtare Ankita Vitthal	16	B+
51	22611	Pisal Suraj Pandurang	14	B
52	22612	Rasal Priti Appa	18	A
53	22613	Rathod Kanchan Pandurang	18	A
54	22614	Salunkhe Shubham Kalyan	20	A+
55	22615	Sawant Aditi Adhik	14	B
56	22616	Shinde Anisha Sunil	16	B+
57	22617	Shinde Harshada Vitthal	18	A
58	22618	Shinde Monali Sampat	20	A+
59	22619	Shinde Urmila Manaji	16	B+
60	22620	Shirke Dhanadhri Rajendrakumar	18	A
61	22621	Shirtode Arati Laxman	16	B+
62	22622	Sonawale Kiran Shankar	16	B+
63	22623	Tate Ashwini Vijay	14	B
64	22624	Tate Pranali Prabhakar	18	A
65	22625	Thorat Saurabh Sanjay	18	A
66	22626	Toraskar Sushant Dadaso	20	A+
67	22627	Veer Akash Ashok	14	B
68	22628	Vibhute Sneha Jagannath	16	B+

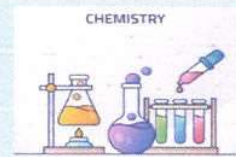
69	22629	Shinde Ganesh Bhimasen	18	A
70	22630	Kenjale Omkar Anandrao	20	A+

**Course  
Coordinator**

  
**Head**  
Department of Chemistry  
D. P. Bhosale College, Koregaon



Rayat Shikshan Sanstha's,  
**D. P. Bhosale College, Koregaon**  
**Department of Chemistry**



Purification of Organic Compound (UG)  
Report (2021-22)

The Organic Compounds which are prepared in Chemical Laboratory are either solids or liquids. These crude products are to be purified before spectral analysis. The compounds which are solids, can be recrystallized from a suitable solvents like water, acetone or alcohol as a single solvent or from mixed solvents like PET ether+ethyl acetate or Hexane+ diethyl ether. A care is to be taken while using volatile ethereal solvents.

The compounds which are liquids, and the colour impurity removed by heating with activated charcoal below the B.P. of the compound. The fractional distillation is utilised in order to separate two or more mixtures. It is based on the Principle that, the two miscible solvents having 10-15<sup>0</sup>C difference in B.P. can be separated with purification leaving behind non-volatile compound separated with colour impurities.

The medicinal plant extracts can be steam distilled to get steam volatile Organic Compound along with polar solvent like water. The method involves simple set up to use steam generator and distillation to get distillate.

About 70 students have been participated in the said course with actual demonstration and hands on training with proper guidance. After completion of the Course, certificates are conferred individually at the end of Course.

**Course  
Coordinator**

  
**Head**  
Department of Chemistry  
D. P. Bhosale College, Koregaon

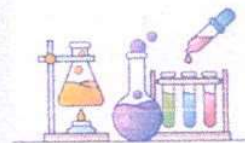


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**D. P. Bhosale College, Koregaon**

**Department of Chemistry**

CHEMISTRY



Purification of Organic Compound - (2021-22)

**Feedback**

Name Of Student	Jadhav Akanksha Arjun
Roll. No	
Mobile. No	-
Email. Id	-

Give your Valuable feedback marking the appropriate option With

Sr. No	Course Particulars	Excellent	Good	Satisfactory	Pour
1	Transparency in conduct of the course	✓		✓	
2	Syllabus	✓			
3	Topics Taught				
4	and Overall Management	✓	✓		
5	Overall impression				

Suggestion for improving, if any

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RAYAT SHIKSHAN SANSTHA'S

**D. P. BHOSALE COLLEGE, KOREGAON**

DIST-SATARA, MAHARASHTRA, INDIA-415501

DEPARTMENT OF CHEMISTRY

## Add on COURSE

### Certificate

This is to certify that, *Mr. Adsul Akash Uttam Class: B.Sc-III*  
Subject: *Chemistry* Successfully completed One month Add on Course on  
"Purification of Organic Compounds" with B+ grade Organized by  
Department of Chemistry, in November 2021.

**Mr. N. M. Gosavi**  
Course Coordinator

**Prof. Dr. S. D. Jadhav**  
HoD Chemistry

**Hon. Dr. V. S. Sawant**  
Principal



RAYAT SHIKSHAN SANSTHA'S

**D. P. BHOSALE COLLEGE, KOREGAON**

DIST-SATARA, MAHARASHTRA, INDIA-415501

DEPARTMENT OF CHEMISTRY

## **Add on COURSE**

### **Certificate**

*This is to certify that, Mr. Ambekar Ganesh Vijay Class: B.Sc-III  
Subject: **Chemistry** Successfully completed One month Add on Course on  
"Purification of Organic Compounds" with A grade Organized by  
Department of Chemistry, in November 2021.*

**Mr. N. M. Gosavi**  
**Course Coordinator**

**Prof. Dr. S. D. Jadhav**  
**HoD Chemistry**

**Hon. Dr. V. S. Sawant**  
**Principal**



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DEPARTMENT OF CHEMISTRY

## Add on COURSE

### Certificate

This is to certify that, *Miss. Bandgar Subhangi Dadasaheb* Class: **B.Sc-III** Subject: **Chemistry** Successfully completed One month Add on Course on "Purification of Organic Compounds" with A+ grade Organized by Department of Chemistry, in November 2021.

**Mr. N. M. Gosavi**  
Course Coordinator

**Prof. Dr. S. D. Jadhav**  
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DEPARTMENT OF CHEMISTRY

## **Add on COURSE**

### **Certificate**

*This is to certify that, Mr. Barge Adarsh Ranchendra Class: B.Sc-III Subject: Chemistry Successfully completed One month Add on Course on "Purification of Organic Compounds" with A+ grade Organized by Department of Chemistry, in November 2021.*

**Mr. N. M. Gosavi**  
**Course Coordinator**

**Prof. Dr. S. D. Jadhav**  
**HoD Chemistry**

**Hon. Dr. V. S. Sawant**  
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**Add on COURSE**

**Certificate**

*This is to certify that, Mr. **Adsul Akash Uttam** Class: **B.Sc-III**  
Subject: **Chemistry** Successfully completed One month Add on Course on  
"Purification of Organic Compounds" with B+ grade Organized by  
Department of Chemistry, in November 2021.*

**Mr. N. M. Gosavi**  
Course Coordinator

**Prof. Dr. S. D. Jadhav**  
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Principal