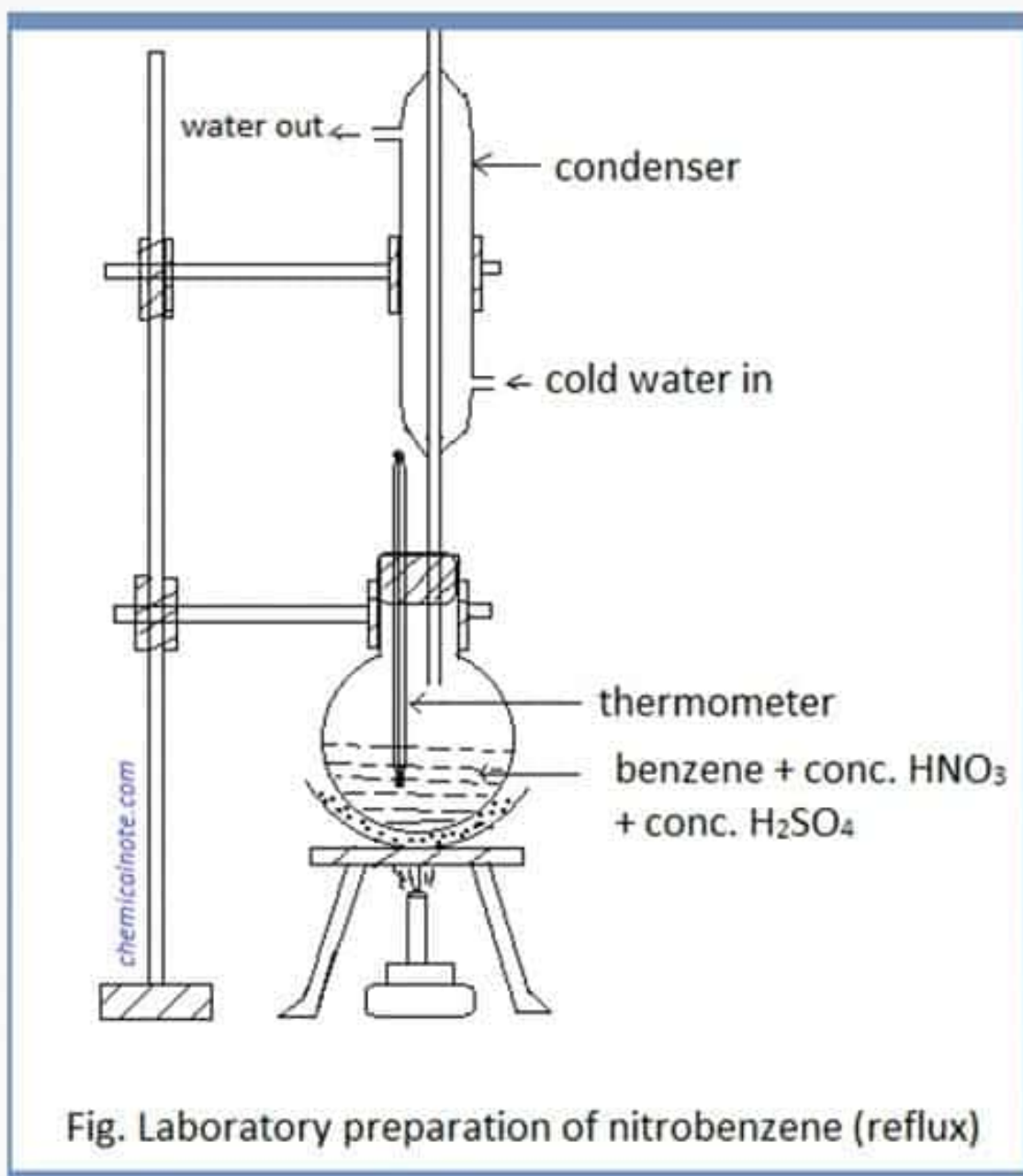
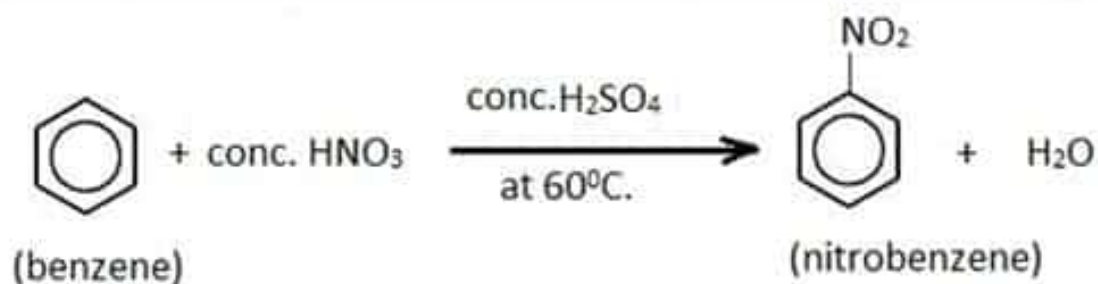


Laboratory Preparation of Nitrobenzene

It is prepared in lab by heating benzene with conc. HNO_3 and conc. H_2SO_4 at 60°C .



Procedure : 50 ml of benzene is taken in a round bottomed flask. To this flask, 60 ml conc. HNO_3 and 60 ml conc. H_2SO_4 (i.e. nitrating mixture) is added a little at a time, shaking and cooling after each addition. Then the mixture is heated (refluxed) in water bath at 60°C for about one and half hour till the yellow oily layer appears on the surface. The flask is then cooled and the layer of nitrobenzene is separated by using separating funnel

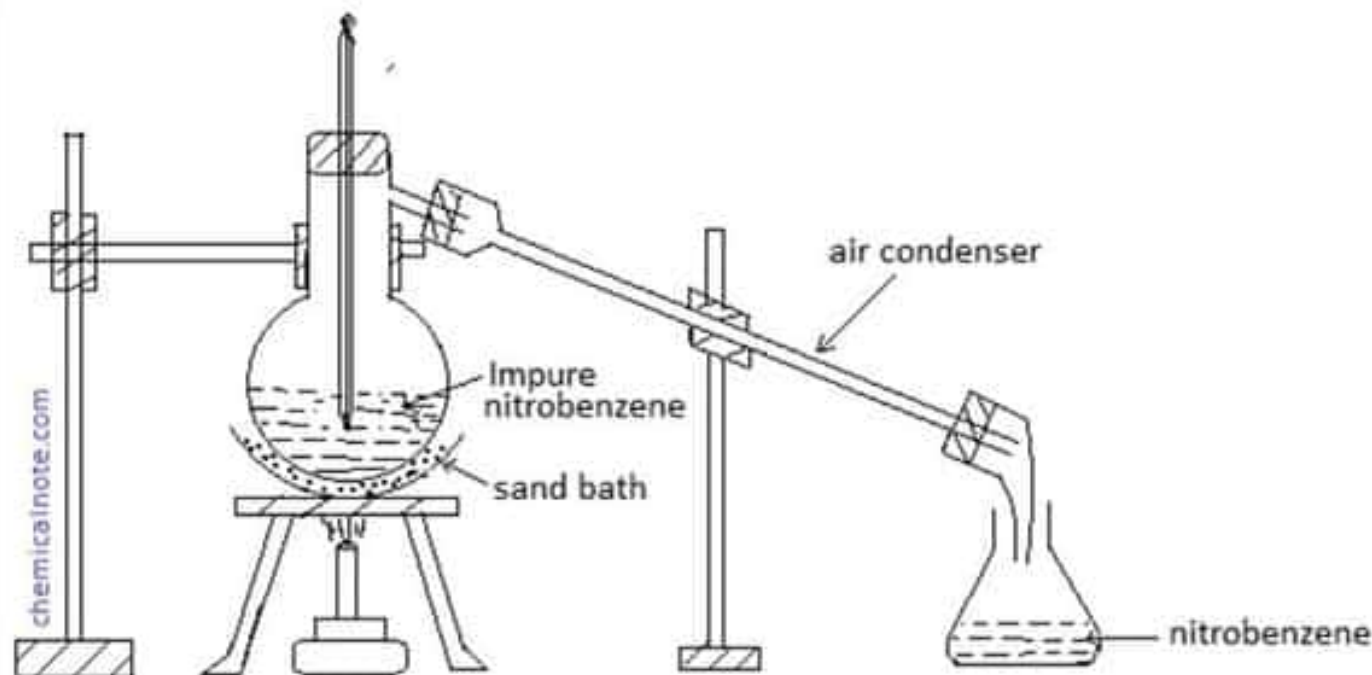


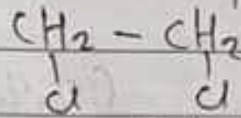
Fig. Distillation of nitrobenzene (for purification)

Purification : It is first washed with dil. Na_2CO_3 to remove the acidic impurities and then with water several times. It is then dried over fused calcium chloride. It is finally distilled at 211°C to get pure nitrobenzene.

polyhaloalkanes

The haloalkanes which have more halogen atoms as functional group are called polyhaloalkanes

Eg: $\text{CH}_2 - \text{Cl}_2$
Dichloromethane



1,2-dichloroethane

CHCl_3
Trichloromethane
(Chloroform)

CHBr_3
tribromomethane
(Bromoform)

CHI_3
Tri-iodomethane
(Iodoform)

CCl_4
Carbon tetrachloride
(Tetra chloro methane)

Laboratory preparation of chloroform or Trichloro methane (CHCl_3)

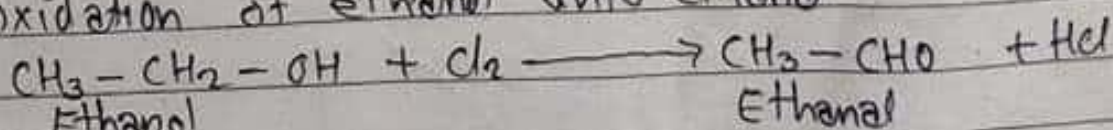
principle:

When ethyle alcohol (ethanol) or Acetone (propan one) is heated with paste of bleaching powder then chloroform or trichloromethane is formed where the paste of bleaching powder acts as oxidising, chloro chlorinating and hydrolysing agent.

(A) From ethanol or ethyl alcohol ($\text{CH}_3 - \text{CH}_2 - \text{OH}$)



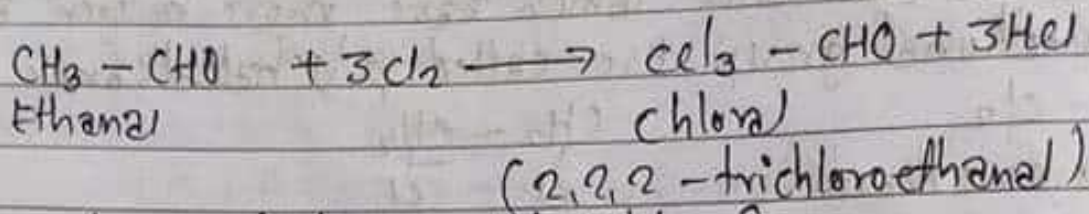
(1) Oxidation of ethanol into ethanal



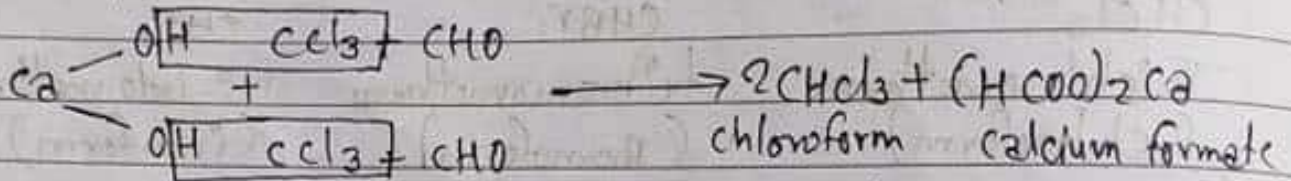
Ethanol

Ethanal

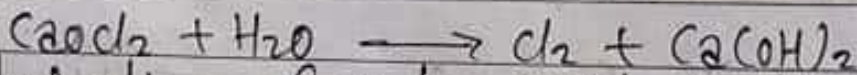
② chlorination of ethanal into chloral.



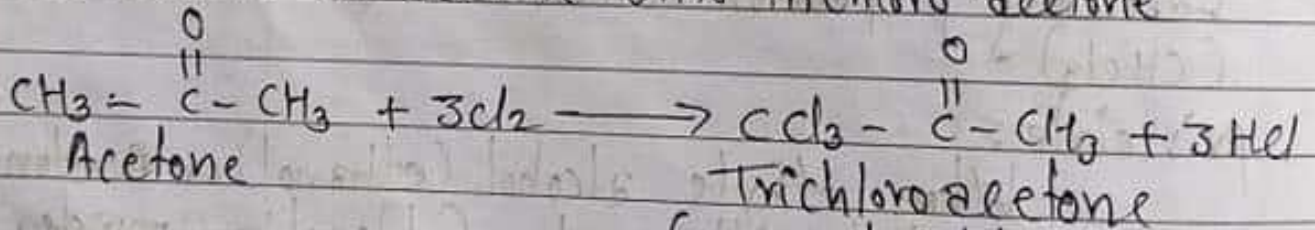
③ Hydrolysis of chloral into chloroform



[B] From acetone or propanone ($\text{CH}_3 - \overset{\text{O}}{\parallel} \text{C} - \text{CH}_3$)

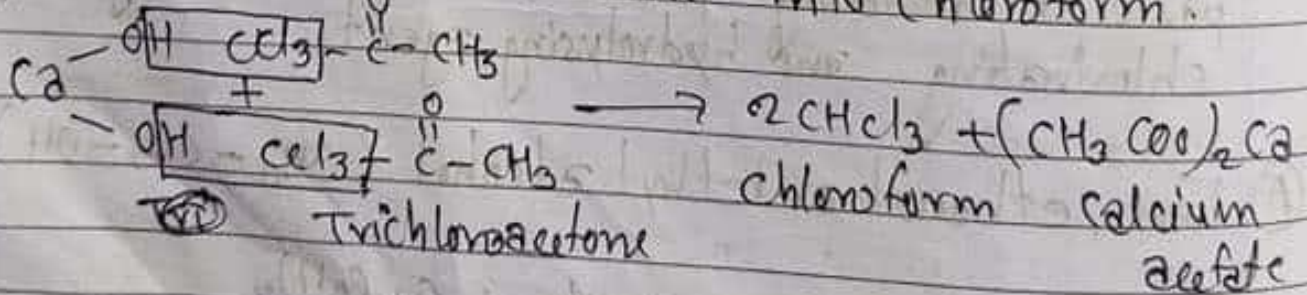


① chlorination of acetone into trichloro acetone



(T, T, T) trichloropropan-2-one

② Hydrolysis of trichloroacetone into chloroform:



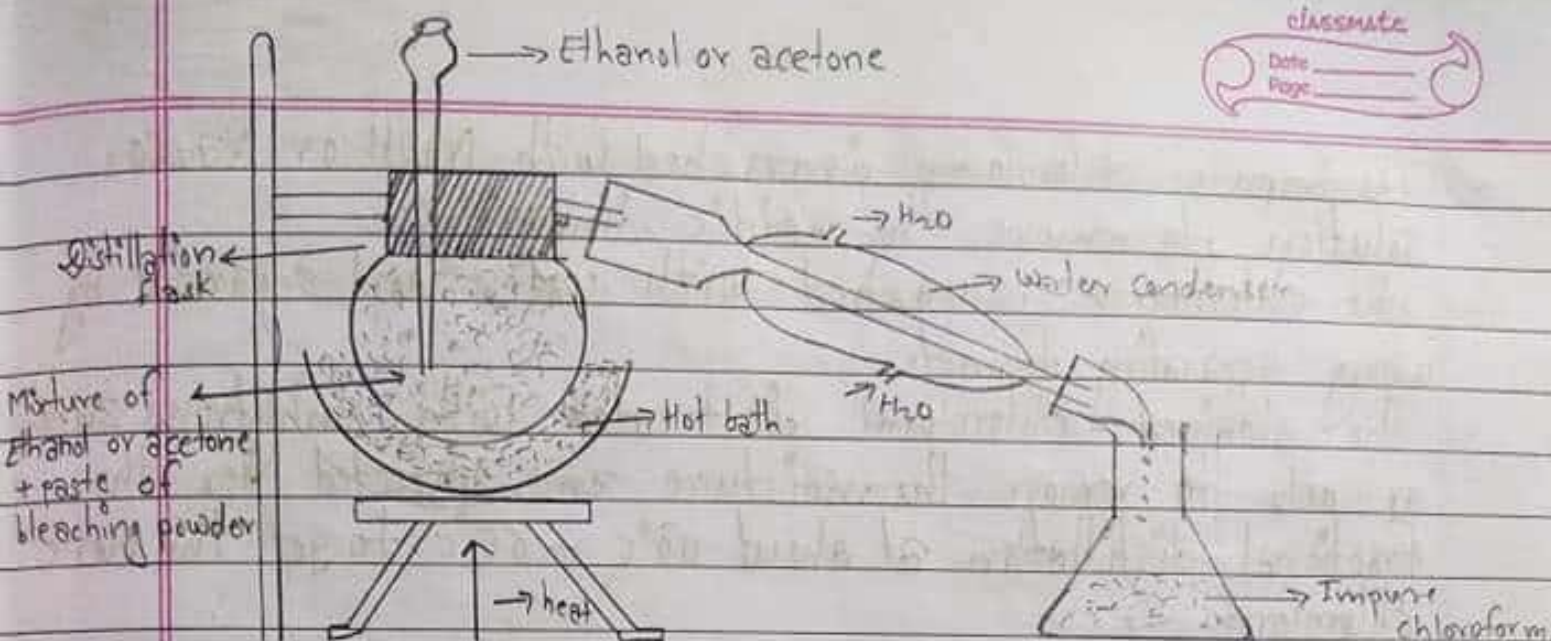


Fig: laboratory preparation of chloroform

The mixture of ethanol or acetone and paste of bleaching powder is taken in a distillation flask or round bottom flask and the apparatus fitted air tight as shown in figure. The mixture containing in the flask is heated over hot bath and the reaction occurs vigorously, the vapourised chloroform is made to pass into the water condenser where it is condensed into liquid form which is collected into receiver i.e. impure chloroform.

purification:

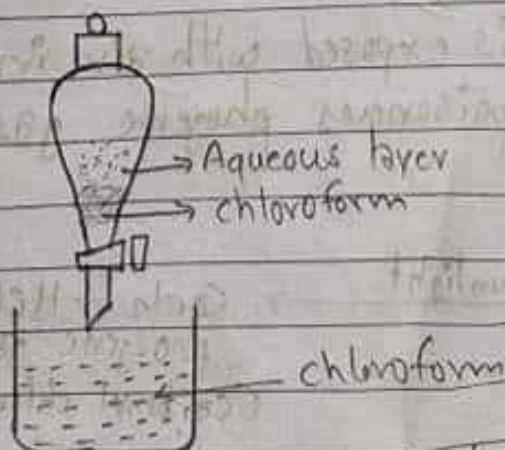


Fig: purification by using separating funnel

- The impure chloroform is washed with NaOH or Na_2CO_3 solution to remove the acidic impurities.
- The chloroform is washed with water and separated by using separating funnel.
- The obtained chloroform is treated with anhydrous ZnCl_2 or CaCl_2 to remove the moisture and subjected for the fractional distillation at about $60^\circ\text{C} - 65^\circ\text{C}$ to get the pure chloroform.

Physical properties

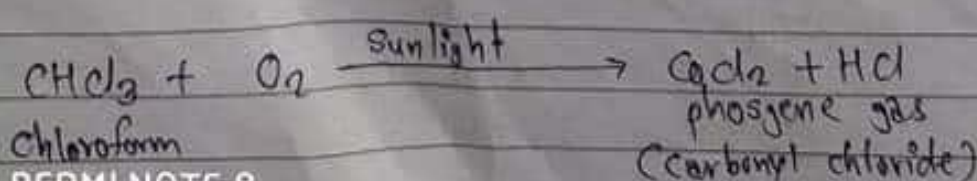
- It is a colourless, heavy, liquid with boiling point 63°C and freezing point -63°C .
- It is a sweet smelling liquid with burning taste.
- It is heavier than water.
- It is sparingly soluble in water but highly soluble in organic solvent like alcohol, ether, acetone etc.
- The vapours of chloroform is highly poisonous and may cause temporary unconsciousness.

Chemical properties

[7] Reaction with air (formation of phosgene gas)

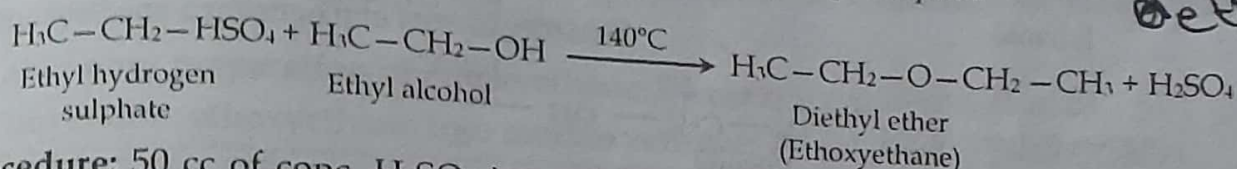
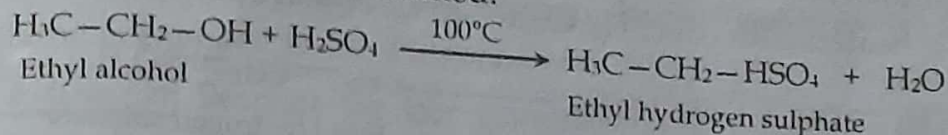
Chemical (Oxidation reaction)

When chloroform is exposed with air in presence of sunlight then the highly poisonous phosgene gas or carbonyl chloride is formed.



26. 2063 Q.No. 23 Describe the preparation of ethoxyethane in the laboratory. Ethanoylchloride [5]

Principle: When excess of ethyl alcohol (ethanol) is heated with conc. H_2SO_4 at 140°C , diethyl ether or ethoxyethane is obtained.



diethyl ether

Procedure: 50 cc of conc. H_2SO_4 is gradually added with constant shaking to 100 cc of ethyl alcohol kept in the distillation flask. The mixture is heated on a water bath at 140°C , when ether begins to distill over. Alcohol is added in the distillation flask from the dropping funnel at nearly the same rate as that of the distillation, the temperature being maintained at 140°C .

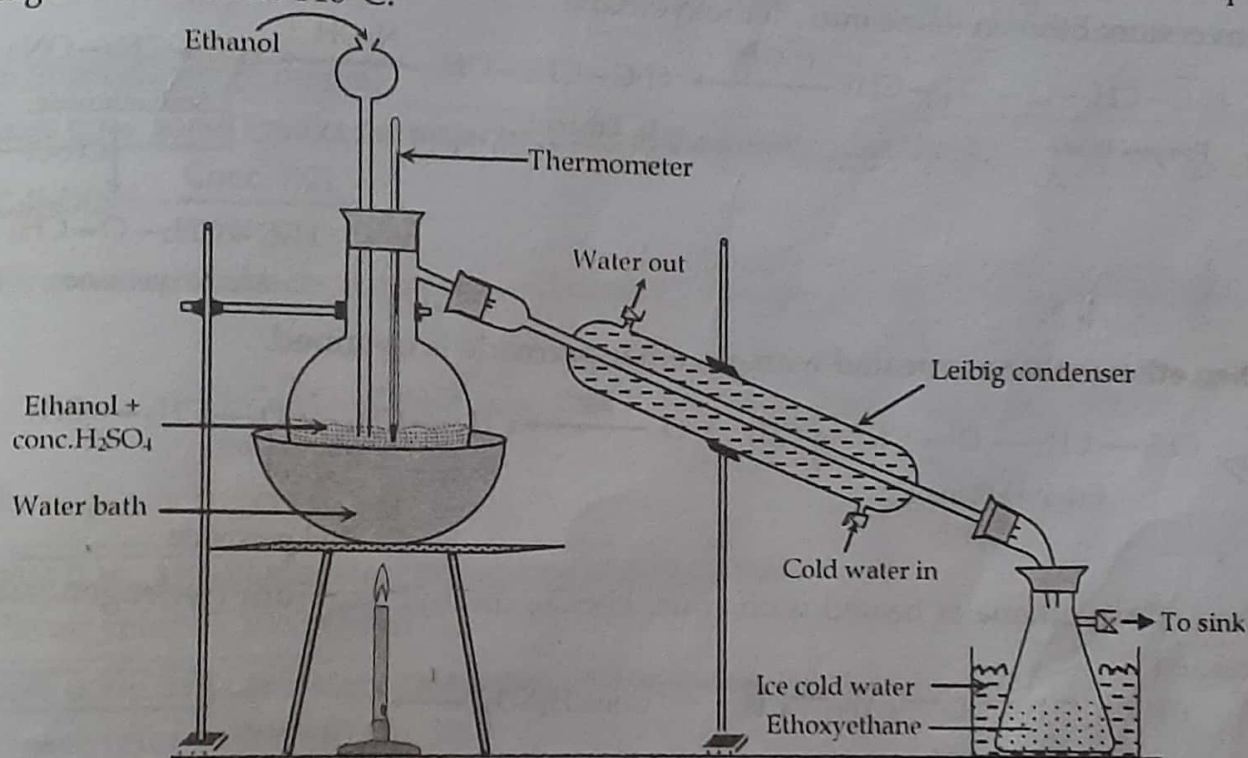


Figure: Laboratory preparation of ethoxyethane

Purification: The distillate contains ether, ethyl alcohol, water and sulphur dioxide. It is first washed with dil. NaOH solution and then with water. The upper layer is separated and dried over anhydrous CaCl_2 . It is then redistilled on a water bath pure ether passes over at $34-35^\circ\text{C}$.

27. [2063 Q.No. 24] Ethoxyethane into methoxyethane

[2.5]