

# **INDUSTRIAL CHEMISTRY**

## **CHAPTER-**

# **INDUSTRIAL ORGANIC SYNTHESIS**

## **ONLINE LECTURE- NO. 1**

**DATE:- 16, OCTOBER 2021**

**TIME: (10.00A.M.)**

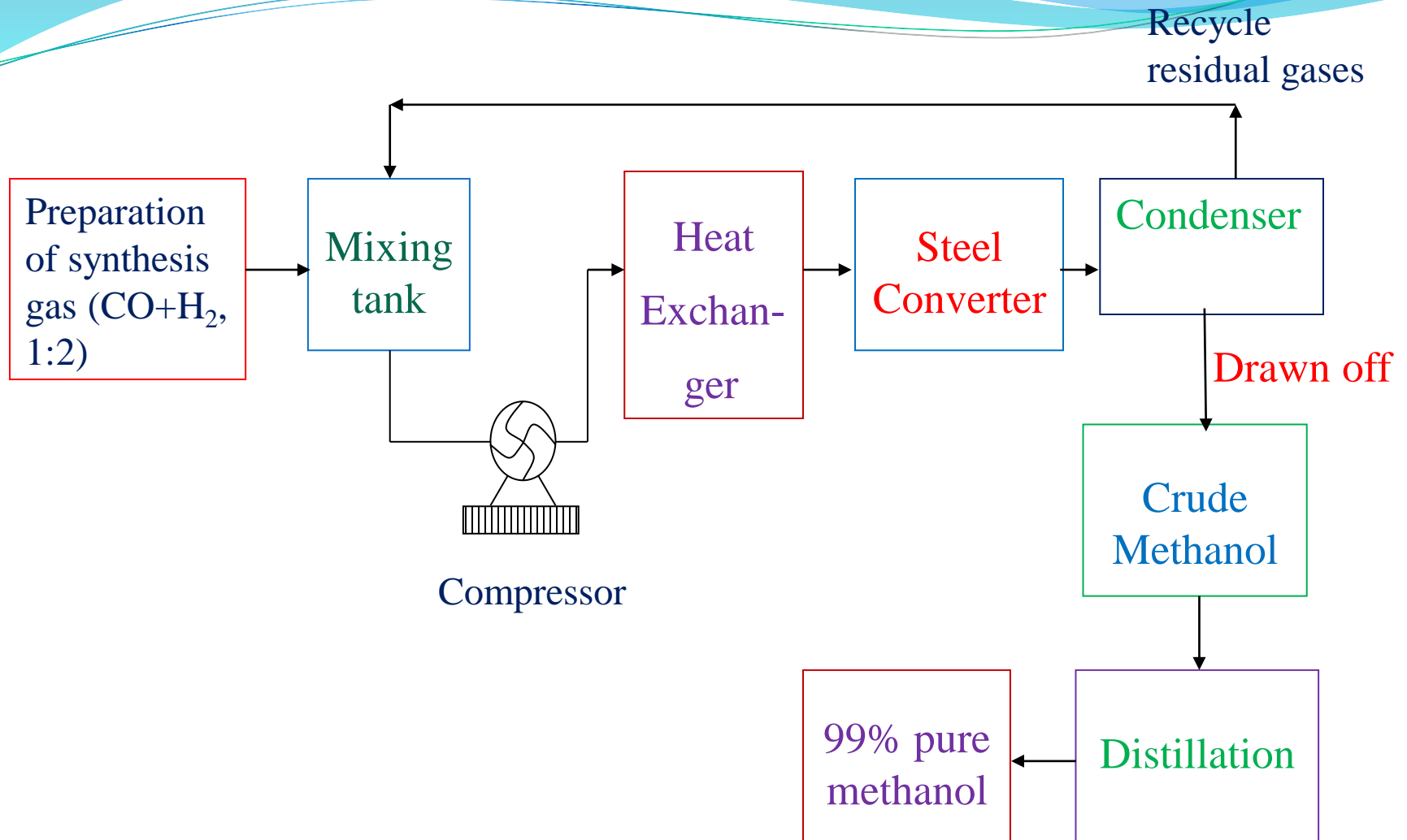
## **Manufacture of methanol [CH<sub>3</sub>OH] from synthesis gas:-**

**(From Carbon monoxide and Hydrogen):-**

**Principle: -** Methanol is synthesized from synthesis gas i.e. [CO + H<sub>2</sub>] under high pressure and in the presence of a catalyst.

**Reaction: -**  $\text{CO} + 2\text{H}_2 \rightarrow \text{CH}_3\text{OH}$   $\Delta\text{H} = - 24.6 \text{ KCal}$

**Raw materials: -** Natural gas(CH<sub>4</sub>), air, CO, H<sub>2</sub>, ZnO + Cr<sub>2</sub>O<sub>3</sub> catalyst.



**Fig.: Flow Chart of Preparation of Methanol from Synthesis Gas**

**Process:** - The preparation of methanol from synthesis gas involves following steps.

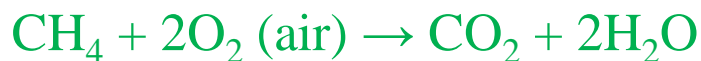
- |                                 |                    |
|---------------------------------|--------------------|
| a) Preparation of synthesis gas | b) Mixing tank     |
| c) Heat exchanger               | d) Steel converter |
| e) Condenser                    | f) Distillation    |

**a) Preparation of synthesis gas: -**

The H<sub>2</sub> and CO required are obtained from the natural gas. The natural gas is desulphurised by passing it over activated carbon, preheated and then mixed with steam and CO<sub>2</sub> under 2 atm. Pressure. This mixture is then passed through alloy steel tubes, which are packed with promoted Ni catalyst and then heated to about 800<sup>0</sup>c in a furnace. As a result the synthesis gas (CO +H<sub>2</sub>) is obtained.



The CO<sub>2</sub> gas required for the reaction is produced by burning natural gas in excess of air.



### **b) Mixing tank: -**

The obtained synthesis gas i.e. a mixture of CO and H<sub>2</sub> (1:2) but actually it is use in the ratio of 1:4 to 1:8 and mixed in mixing tank.

### **c) Heat exchanger: -**

The synthesis gas is compressed to 3000 – 5000 psi and then heated at 300°C by passing through a heat at exchanger.

### **d) Steel converter: -**

The preheated mixture is passed into a copper lined steel converter containing a bed of mixed catalyst i.e. zinc oxide (ZnO) with 10% chromium oxide (Cr<sub>2</sub>O<sub>3</sub>).

The reaction is highly exothermic and takes place with a decrease in volume. The enhanced pressure would, therefore, results in more favorable equilibrium (Le Chatelier principle), since the reaction is exothermic. Once the reaction has started, the temperature of the reaction is maintained at about 300°C by cooling.

**e) Condenser:** - The exhaust gases from converter are cooled to 0 – 20°C and then condensed in high pressure condensers, where methanol is condensed at 3000 – 4000 psi. The liquid methanol is drawn off and the residual gases are recycled.

**f) Distillation:** - The distillation of crude methanol gives 99% pure methanol. The yield is about 60% without recycling while others are obtained as byproducts such as Dimethyl ether (1-2%) and some higher alcohols like n-propanol and isopropanol (0.3-0.5%).

**Uses of Methanol:** - Methanol has a wide variety of direct industrial uses such as antifreeze for automobile radiators, varnishes, dyes, solvent for paints, lacquers and as a high octane admixture to motor fuels, etc.

It is an important raw material for the manufacture of

1. Formaldehyde
2. Methyl amine
3. Formic acid
4. Dimethyl ether
5. Methyl acetate
6. Dimethyl tetephtalate etc.

# **INORGANIC CHEMISTRY**

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# **INDUSTRIAL ORGANIC SYNTHESIS**

## **ONLINE LECTURE- NO. 2**

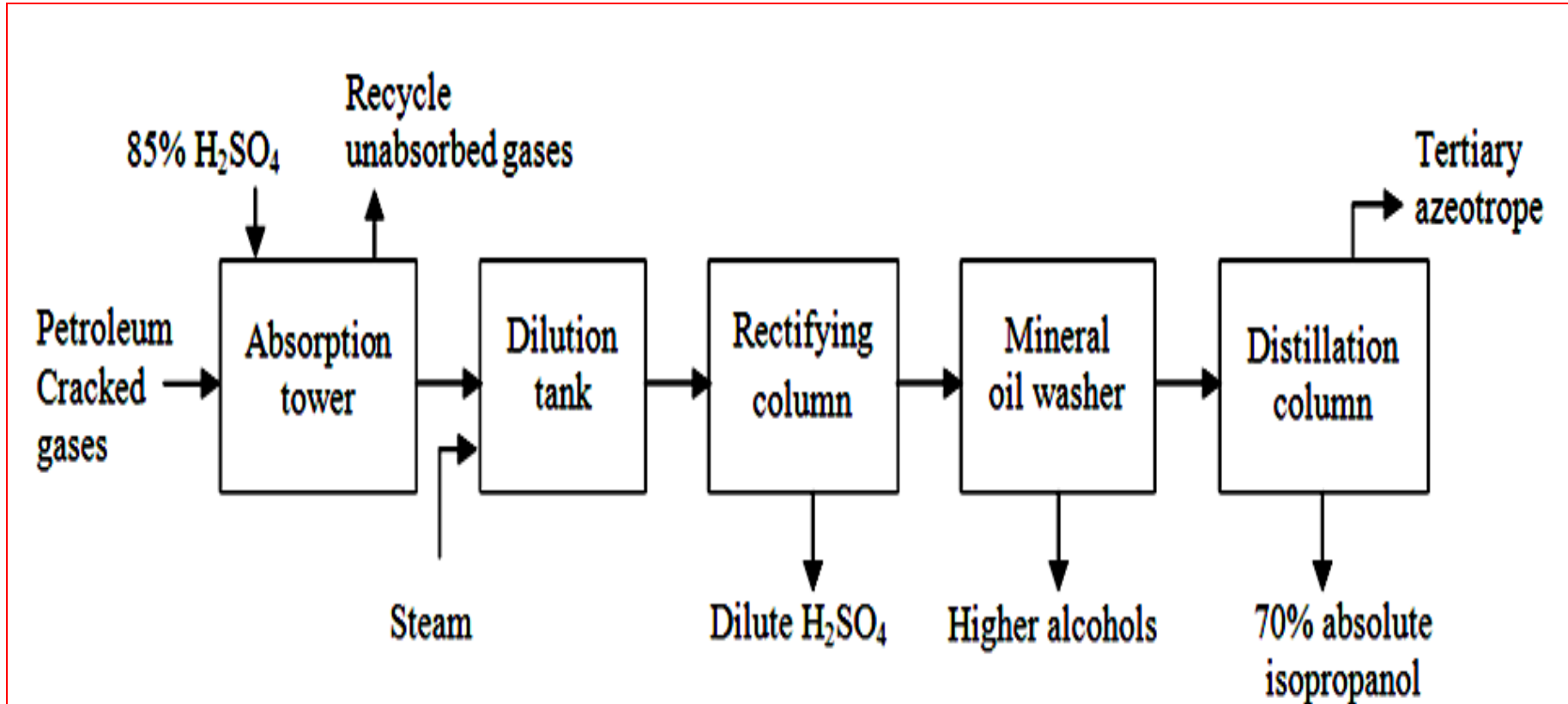
**DATE:- 18 OCTOBER 2021**

**TIME: (11.00A.M.)**





## Flowsheet: -



**Fig. : Flow Chart of Preparation of Isopropanol from Propylene**

**Process:** - The preparation of isopropanol involves the following steps.

- 1) Absorption tower
- 2) Dilution tank
- 3) Rectifying column
- 4) Mineral oil washer
- 5) Distillation column

**a) Absorption tower: -**

Firstly a mixture of petroleum cracked gases rich in propylene is desulphurised and then passed into the bottom of an absorption tower at 27°C and from the top of which 85%  $\text{H}_2\text{SO}_4$  trickles down. Sulphuric acid meets the propylene gas in a counter current manner and the reaction is exothermic in nature. Therefore, tower is cooled by circulating brine to prevent the rise in temperature. Nearly half of the propylene is absorbed in the sulphuric acid and isopropyl hydrogen sulphate is the main product formed with a little di-isopropyl sulphate. While unabsorbed gases are recycled.

### **b) Dilution tank: -**

The reaction mixture containing isopropyl hydrogen sulphate and di-isopropyl sulphate is removed from the bottom of the absorption tower and is charged into a closed lead lined tank, where the products are diluted to 20% strength with water. The heat of dilution hydrolyses the isopropyl hydrogen sulphate into isopropanol and sulphuric acid.

### **c) Rectifying or Distillation column; -**

The isopropyl alcohol is removed by passing steam through the reaction mixture and then purified by distillation. An azeotrope with 91% isopropanol and 9% water distills from the top of the rectifying column.

### **d) Mineral Oil Washer: -**

The above azeotropic mixture from rectifying column also contains some higher alcohols which are removed by passing an azeotropic mixture through a mineral oil washer, which selectively absorbs the higher alcohols.

### e) Distillation column: -

An absolute isopropanol (99% pure) is obtained by redistillation of above 91% isopropanol with a little di-isopropyl ether. At about 61.4°C, a ternary azeotrope (contains 91.1% di-isopropyl ether, 5.8% isopropanol and 3.1% water) is removed from the top and the anhydrous isopropanol is drawn off from the bottom of the distilling column. The yield of isopropanol is about 70% while the dilute H<sub>2</sub>SO<sub>4</sub> formed in the reaction is concentrated and then recycled to use again till tarry matter accumulates in it.

**Uses of Isopropanol: -** It is used in preparation of

- a) Isopropyl chloride
- b) Isopropyl amine
- c) Acetone, etc

It is also used as solvent, as an antifreeze and a component of shaving lotions, nail polishes etc.

# **INDUSTRIAL CHEMISTRY**

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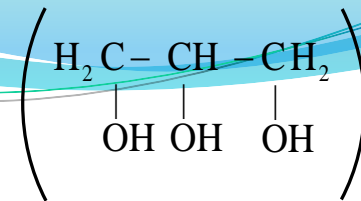
# **INDUSTRIAL ORGANIC SYNTHESIS**

## **ONLINE LECTURE- NO. 3**

**DATE:- 24, OCTOBER 2021**

**TIME: (9.00A.M.)**

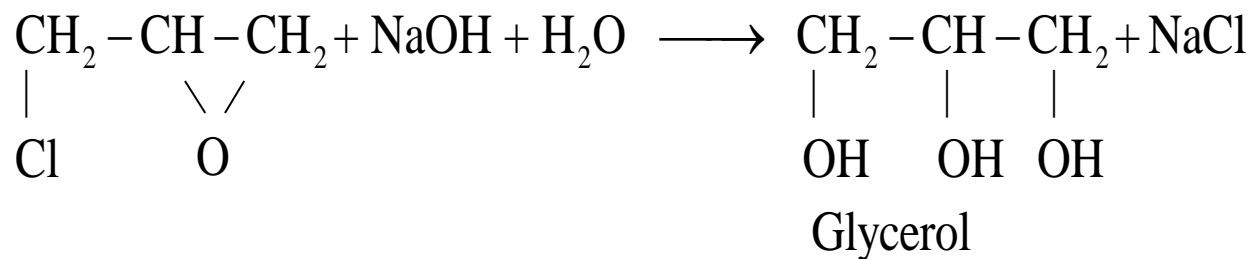
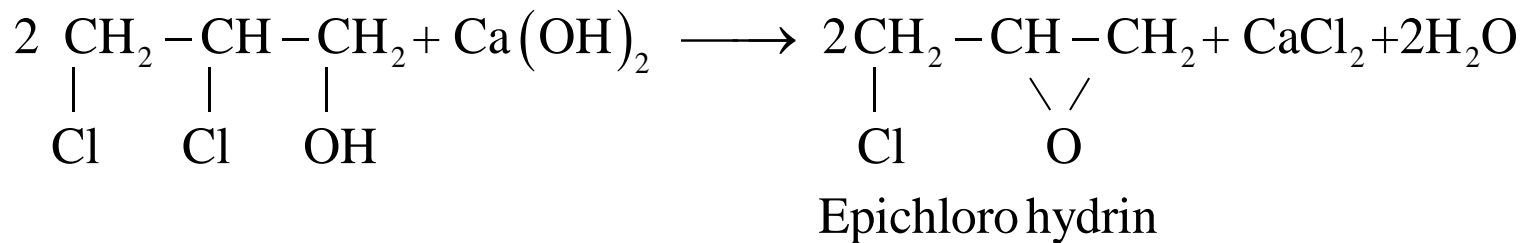
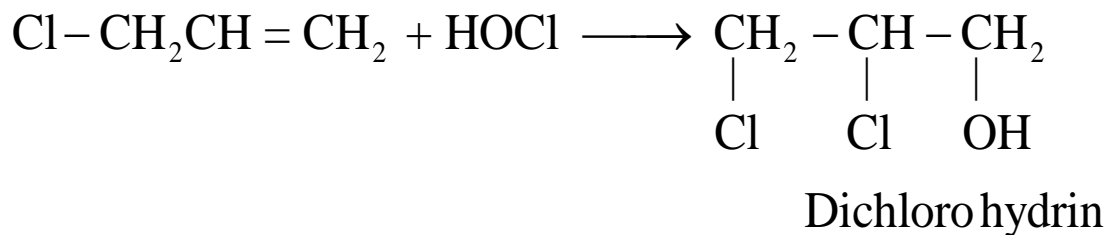
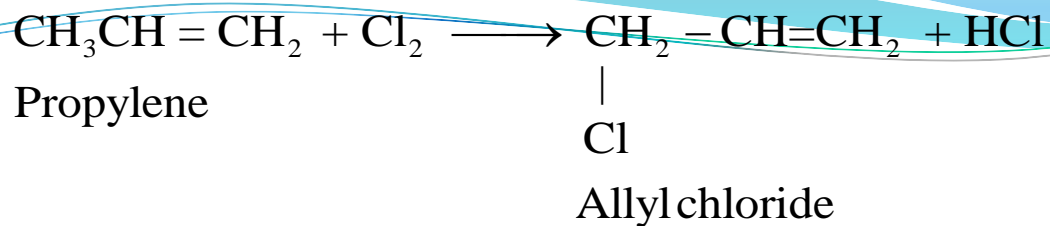
## Glycerine or Glycerol from Propylene via Allyl Chloride: -



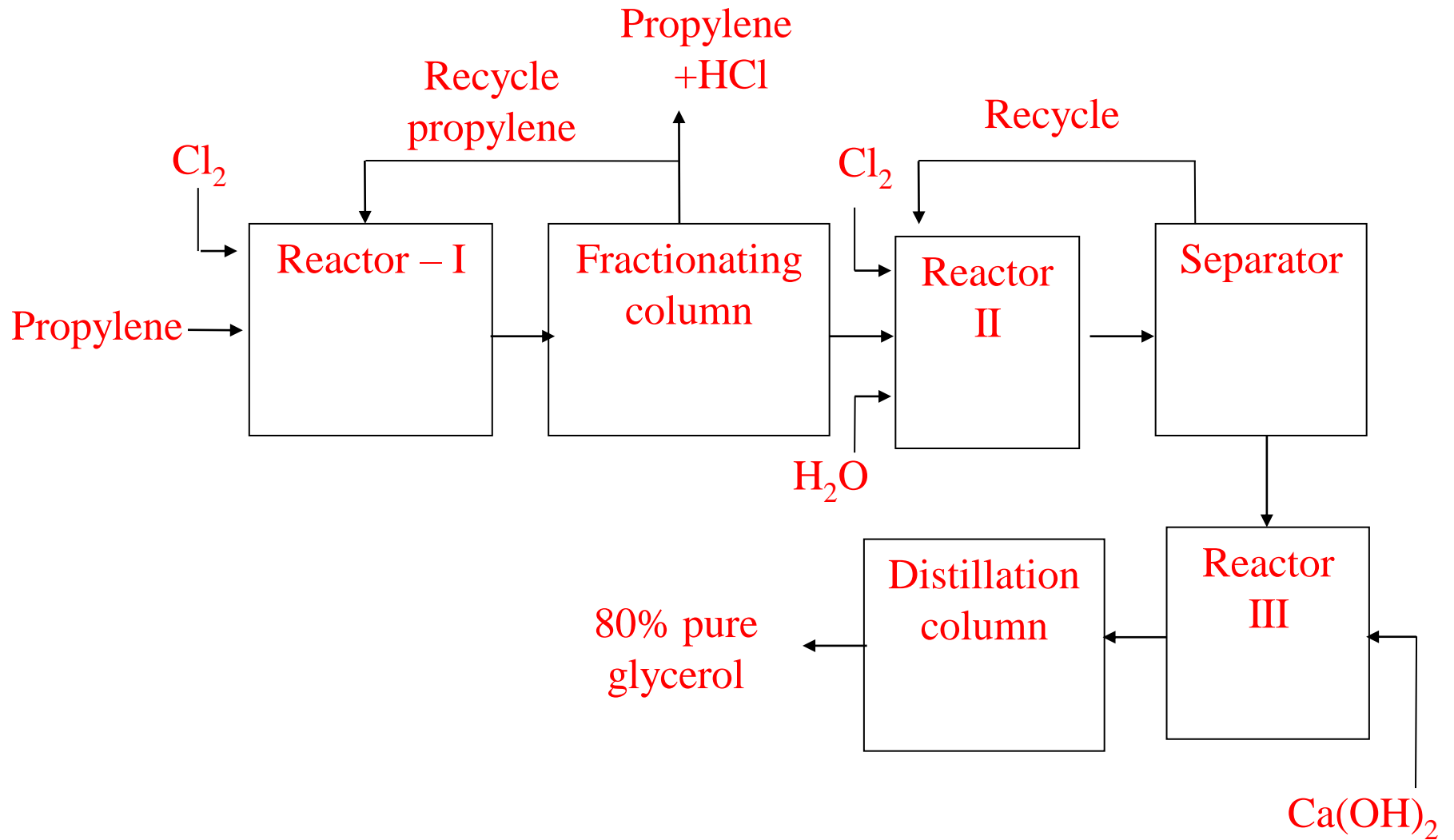
**Principle:** - The mixture of petroleum cracked gases rich in propylene is firstly treated with chlorine gas at 400 – 500°C for 2 – 3 seconds to give allyl chloride. Allyl chloride is then treated with chlorine and water (i.e. hypochlorous acid) at 35°C. As a result dichlorohydrin is formed, which is further reacted with milk of lime at 60°C to produce epichlorohydrin, which is hydrolysed with 10% caustic soda solution at about 150°C for half an hour to obtain glycerine.

**Raw Materials:** - Propylene, Cl<sub>2</sub>, Ca(OH)<sub>2</sub>, NaOH, Water.

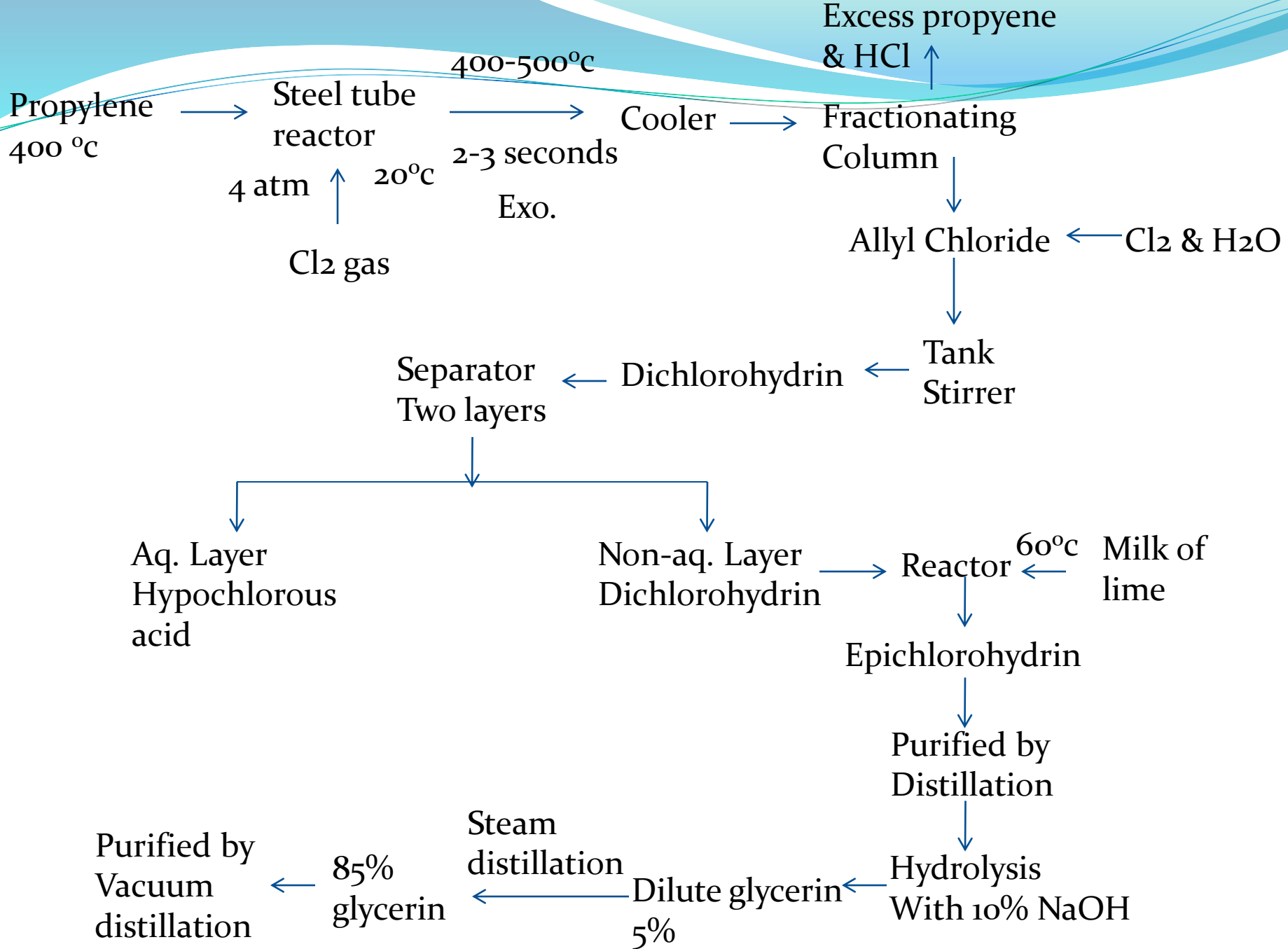
**Reaction: -**



Flowsheet: -







**Process:** - The process involves following steps;

- a) Reactor – I
- b) Fractionating column
- c) Reactor – II
- d) Separator
- e) Reactor – III and
- f) Distillation column

**a) Reactor – I:**

Dry propylene gas obtained from cracked petroleum is preheated to about 400°C and mixed with chlorine gas under a pressure of 4 atm. The mixed gases are heated in a steel tube reactor at 400-500°C for 2-3 seconds. The reaction is exothermic and so the temperature is maintained constant by means of insulation provided with the reactor.

**b) Fractionating column:**

The product is then cooled by passing through a cooler and then introduced into a fractionating column, where allyl chloride is collected at the bottom and excess of propylene, along with HCl leaves the column at the top and propylene are separated from HCl is again recycled.

**c) Reactor – II:** Allyl chloride is then introduced in a tank fitted with a stirrer and treated with chlorine and water (i.e. hypochlorous acid) at about 35°C, to give dichlorohydrin.

**d) Separator:** - The product is sent to a separator, where it is separated into two layers, the aqueous layer of hypochlorous acid and non aqueous layer of dichlorohydrin as the main product. The aqueous layer of hypochlorous acid is separated from the non aqueous layer and recycled. The non aqueous layer is sent to a reactor – III.

**e) Reactor – III:** - In this reactor, non aqueous layer is allowed to react with milk of lime  $[\text{Ca}(\text{OH})_2]$  at about 60°C and the product is steam distilled. As a result, epichlorohydrin distilled over with steam.

**f) Distillation column:** - Epichlorohydrin is purified by distillation and then hydrolysed with 10% caustic soda solution at about 150°C for half an hour. Dilute glycerin (5%) and NaCl are thus formed. The product is first steam distilled under Vacuum to get 85% refined glycerol, which is further purified by vacuum Distillation to give pure glycerol (80% yield).

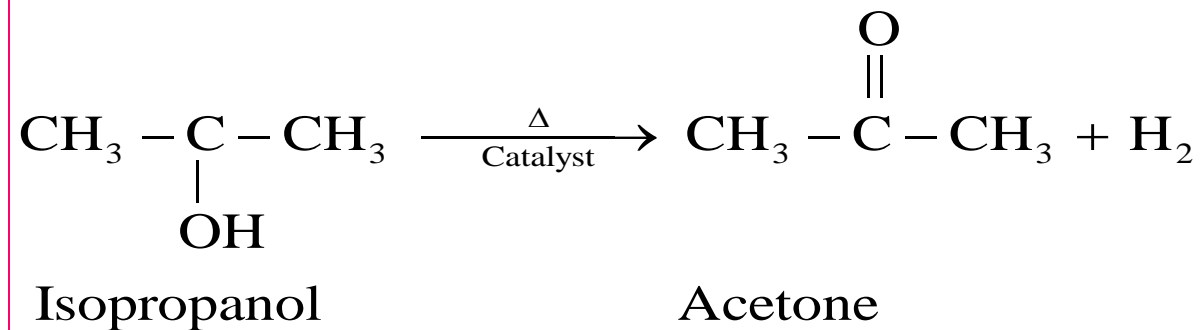
**Uses of glycerine: -**

1. It is used in synthesis of drugs, food and beverage, tooth pastes.
2. It is also used in the manufacture of alkyd resins, printing inks, explosives etc.
3. It is used in synthesis of toilet soaps and transparent soaps.

## Acetone or Propanone by Catalytic Dehydrogenation of Isopropanol: -

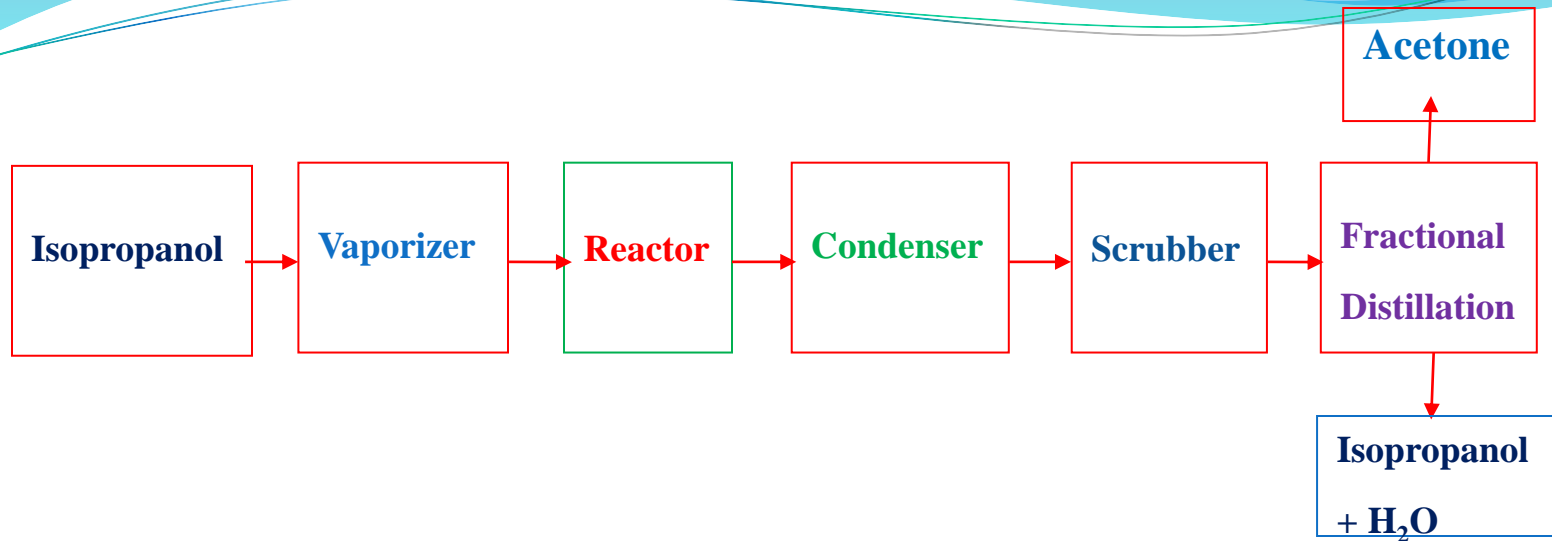
**Principle:** - The vapour of isopropanol passed into a reactor containing finely divided copper catalyst maintained at about 400°C, that results in the formation of acetone. Using catalytic dehydrogenation of isopropanol, about 90% pure acetone is obtained.

**Reaction:** -



**Raw Materials:** - Isopropanol, Copper catalyst, Water, etc.

## Flowsheet: -



**Fig. : Flow Chart of Preparation of Acetone by Catalytic Dehydrogenation of Isopropanol**

**Process:** - The industrial manufacturing process of acetone involves following steps.

- Vaporizer
- Reactor
- Condenser
- Scrubber
- Fractional distillation

**a) Vaporizer:** - The isopropanol is preheated in a heat exchanger and then passed under a pressure of 3 – 4 atmospheres into a reactor.

**b) Reactor:** - The reactor contains finely divided copper catalyst maintained at about 400°C. The vapours of isopropanol when comes in contact with catalyst, the dehydrogenation of isopropanol takes place, which results in the formation of acetone.

**c) Condenser:** - The hot vapours of acetone along with by-product  $H_2$  and unreacted isopropanol are passed through a water cooled condensing unit and then to a water scrubber.

**d) Scrubber:** - In water scrubber the gases enter from the bottom and water is sprayed from the top. Uncondensed acetone as well as unreacted isopropanol are absorbed by water and hydrogen gas is collected as a byproduct at the top.

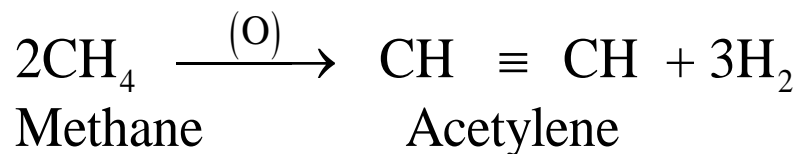
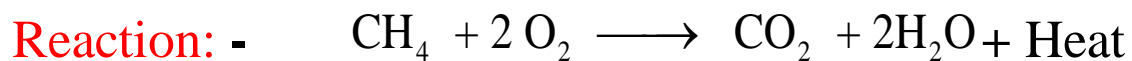
**e) Fractional distillation:** - Aqueous acetone and isopropanol are fractionally distilled in a fractionating column. Acetone is obtained as overhead has almost pure acetone [i.e. 90% yield] and isopropanol water mixture is removed from the bottom.

## Uses of acetone: -

- It is one of the most important solvents in organic synthesis.
- It is used as a solvent for acetylene, nitro-cellulose and cellulose acetate.
- It is also used in the preparation of sulphonal (a soporific), irone (an artificial scent), cordite (smokeless powder), plaxiglass (a plastic), chloroform, iodoform etc.

## Unsaturated Hydrocarbons-Preparation of Acetylene from Natural Gas: -

**Principle:** - Methane obtained from natural gas is partially oxidized by oxygen into  $\text{CO}_2$  and water. The excess methane is then converted into acetylene in presence of heat evolved in the former reaction.





# **INORGANIC CHEMISTRY**

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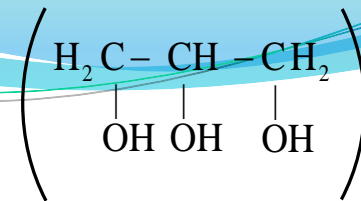
### **INDUSTRIAL ORGANIC SYNTHESIS**

#### **ONLINE LECTURE- NO. 4**

**DATE:- 2, NOVEMBER 2020**

**TIME: (10.00A.M.)**

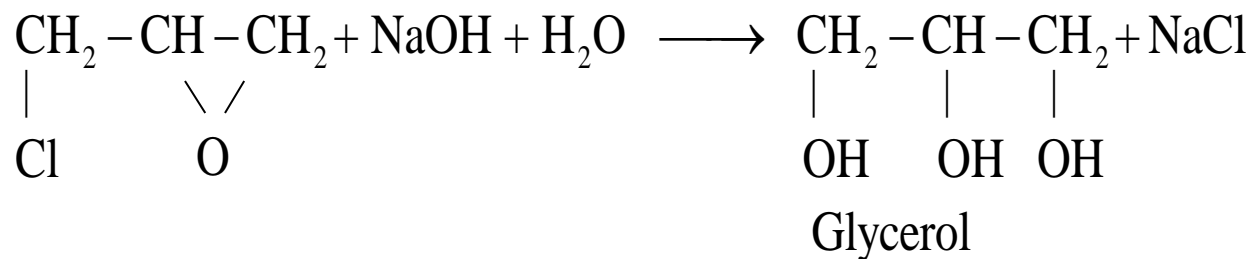
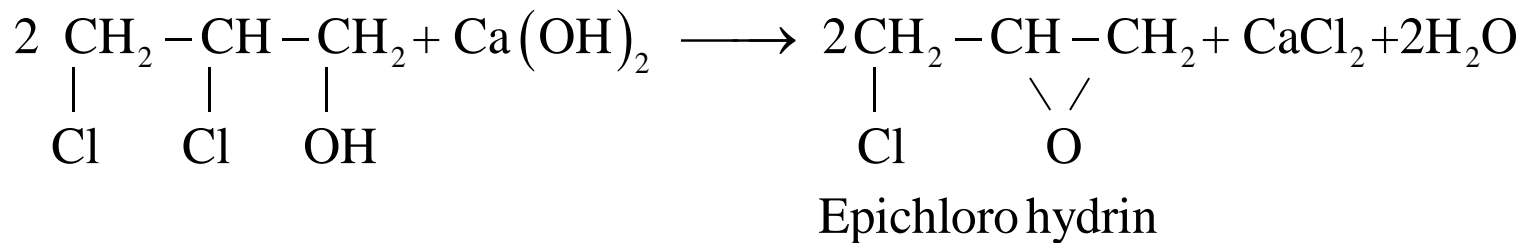
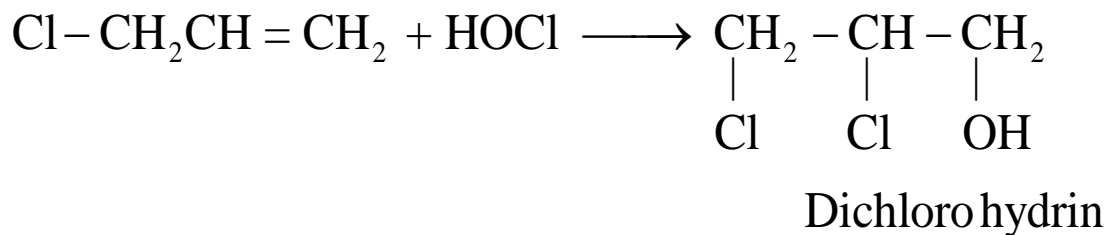
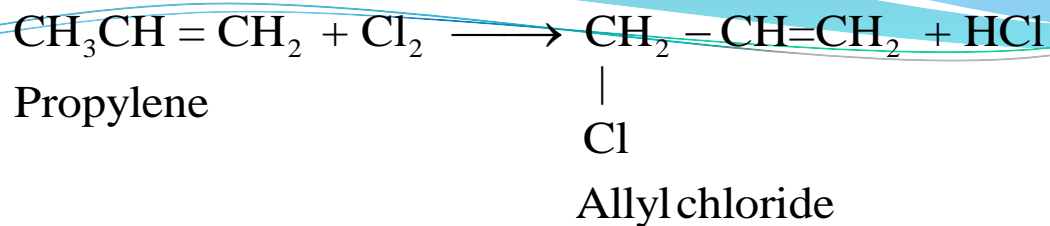
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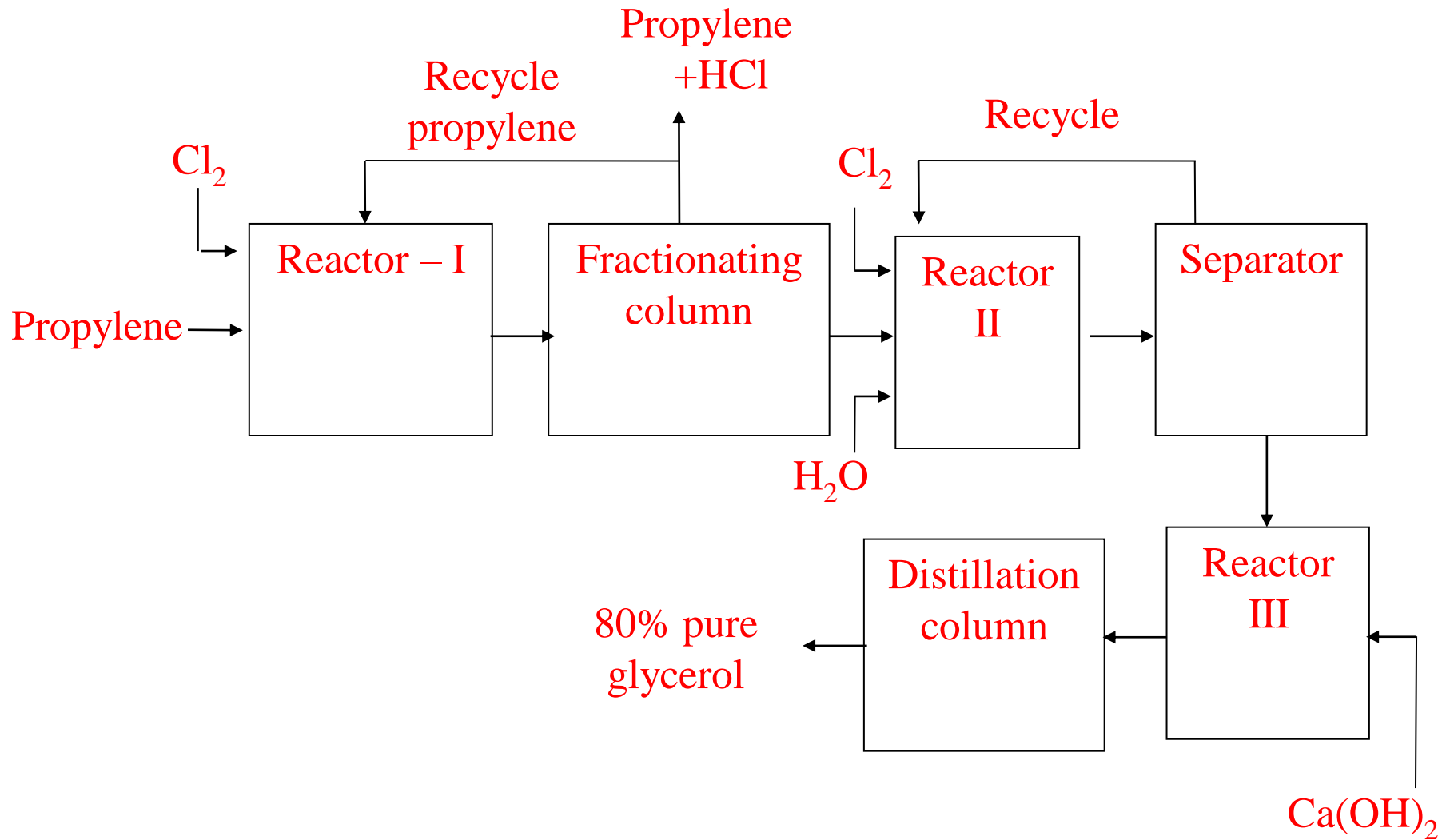
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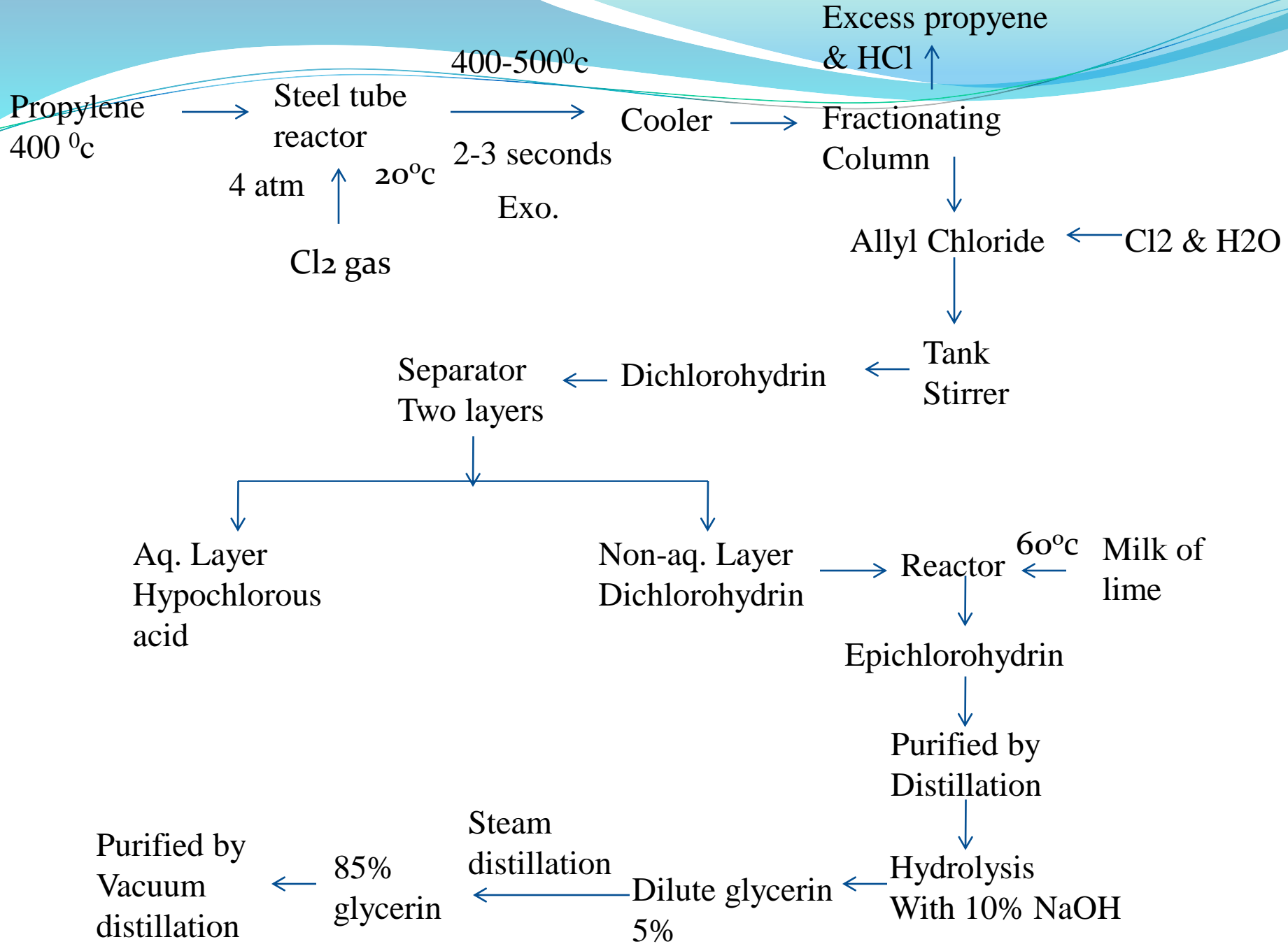
**Raw Materials:** - Propylene, Cl<sub>2</sub>, Ca(OH)<sub>2</sub>, NaOH, Water.

**Reaction: -**



Flowsheet: -





**Process:** - The process involves following steps;

- a) Reactor – I      b) Fractionating column      c) Reactor – II
- d) Separator      e) Reactor – III and      f) Distillation column

**a) Reactor – I:**

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**d) Separator:** - The product is sent to a separator, where it is separated into two layers, the aqueous layer of hypochlorous acid and non aqueous layer of dichlorohydrin as the main product. The aqueous layer of hypochlorous acid is separated from the non aqueous layer and recycled. The non aqueous layer is sent to a reactor – III. ( $\text{H}_2\text{O} + \text{Cl}_2 \rightarrow \text{HOCl} + \text{HCl}$ )

**e) Reactor – III:** - In this reactor, non aqueous layer is allowed to react with milk of lime [ $\text{Ca}(\text{OH})_2$ ] at about 60°C and the product is steam distilled. As a result, epichlorohydrin distilled over with steam.

**f) Distillation column:** - Epichlorohydrin is purified by distillation and then hydrolysed with 10% caustic soda solution at about 150°C for half an hour. Dilute glycerin (5%) and NaCl are thus formed. The product is first steam distilled under Vacuum to get 85% refined glycerol, which is further purified by vacuum Distillation to give pure glycerol (80% yield).

**Uses of glycerine: -**

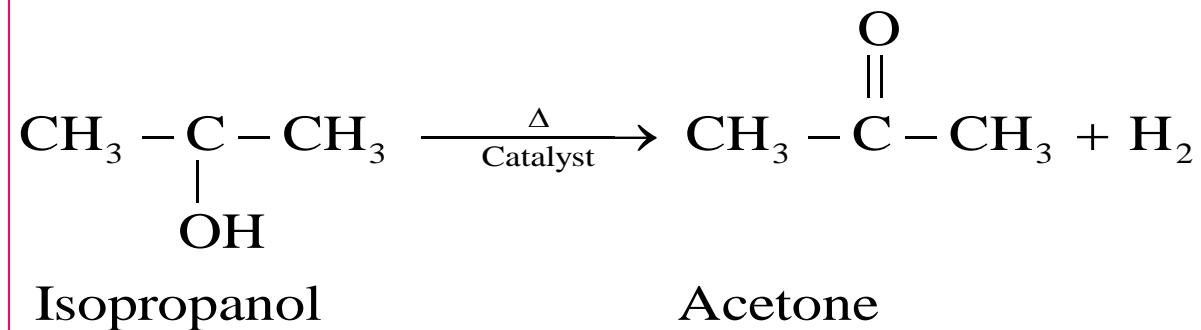
1. It is used in synthesis of drugs, food and beverage, tooth pastes.
2. It is also used in the manufacture of alkyd resins, printing inks, explosives etc.
3. It is used in synthesis of toilet soaps and transparent soaps.



## Acetone or Propanone by Catalytic Dehydrogenation of Isopropanol: -

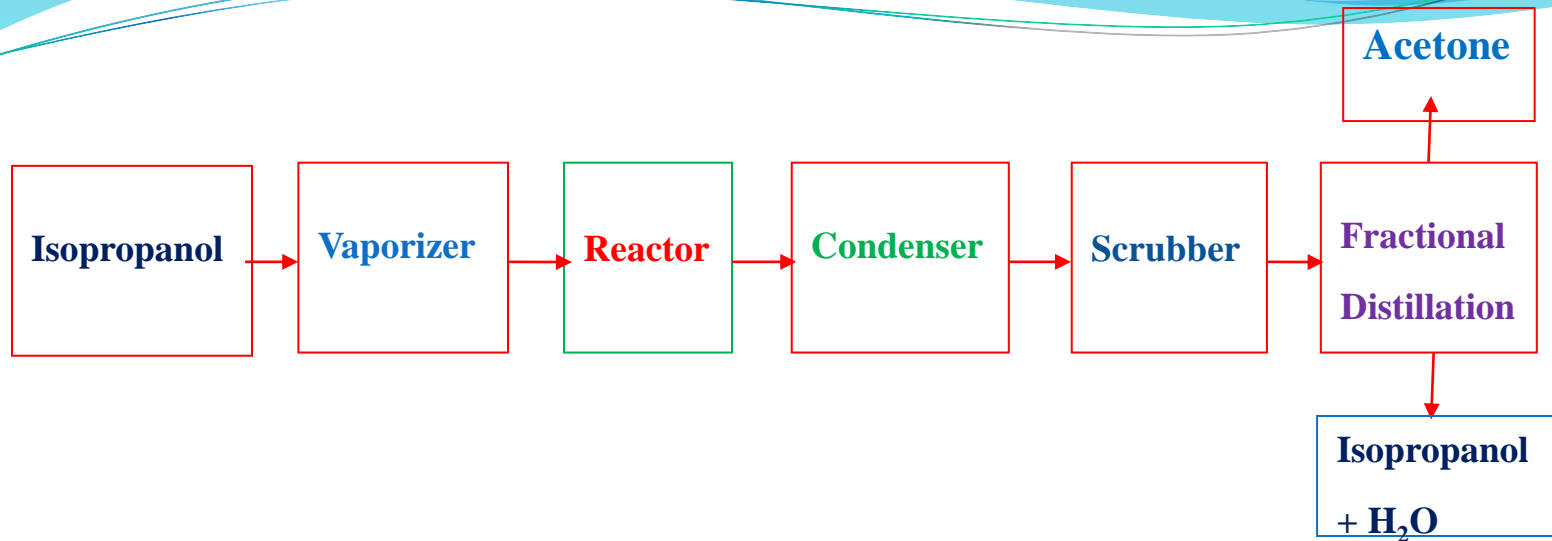
**Principle:** - The vapour of isopropanol passed into a reactor containing finely divided copper catalyst maintained at about 400°C, that results in the formation of acetone. Using catalytic dehydrogenation of isopropanol, about 90% pure acetone is obtained.

**Reaction:** -



**Raw Materials:** - Isopropanol, Copper catalyst, Water, etc.

## Flowsheet: -



**Fig. : Flow Chart of Preparation of Acetone by Catalytic Dehydrogenation of Isopropanol**

**Process:** - The industrial manufacturing process of acetone involves following steps.

- Vaporizer
- Reactor
- Condenser
- Scrubber
- Fractional distillation

**a) Vaporizer:** - The isopropanol is preheated in a heat exchanger and then passed under a pressure of 3 – 4 atmospheres into a reactor.

**b) Reactor:** - The reactor contains finely divided copper catalyst maintained at about 400°C. The vapours of isopropanol when comes in contact with catalyst, the dehydrogenation of isopropanol takes place, which results in the formation of acetone.

**c) Condenser:** - The hot vapours of acetone along with by-product H<sub>2</sub> and unreacted isopropanol are passed through a water cooled condensing unit and then to a water scrubber.

**d) Scrubber:** - In water scrubber the gases enter from the bottom and water is sprayed from the top. Uncondensed acetone as well as unreacted isopropanol are absorbed by water and hydrogen gas is collected as a byproduct at the top.

**e) Fractional distillation:** - Aqueous acetone and isopropanol are fractionally distilled in a fractionating column. Acetone is obtained as overhead has almost pure acetone [i.e. 90% yield] and isopropanol water mixture is removed from the bottom.

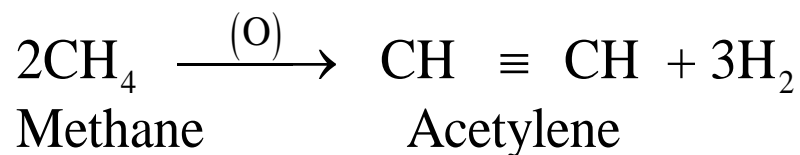
## Uses of acetone: -

- It is one of the most important solvents in organic synthesis.
- It is used as a solvent for acetylene, nitro-cellulose and cellulose acetate.
- It is also used in the preparation of sulphonal (a soporific), irone (an artificial scent), cordite (smokeless powder), plaxiglass (a plastic), chloroform, iodoform etc.

## Unsaturated Hydrocarbons-Preparation of Acetylene from Natural Gas: -

**Principle:** - Methane obtained from natural gas is partially oxidized by oxygen into CO<sub>2</sub> and water. The excess methane is then converted into acetylene in presence of heat evolved in the former reaction.

**Reaction:** -



**Raw Materials:** - Natural gas, Oxygen, water, dimethyl formamide, etc.

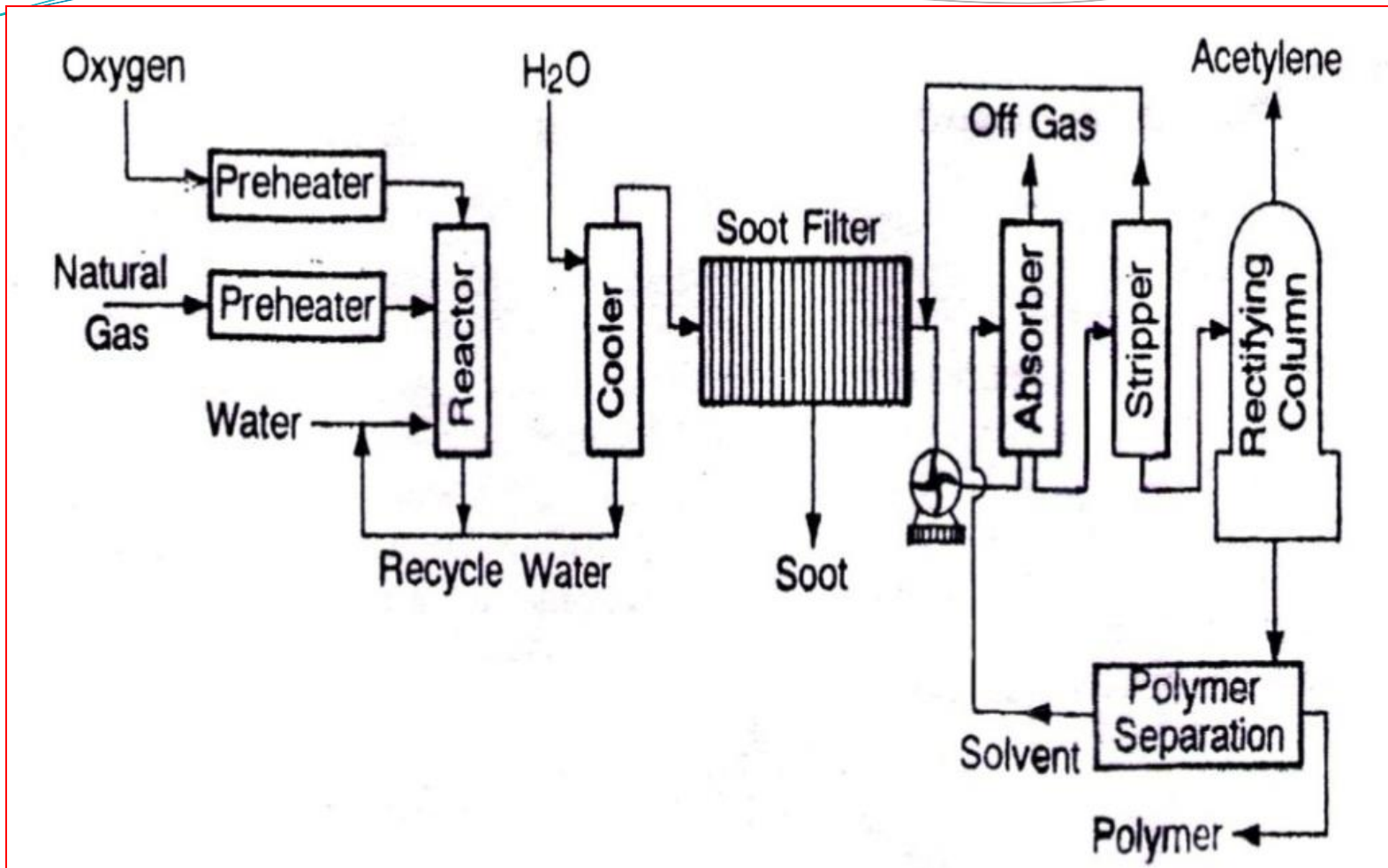


Fig.: Preparation of Acetylene from Natural Gas

## Process: -

- 1) Firstly natural gas and pure oxygen are separately preheated in pre-heaters at about  $550 - 650^{\circ}\text{C}$  and such hot gases then fed to a reactor.
- 2) The reactor consists of three zones namely, mixing, flame chamber and quenching. At first stage, the gases are rapidly mixed in the mixing zone. Then the mixture passes to the flame chamber having specially designed burners, where methane burns in oxygen and heat of combustion increases the temperature of the gases to about  $1500^{\circ}\text{C}$ , because it is an exothermic reaction. Within a contact period of 0.001 to 0.01 seconds, methane is cracked to acetylene. Since, cracking of methane to acetylene is an endothermic reaction, the reaction products are quenched quickly with water jets in the quenching zone to avoid further reaction.
- 3) The reaction products are further cooled in a water cooling chamber to about  $35-40^{\circ}\text{C}$  in order to prevent decomposition of acetylene.

- 4) The out coming cooled gases are then made free from carbon i.e. soot by passing through a soot filter. Thus, obtained gases contain 8–10% acetylene along with hydrogen, methane, carbon monoxide, carbon dioxide and some polymerized acetylene.
- 5) These gases are then passed through compressor where gases are compressed to 8-10 atmospheric pressure and then passed into an absorber containing dimethyl formamide as solvent. Acetylene, polymers of acetylene, some ethylene and CO<sub>2</sub> are absorbed by the solvent and the unabsorbed gases leave the absorber as overhead.
- 6) The solvent containing absorbed acetylene is passed through a stripping column, in order to remove less soluble components.
- 7) Further it is sent to a rectifying column in order to obtain the pure 99.5% acetylene as overhead.



## Uses of Acetylene: -

Acetylene is used for the preparation of following compounds.

- |                        |                            |
|------------------------|----------------------------|
| i. Acetaldehyde        | viii. Tetra chloro ethane  |
| ii. Acetic acid        | ix. Pentachloro ethane     |
| iii. Per acetic acid   | x. Ethylene chloride       |
| iv. Vinyl chloride     | xi. Trichloro ethylene     |
| v. Vinyl acetate       | xii. Tetra chloro ethylene |
| vi. Methyl vinyl ether | xiii. Butadiene etc.       |
| vii. Trichloro ethane  |                            |

# **INORGANIC CHEMISTRY**

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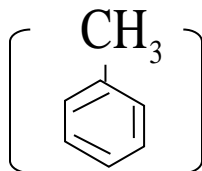
# **INDUSTRIAL ORGANIC SYNTHESIS**

## **ONLINE LECTURE- NO. 5**

**DATE:- 3, NOVEMBER 2020**

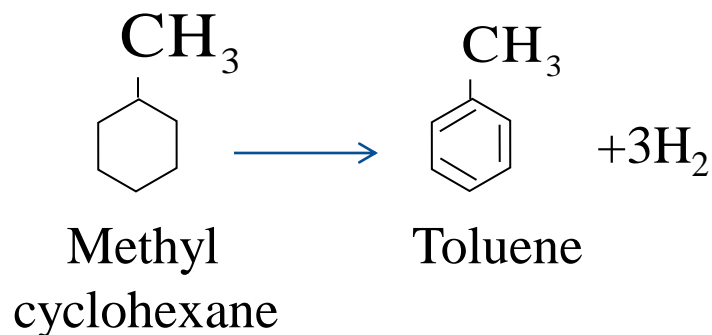
**TIME: (10.00A.M.)**

## Aromatic Hydrocarbons-Toluene or Methyl Benzene: -



**Principle:** - During the catalytic reforming of a selected fraction of petroleum (known as naphtha), the aromatic hydrocarbons such as toluenes and xylenes are obtained in large amounts. This fraction of petroleum mainly contains methyl cyclohexane, ethyl cyclopentane and diethyl pentane. From these, methyl cyclohexane forms toluene and  $H_2$  by dehydrogenation process.

**Reaction:** -



**Raw Materials:** - Naphtha, Molybdenum dioxide on alumina catalyst, Methyl ethyl ketone, Conc.  $H_2SO_4$ , NaOH, Water, etc.

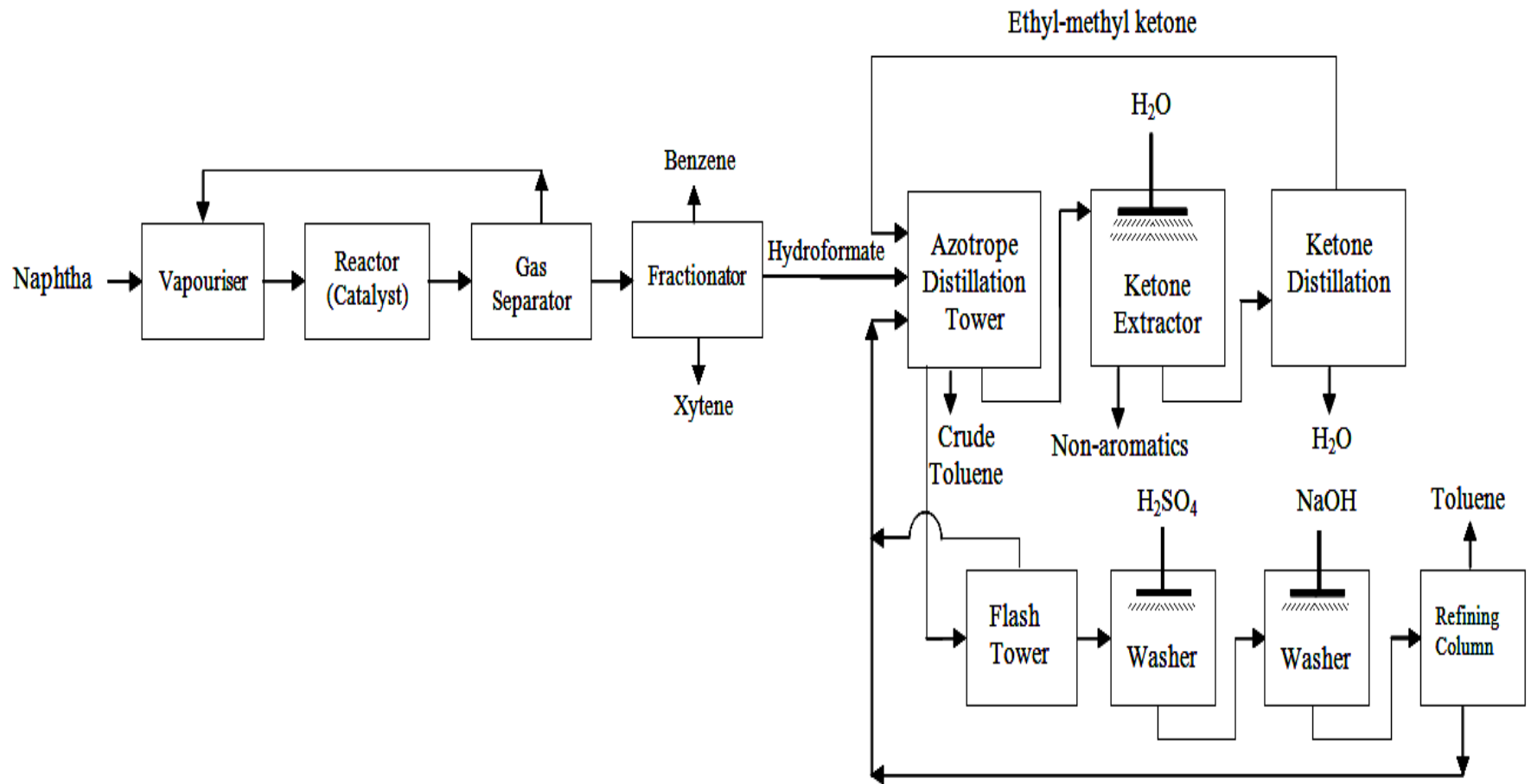


Fig. 3.6: Preparation of aromatic hydrocarbons from naphtha

**Process:** - The preparation of aromatic hydrocarbons (toluene, xylenes) from naphtha involves following steps.

- i) Vaporiser
- ii) Reactor
- iii) Gas separator
- iv) Fractionator
- v) Azeotropic distillation
- vi) Flash tower

**i) Vaporiser:**

The naphtha fraction is first preheated by passing through a heat exchanger and converted into vapour in furnace, where it is mixed with recycled gas containing excess of hydrogen and heated to 575°C.

**ii) Reactor:**

The hot gaseous mixture under a pressure of 10-20 atmospheres and a temperature upto 560-575°C is passed through a reactor containing a catalyst of 10% molybdenum dioxide on alumina. In reactor, the time of contact between gases and catalyst is about 15 sec. per pass and the conversion is about 80-90%.

**iii) Gas separator:** The gases from the reactor are allowed to pass through heat exchangers where liquid condensate and separated from gas. The gas containing excess of hydrogen is compressed to 5-15 atmospheres and recycled to the reactor.

**iv) Fractionator:**

The remaining gas and liquid are passed to the fractionating columns, where these are separated into the following fractions.

a) Aliphatic hydrocarbons (upto 4 carbon atoms) - These are used as fuel.

b) Gasoline fraction (containing  $C_5 - C_8$  hydrocarbons)

c) Hydroformates (containing 21% toluene) –

The third fraction is subjected to fractional distillation and different fractions are collected. From distillation, the first fraction is benzene fraction obtained as overhead, the second or middle fraction is toluene [contains about 65% toluene] and third or last fraction is xylene, which is removed from bottom of the fractionating column.

### v) Azeotropic distillation:

The middle fraction containing about 65% toluene is introduced into an azeotropic distillation tower containing a mixture of 90% ethyl methyl ketone and 10% water. The non aromatics such as paraffins and naphthenes get associated with the solvent, which carries them out of distillation tower. Toluene moves down the tower and collected from the bottom as crude toluene.

### vi) Flash tower:

Crude toluene is then passed through a flash tower where ketone is obtained as overhead, which can be recycled to the azeotropic distillation tower. The ketone free toluene from the bottom of the flash tower is washed with conc.  $\text{H}_2\text{SO}_4$  to remove adhering olefins, then with water and finally with caustic soda solution. The resulting toluene is then distilled to get about 90% pure toluene.

Uses of Toluene: - Toluene is used in the preparation of

- i. Benzyl chloride
- ii. Benzoyl chloride
- iii. Benzyl alcohol
- iv. Benzoic acid
- v. Benzaldehyde
- vi. Trinitro toluene [T.N.T.]
- vii. Phthalic anhydride etc.





**Thank You.**

*Stay Home, Stay Safe.*