

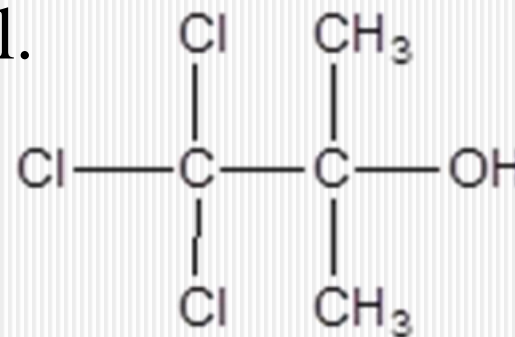
Preparation of chlorobutanol

2nd course

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Preparation of chlorobutanol

The IUPAC name is 1,1,1-trichloro-2-methylpropan-2-ol and the other names are chlorbutol or 1,1,1-trichloro-2-methyl-2-propanol.



Molecular formula $C_4H_7Cl_3O$

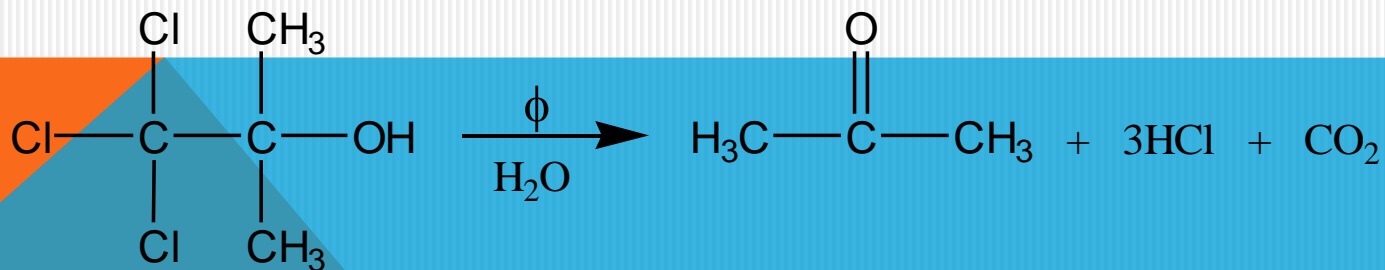
It can be used for many therapeutic indications.

1. Bacteriostatic use as preservatives in many injectable, ophthalmic and intranasal preparations.
2. Sedative, hypnotic and in motion sickness.
3. Local anesthetic in many painful IM injections and dental preparations.

Physical properties:

It is white crystalline powder found in two forms: anhydrous and hydrated, also it has characteristic camphor-like odor and taste. The melting point is 95-99 °C and boiling point is 167 °C

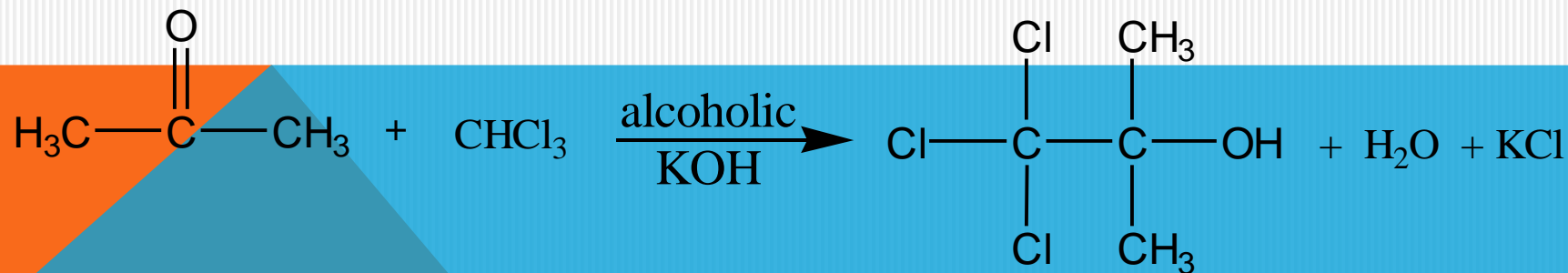
It is freely soluble in alcohol (1:1) slightly soluble in cold water (1:125) and more soluble in boiling water but such high temperature may lead to hydrolysis of chlorobutanol.



So it must be recrystallized from water/alcohol mixture. Water is not good solvent for recrystallization and often hydroalcoholic mixtures are used for this purpose.

Preparation of chlorobutanol:

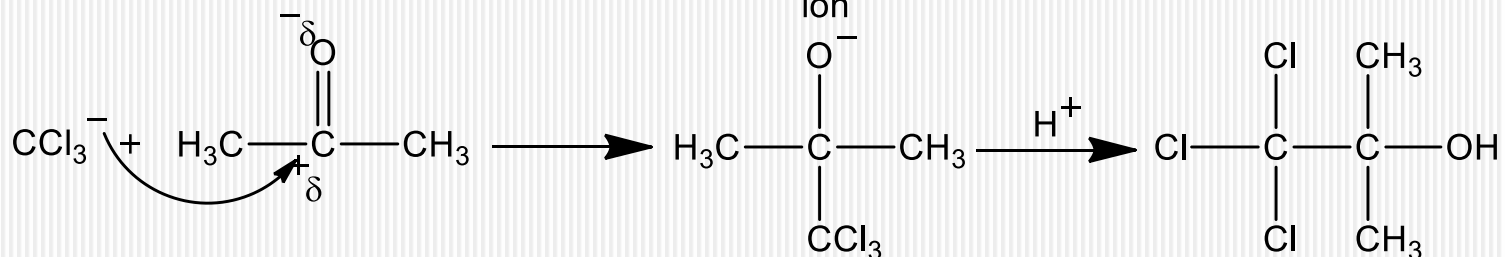
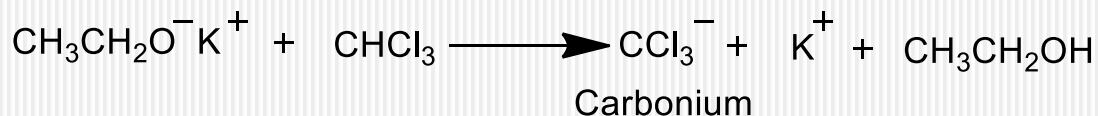
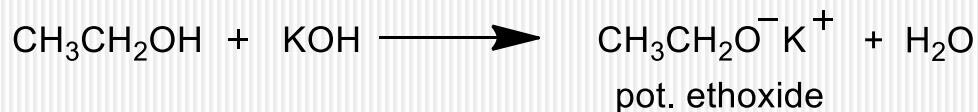
It is prepared from acetone and chloroform using KOH to give chlorobutanol.



Chlorbutanol is formed by the simple nucleophilic addition of chloroform and acetone, this reaction is base driven by potassium or sodium hydroxide.

Alcoholic KOH is used in order to accelerate the reaction towards formation of chlorobutanol.

Mechanism of reaction:



The source of Cl^- came from dissociation of another chloroform molecule.

Firstly, we obtain white ppt of KCl , we must get rid of it by filtration then we must evaporate alcohol to obtain chlorobutanol.

Procedure:

1. In a dry conical flask put 5 mL of acetone with 2 mL of chloroform.
2. Cool the mixture.
3. Alcoholic solution is prepared from dissolving 0.35 g of KOH in the minimum amount of ethanol (rectified spirit about 5mL).
4. Add alcoholic solution in step 3 to the mixture of step 2.
5. Filter the precipitated KCl and wash it twice with small portions of acetone.
6. Evaporate in water bath.
7. Recryastallize from the mixture of water and ethanol.