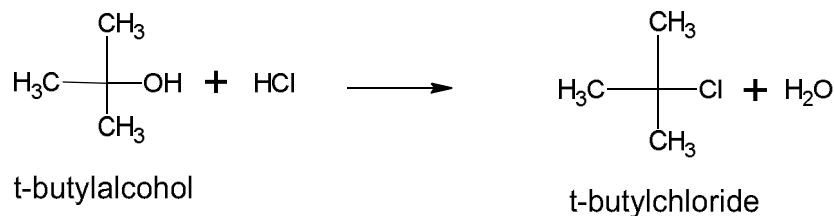
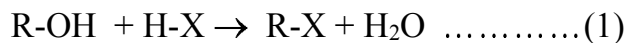


Preparation of t-butylchloride**Purpose of experimental**

To convert t-butylalcohol to butylchloride using an S_N1 reaction with HCl.

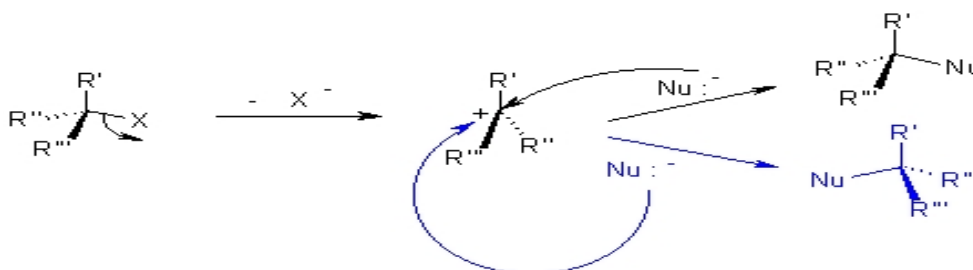
Theory part of experimental

The most common of the variety of methods available for preparing alkyl halides is the replacement of the hydroxyl group of an alcohol. This type of reaction is representative of an important class of reactions in organic chemistry called *nucleophilic aliphatic substitution reactions*. In this experiment you will convert an alcohol to an alkyl halide by reaction with a hydrohalic acid. The overall reaction is shown in equation (1); however the mechanism of the reaction depends on the structure of the alkyl group bearing the functional group being replaced.

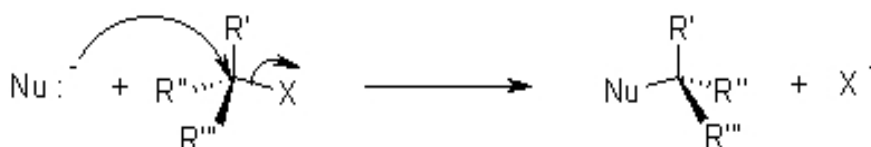


The reaction may occur by one of two mechanisms designated S_N1 or S_N2. Which mechanism operates depends on the structure of the R group and the reaction conditions.

When (R) is a tertiary alkyl group and is called S_N1 (substitution, nucleophilic, unimolecular).

S_N1 Mechanism:

If (R) is a primary alkyl group, then substitution occurs generally without formation of the carbocation by an S_N2 mechanism (why no carbocation? Because 1° carbocations are generally unstable). When (R) is a secondary group, an S_N1 or an S_N2 mechanism may be involved, depending on the reaction conditions.

S_N2 Mechanism:**Methods for preparation of alkyl halides**

1. From alcohols : $\text{R-OH} + \text{HX} \longrightarrow \text{R-X}$
2. Halogenation of hydrocarbons : $\text{R-H} + \text{X}_2 \longrightarrow \text{R-X}$
3. Addition of HX to alkenes : $\text{CH}_2=\text{CH}_2 + \text{HX} \longrightarrow \text{CH}_3\text{-CH}_2\text{-X}$

Physical properties of alkyl halides

1. Haloalkanes have higher boiling points than alkanes with the same number of carbons.
2. The boiling point increases with increasing atomic weight of the halogen and carbon number.
3. Haloalkanes have low polarity, and are soluble in the typical organic solvents of low polarity, like benzene, ether, chloroform. They are insoluble in water, probably because of their inability to form hydrogen bonds.

Chemical and Apparatus

t-butyl alcohol, conc.HCl, 5% Na₂CO₃ , beaker, separatory funnel, cylinder

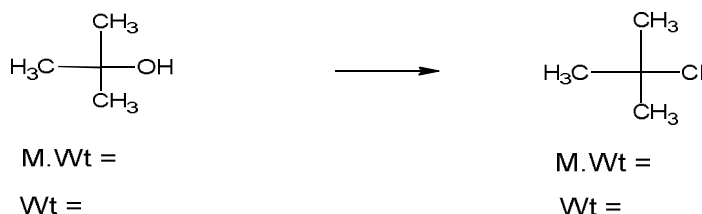
Procedure of Experimental

1. Place (6.0 mL) of *t*-butyl alcohol in separatory funnel
2. Cautiously add (12 mL) of conc.HCl and shaking the mixture and allow the mixture to separate completely into two layers, cancel the lower aqueous layer.

Note : to verify that *t*-butyl chloride should be the upper layer, add a few drops of water and observe. The water should not dissolve in the upper layer.

3. Add (10 mL) of 5% Na₂CO₃ to the *t*-butyl chloride layer (in separatory funnel) and shaking the mixture for separate completely to two layers, cancel the lower aqueous layer and measure the volume the upper layer (*t*-butyl chloride) by cylinder.
4. Add a minute amount of anh.Na₂SO₄ to upper layer (to drying) and swirl the solution. Filter the solution and distillation the solution to get pure *t*-butyl chloride.

5. **Test for *t*-butyl chloride with AgNO₃:** Place a few drops of the *t*-butyl chloride in test tube and Add (2 drops) of 0.1M AgNO₃ and mix. If no reaction is observed in 5 minutes at room temperature, warm the mixture and observe any change. The appearance of a white precipitate indicates that a reaction has taken place between the alkyl halide and silver nitrate.

Calculation**Wt. = density x volume**

$$\text{Theoretical weight of } t\text{-butyl chloride} = \frac{\text{Wt. of R - OH} \times \text{M.Wt. of R - Cl}}{\text{M.Wt of R - OH}}$$

$$\% \text{ of } t\text{-butyl chloride} = \frac{\text{Experimental weight of } t\text{-butyl chloride}}{\text{Theoretical weight of } t\text{-butyl chloride}} \times 100$$

Questions for discussion

1. Why is the *t*-butyl chloride phase the upper layer?
2. What was the purpose of addition Na₂CO₃ solution? Give equations.
3. Why is it undesirable to add aqueous NaOH (instead of Na₂CO₃ solution) to the *t*-butyl chloride solution (in the step 3) ?
4. Write the mechanism of reaction *t*-butyl alcohol with HCl for prepare *t*-butyl chloride.