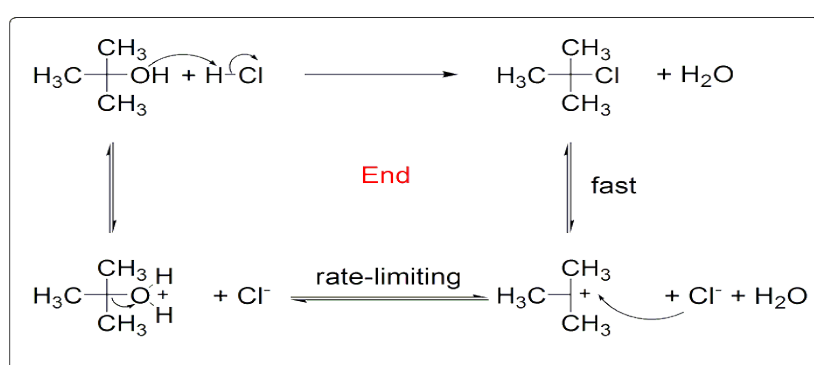


Exp.07: preparation of t-butyl chloride

Objectives:

-Prepare *tert*-butyl chloride (2-chloro-2-methylpropane) from *tert*-butyl alcohol (*tert*-butanol) using an acid catalyzed dehydration reaction. (Note: the correct IUPAC name for this compound is 2-methyl-2-propanol).

We will also learn how to use a separatory funnel, the use and purpose of a drying agent and the technique of distillation. The reaction is shown in Figure 3.1



The first step of the overall reaction is an acid-base reaction between the *t*-butanol and the hydrochloric acid. The *t*-butanol is a weak base and the hydrochloric acid is a strong acid. The alcoholic oxygen becomes fully protonated and so the equilibrium lies far to the right. In the second step we have the slow loss of water to form a carbocation intermediate. This species is very reactive and is immediately attacked by the chloride ion liberated in the first step to form the final product. This is an example of an S_N1 reaction (Substitution Nucleophilic Unimolecular).

Experimental Procedure

- In your separatory funnel (250 mL), place 120 mL of concentrated hydrochloric acid that has first been cooled to 5°C in an ice bath. Add 28 mL (22 g) of *t*-butanol and swirl gently with the stopper off the separatory funnel, release the pressure after every swirl for 20 minutes.
- Once the layers have been thoroughly mixed, allow the separatory funnel to stand with the stopper off. You will gradually see two layers forming. (What are they?)

- Drain off the bottom layer. This is the aqueous layer. It is waste, but be sure to save it until you have finished with the experiment.
- Add 100 mL of cool NaOH 5% to the separatory funnel and shake again several times to mix the two layers. Again separate the aqueous layer and set it aside. (You can save this with the first aqueous layer.)
- Add 10 mL sodium bicarbonate solution to the separatory funnel. Be careful here. The sodium bicarbonate is a weak base. The purpose for adding this is to neutralize any hydrochloric acid that may be remaining. When it reacts with the hydrochloric acid, carbon dioxide gas is given off. Pressure can build up very quickly in a closed separatory funnel and your solution will shoot out of the funnel

Laboratory Report

Name: -----

Date: -----

Experiment Subject: -----

- Reaction:

Calculations:

Compound	Mol. Formula	Mol. Weight	Moles	Weight, mg	Density	Volume	Limiting reagent

Purification:

Recrystallization solvent: -----

Purity check by melting range: -----

TLC: -----

Physical Data (Product):

State: -----

Melting Point: -----

Color: -----

Solubility: -----

Yield:

Characterization:
