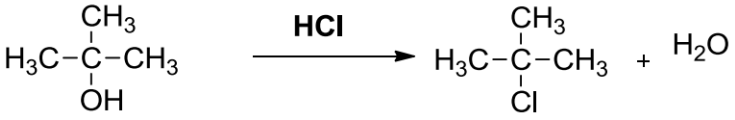
# *Lab N °08: Transformation of an alcohol into a halogenated derivative (synthesis of 2-chloro-2-methylpropane from 2-methylpropan-2-ol)*

## *Introduction*

Nucleophilic substitution results from the nucleophilic attack (NA) by an electron-rich species and the leaving group departure (LG) of an element carrying a lone pair of electrons.

Haloalkanes (R-X) are highly utilized reagents in organic chemistry because X (the halogens) can be replaced by numerous functional groups during nucleophilic substitution reactions, thus providing access to a wide array of new molecules.

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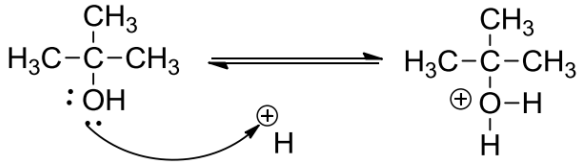
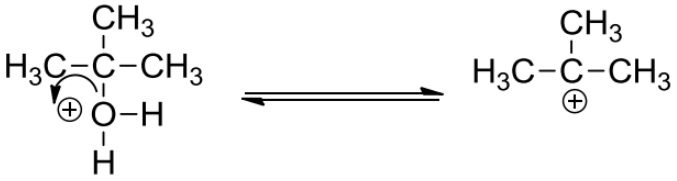
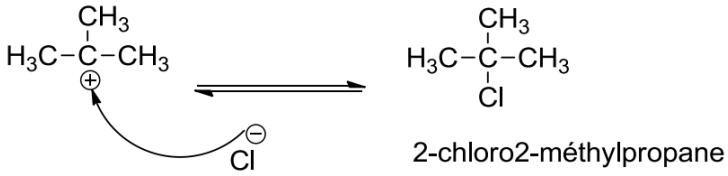
|---|---|
| **1st step:** Protonation of the alcohol |  |
| **2nd step:** Formation of the relatively stable tertiary carbocation |  |
| **3rd step:** Nucleophilic attack of the carbocation by the chloride ion |   |

## PROCEDURE

### *Halogenation*

1. In a 250 mL flask equipped with a thermometer, a bromine ampoule, a reflux condenser, and good agitation; introduce 12.5 mL of commercial 2-methylpropan-2-ol (if necessary, melt the alcohol using a water bath).
2. Through the bromine ampoule, add, with agitation, 30 mL of concentrated hydrochloric acid.
3. Let the reaction mixture stir for 20 minutes.

***Extraction and Washing***

1. Pour the solution into a separating funnel. Let the mixture settle until phase separation occurs, then remove the aqueous phase.
2. Quickly wash the organic phase with 10 mL of cold water, followed by 5 mL of 50 g/L sodium bicarbonate ($NaHCO_3$) solution (caution! gas evolution), and then 10 mL of cold water.
3. Measure the pH of the washing waters.
4. Collect the organic phase in a dry Erlenmeyer flask and dry it with 1 g of anhydrous calcium chloride using a spatula.

***Purification***

1. Prepare a distillation setup.
2. Filter into a dry 100 mL distillation flask, add pumice stone, then distill the product with gentle heating.
3. Collect in a dry Erlenmeyer flask, placed in an ice bath, the fraction distilling between 49°C and 51°C.
4. Determine the refractive index.

***Questions:***

1. Sketch and label the reflux setup used.
2. Calculate the yield of the synthesis.
3. Show that hydrochloric acid was introduced in excess.
4. How to distinguish the organic phase from the aqueous phase in the separating funnel?
5. What is the purpose of the sodium bicarbonate solution?
6. Explain the gas evolution observed in the separating funnel. Write the equation of the reaction.
7. Why should the pH of the washing water be measured?
8. Indicate the role of anhydrous calcium chloride.
9. Why is the purified tert-butyl chloride collected in an ice bath?
10. What type of reaction is it?