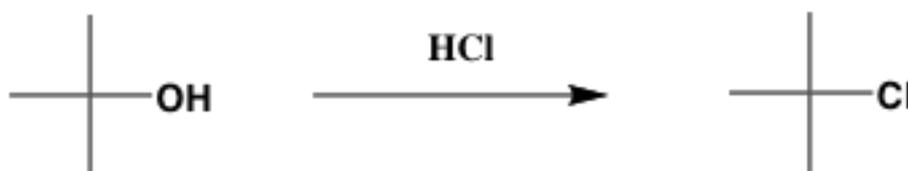


## Exp. 2 2-Chloro-2-methylpropane (tert. Butylchloride)

### *Nucleophilic substitution ( $S_N1$ ) of tert. butanol with HCl (Lucas-reaction)*

Halogenated alkanes are very important intermediates in synthetic chemistry but also have a lot of applications itself. Trichloromethane known as chloroform was discovered at the beginning of the nineteenth century and used as the first inhalation narcotic during surgeries (replaced today by less toxic narcotics). Ethylchloride is in use as a local anesthetic. We synthesize tert. butylchloride as an example of these important compound group because the reaction is quite fast and the product can be easily purified by a simple distillation. It can be used for example to introduce the tert. butyl group into aromatic rings (Friedel-Crafts-alkylation) to block a certain position to protect it during further transformations and could be removed later by a retro reaction.

*Reaction in a separatory funnel, extractive washings and phase separations, filtration and drying of a liquid followed by a final purification through distillation are practiced in this experiment. The boiling point and refractive index are determined.*



### *Preparation*

Read the supplement of this prescription (Gilbert & Martin, p.435ff. and Solomons /Fryhle, chapter 6)

### *Techniques*

Extraction, phase separation, liquid-phase drying, filtration, distillation

### *Equipment*

Separatory funnel, Erlenmeyer (conical)-flasks and beakers, magnetic stirrer with oil bath and thermometer, simple distillation set-up.

### *Chemicals*

tert.-Butanol, concentrated hydrochloric acid, saturated sodium chloride and sodium carbonate solution, dried calcium chloride

### *Safety*

conc. hydrochloric acid is a very corrosive liquid that exhales HCl vapor. Work under the hood and avoid any spills and contact with the skin.

Spills on the skin must be rinsed away immediately with water.

Read the MSDSs for the chemicals used in this procedure.

### Procedure

- Fill 100ml of conc. hydrochloric acid (about 1.2mol) into a 250ml conical (Erlenmeyer) flask equipped with the long stir bar and add 40ml of tert.-butanol.
- Cover the flask with a watch glass and stir the reaction mixture vigorously for 15min.
- Transfer the liquid into the separatory funnel and allow the mixture to separate into two layers.
- Collect both phases and check which one is the organic.
- Place the organic phase back into the separatory funnel and shake it with 20ml of saturated sodium carbonate solution (caution: CO<sub>2</sub>-evolution!).
- Separate again and repeat the washing procedure with 10ml of saturated sodium chloride solution.
- Collect the organic layer in an Erlenmeyer flask and add a spatula full of calcium chloride powder, close the flask and swirl it from time to time for 5 minutes. If the liquid remains cloudy add some more calcium chloride.
- Then filter the crude product into a 100ml round-bottom flask and determine the weight.
- Set up the apparatus for simple distillation and distill the crude product with a heating bath temperature of about 80°C.

### Tasks

Determine the boiling point, the weight and the refractive index of the product and compare your results with the literature values and calculate the yield.

Density of tert. butanol,  $d = 0.775 \text{ g/ml}$

Boiling point of tert butylchloride bp. = 51°C

Refractive index  $n=1.384$

**Protocol**

**Experiment 2**

**Date:**

Aim:

Apparatus and Materials:

Theory and Mechanism:

Procedure and Observations:

## Results and Calculations: