

## Exp. 1: Separation of a two component mixture

*A mixture of two organic solids have to be separated using their different solubilities depending on the acidity or basicity of the single components.*

*The fundamental day by day operations for a synthetic organic chemist like: extraction - phase separation - solvent drying – filtration techniques – Rotavap use – recrystallization are practiced in this experiment. By melting point determination of the purified compounds their structures are assigned by comparison with a melting point list.*

### Procedure

- Place the solid mixture into a beaker equipped with a stirbar, add 80ml of dichloromethane and stir the suspension for a few minutes until you obtain a clear solution.
- Add 100ml of a 0.2M NaOH-solution into the separatory funnel. Shake the CH<sub>2</sub>Cl<sub>2</sub>-solution with the NaOH-solution for about two minutes. Separate the phases into two different Erlenmeyer flasks (which is the organic phase?).
- Dry the organic layer with a small amount of Na<sub>2</sub>SO<sub>4</sub> for five minutes. After filtration into a 250ml round-bottom flask the solvent is removed at the rotavap.
- Recrystallize the resulting solid from ethanol (compound1).
- Put the aqueous solution into an Erlenmeyer flask and carefully add hydrochloric acid (20%) under stirring until the pH-value turns to strongly acidic.
- Vacuum filtrate the precipitate and wash it with some cold water (compound 2).
- Keep compounds 1 and 2 overnight on air to let them dry.

### Task

Determine the weights and the melting points of both compounds and decide which of the compounds they are from a posted melting point list (whiteboard).

**Protocol**

**Experiment 1**

**Date:**

Aim:

Apparatus and Materials:

Theory and Mechanism:

Procedure and Observations:

Results and Calculations: