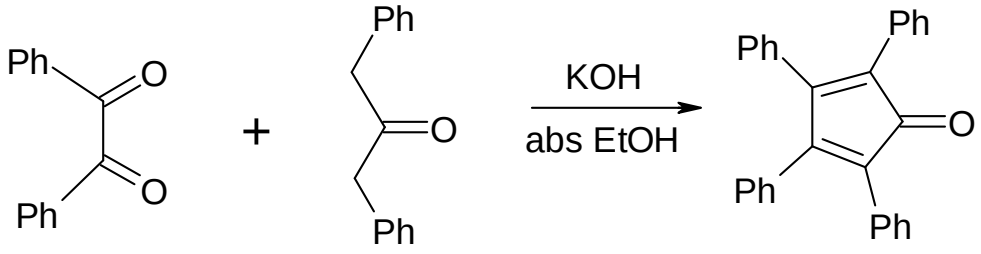
15. Baltic Chemistry Olympiad

Synthesis of tetraphenylcyclopentadienone



Reagents and apparatus

200 mg of benzil on plastic dish
200 mg of dibenzylketone in 10 ml round bottomed flask
Abs. ethanol (5 ml, in test tube **abs. EtOH**)
KOH solution in abs. ethanol (with syringe for measuring)
95% ethanol for separation of the product (in test tube **EtOH**)
Toluene/95% ethanol 1:1 mixture for recrystallization (in test tube **PhMe/EtOH**)
Destilled water (several plastic bottles in lab for all participants)
Crushed ice (two common containers in each lab)

Round bottomed flask (10 ml)
Air condenser
Pasteur pipette with rubber bulb
Spatula
Magnetic stirring bar
Magnetic stirrer with heating plate
Glass filter funnel
Equipment for filtration (one per two participants)
Folium to cover the flask during heating
Plastic dish for product

Equipment for TLC analysis

Elution chamber
TLC plate
Capillaries
Tweezers

Eppendorf tube for preparation of solution of the product
Eluent: hexane/EtOAc 3:1 (two Erlemeyer flasks in each lab)
UV-lamp (two lamps in each lab)
EtOAc for dissolving the sample

Procedure

Mount the 10 mL round bottomed flask (precharged with 200 mg of dibenzylketone) with the clamp to stand and add 200 mg of benzil, magnetic stirring bar and 1,5–2,0 ml abs. ethanol. Switch on the stirring and heating plate, increase the temperature gradually until the condensate appears at the lower end of condenser and add 0,3 mL of KOH solution in abs. ethanol with plastic syringe. During the heating you can answer the theoretical questions. Switch off the heating after 15 min and cool the reaction mixture to room temperature and finally in the ice bath for 5 min. Separate the product by vacuum filtration.

Rinse the flask with some 95% ethanol. Transfer the product back into the flask and recystallize the product from toluene-95% ethanol (1:1) mixture under stirring. Separate the crystals by vacuum filtration. Wash the crystals with minimal volume of 95% ethanol. Load the cystalline product onto the plastic dish and leave it to dry at room temperature. During this time you could do the TLC analysis.

TLC analysis

Dissolve some crystals of the product in small volume of EtOAc in Eppendorf tube and take the sample with capillary to the TLC plate. Eluate the plate with the given solvent mixture and dry thereafter in air. Inspect the plate under UV lamp.

If you have not obtained the product you can ask the sample material for TLC analysis from supervisiors without loosing any points you can get for the TLC.

Weight the product, **FILL IN THE ANSWER SHEET AND ANSWER THE QUESTIONS!**

Titration of HCl by generation of I2

Concentrations of strong acids are usually determined by titration with strong bases (for example NaOH). However redox titration can be used as well.

In this experiment concentration of hydrochloric acid is determined by reaction with potassium iodate, KIO_3. I_2 is generated by redox reaction between IO_3^- and I^-, the reaction is limited by the amount of H^+ ions present. Generated I_2 is titrated with $Na_2S_2O_3$.

10 ml of given HCl solution is pipetted into a 250 ml Erlenmeyer flask. 2 ml of 10% KI solution and 10 ml of KIO_3 solution are added to the solution by using graduated test tubes. Color of the solution turns brown. Titration flask is covered with watch-glass and stored in dark for 5-10 minutes.

Thereafter watch-glass is rinsed with distilled water and I_2 is titrated with $Na_2S_2O_3$. When the color of solution turns into light yellow then about 0.5 ml of starch solution is added. Endpoint is determined by the disappearance of blue color. The volume of used $Na_2S_2O_3$ solution is record. Experiment is repeated until three coinciding results are obtained. Calculate the concentration (mol/l) of HCl.