ORGANIC SYNTHESIS: ALDOL CONDENSATION REACTION

EXPERIMENTAL PROCEDURE



- Ethanol is highly flammable and is toxic.
- Aqueous sodium hydroxide is corrosive.
- Benzaldehyde is combustible liquid and an irritant, avoid skin contact.
- Acetone is flammable, is toxic and an irritant.

Preparation of a conjugated carbonyl using an Aldol Condensation Reaction

Using a 50 mL Erlenmeyer flask, dissolve 2.5 mL of benzaldehyde in 20 mL of 95% ethanol. Slowly add 3 mL of 2M NaOH (aq.) and swirl the flask to mix. Add 0.5 mL of acetone and let the solution sit for 5 min, then swirl the Erlenmeyer flask periodically for the next 25 minutes. The insoluble solid product should form during this time. While you are waiting for your product to form you should set up the vacuum filtration apparatus and wash / clean any glassware used in the previous steps.

Collect the solid product by vacuum filtration, washing the solid with about 10 mL of cold water to remove any remaining NaOH from the solid. Test with litmus paper that the washings are neutral after each wash. Repeat the cold water wash if required. Finally, wash with 10 mL of cold 95% ethanol and then allow the solid to dry in the Buchner funnel under vacuum for a few minutes. Weigh your crude product.

Recrystallise approximately 200 mg of crude solid from a minimum volume of 70% ethanol (heat the solvent using a hot plate). Collect the purified solid and allow it to dry in the Buchner funnel under vacuum for a few minutes.

Determine the melting point of the purified solid and submit a labelled sample of your products to your TA.

CLEAN UP

- All organic solutions should be placed in the organic waste container.
- All aqueous solutions should be placed in the aqueous waste container.

REFERENCES

- Mohrig, J.R.; Hammond, C.N.; Morrill, T.C.; Neckers, D.C. in "Experimental Organic Chemistry", W.H. Freeman: New York, 1997; pp 408-414.
- M. Jones and S. Fleming in "Organic Chemistry", Norton ,4th ed., 2010, Chapter 19, p 932-46, 965-85; 5th ed., 2014 Chapter 19, p 930-44, 961-82. Organic Chemistry On-Line Learning Center Chapter 18; <u>https://www.chem.ucalgary.ca/courses/351/Carey5th/Ch18/ch18-0.html</u>