

Equipment & Chemical Preparation

Experiment 6: Dibenzalacetone by the Aldol Condensation

In this experiment, students will work in pairs to prepare dibenzalacetone from benzaldehyde and acetone. Students will analyze their products by melting point and TLC analysis, and determine the percent yield of the reaction. The reaction is very straight-forward and is done in a 250 Erlenmeyer flask. Students add solid NaOH (5g) to the flask along with 50ml of DI water and 40ml of ethanol. The solution is stirred until the NaOH dissolves. Benzaldehyde is then added (5 ml) followed by addition of acetone in two, 1ml aliquots. After the reaction is complete, the students will filter the product from the reaction mixture by vacuum filtration. After the product is filtered, it will be recrystallized from ethanol. The recrystallization will require that the students dissolve the crude reaction product in 10 ml of ethanol. The solution is heated briefly in a water bath, then cooled in an ice bath. A second vacuum filtration will be done to isolate the purified product. Melting points will be determined. Benzoic acid will be used to calibrate the melting point thermometers. TLC analysis will be done on the product and compared with the benzaldehyde starting material. The product (~30mg) will be dissolved in dichloromethane ~3ml to spot the plate. Students will also spot the plate with a solution of benzaldehyde for comparison. Ethanol and hexane will be used as developing solvents.

Equipment
12 Hot Plate/Stirrers (one in each hood)
24 Ring Stands with a ring and clamp (2 in each hood)
12 Magnetic Stir Bars
12 250 ml Erlenmeyer Flasks
12 125ml filtration flasks
12 adapters (for Buchner funnels)
12 Buchner funnels
6 packs of filter paper (to fit Buchner funnels)
12 water baths (must be glass) (to fit around 125 ml Erlenmeyer flasks)
12 ice baths (plastic okay) (to fit around 125 ml Erlenmeyer flasks with ice and water)
6 jars TLC plates (~25 plates), 6 UV Lamps
6 iodine chambers, 12 pencils
6 packs of microcapillary tubes, 12 rulers

Chemicals
Benzaldehyde (500ml)
Acetone (150ml)
NaOH (500g)
Ethanol (4liters)
Hexane (2 liters)

***250 ml Erlenmeyer flasks and 125ml filtration flasks should be in student drawers, but we should have 12 or so additional flasks on reserve for use. Buchner funnels, vacuum flasks, filter paper should be in student/common drawers. Hot plate/stirrers and ring stands should be in the hoods, but rings are in the storage drawers and should be set out in the hood. Double check to make sure these items are available.*

Instructions

- Set up six reagent bins, one for each bench, containing each of the following items.

Equipment	Chemicals
1 plastic ice bath	75ml benzaldehyde
1 glass water bath	25ml acetone
1 magnetic stir bar	75g NaOH
	1g Benzoic acid (mp calibration)
	~5ml benzaldehyde solution in CH ₂ Cl ₂ **

***The benzaldehyde solution can be made up by dissolving ~5ml in 50ml CH₂Cl₂, then dispensed into vials*

- Set up six TLC bins, one for each bench, containing each of the following items.

TLC Bins	
1 jar TLC plates (~25plates)	1 UV lamp
1 iodine chamber	2 pencils
1 pack microcapillary pipets	2 rulers
~100 ml ethanol	~200 ml hexane

- Set out a pack of weigh paper next to each balance and a box of cover slips next to each melting point apparatus.
- Check bins throughout the week and refill as needed.