

# Exp

[Science](#), [Chemistry](#)



Paraphrasing of instructions for a chemistry experiment Part A: Reduction of benzophenone Dissolve 0.0074 mol of benzophenone in 9 mL of methanol in a 50-mL Erlenmeyer flask.

2. Dissolve 0.0026 mol of sodium borohydride in 4.5 mL of cold water in a second Erlenmeyer.

3. Add the sodium borohydride solution to the ketone solution dropwise. Use a Pasteur pipette, swirling

the flask between each addition to disperse any cloudiness before adding the next drop.

4. Once the dropwise addition is complete, place a stirbar in the reaction flask and stir until a heavy

slurry of precipitate forms. This should take between 30 minutes to 1 hour.

Part B: Collection of the Diphenylmethanol. USE GLOVES FOR THIS PART

1. In a 250-mL beaker, place 30 g of crushed ice and then carefully add 3 mL of conc. HCl. Remember

to add acid to water, not water to acid.

2. IN THE FUMEHOOD, slowly pour the reaction mixture into the acid. Do this carefully to avoid

foaming over.

3. Collect the solid product by suction filtration. Wash the crystals twice with 15 mL of ice-cold water.

4. Dry the crystals thoroughly before proceeding to the next step. If they are still wet, the

recrystallisation will be problematic because hexane is immiscible with water.

5. Once dry, weight the crude product and calculate a crude yield. Place 0.1 of the crude product aside for TLC analysis later.

#### Part C: Recrystallisation of Diphenylmethanol

1. Recrystallise the crude product from hexane, being careful not use too much solvent. Hexane is flammable, and low-boiling, so keep the hot plate settings below maximum to minimise the risk of fire.
2. Collect the purified diphenylmethanol, and, once dry, determine its mass. Calculate the percent recovery of the recrystallisation, as well as the overall yield of the reaction.
3. Keep 0.1 g of the recrystallised product aside for TLC analysis later.
4. Determine the melting point of the recrystallised material at some point, this can be done now, or sometime during Part D below.

#### Part D: TLC Analysis

1. Prepare solutions of benzophenone, crude diphenylmethanol and recrystallised diphenylmethanol by dissolving 0.1 g of solid in 1 mL of dichloromethane. Use vials instead of test tubes, if possible.
2. The eluant for developing the TLC is 1 mL of ethyl acetate dissolved in 5 mL of ligroin. You will need 12 mL of this eluant.
3. Spot the TLC with the three solutions and develop the plate.
4. Circle the spots under the UV lamp, and calculate R<sub>f</sub> values.

For the reduction of benzophenone, 0.0074 mol of benzophenone was dissolved in 9 mL of methanol in a 50 mL Erlenmeyer flask, and 0.0026 mol of sodium borohydride was dissolved in 4.5 mL of cold water in a similar separate flask. Sodium borohydride solution was added to the ketone solution dropwise, and a pasteur pipette was used to swirl the flask between each addition to disperse cloudiness prior to adding the next drop. When the dropwise addition was completed, a stirbar was placed in the reaction flask, and the flask was stirred until a heavy slurry of precipitate formed, which took between 30 minutes and 1 hour.

Using gloves during the collection of the diphenylmethanol, 30 g of crushed ice was placed in a 250 mL beaker, and 3 mL of concentrated HCl was then carefully added to the frozen water. The reaction mixture was then slowly poured into the acid in the fumehood very carefully so as to avoid foaming over. The solid product was collected by suction filtration, and the crystals were washed twice with 15 mL of ice-cold water. The crystals were dried thoroughly to avoid recrystallisation due to hexane being immiscible with water. Once dried, the crude product was weighed, and a crude yield was calculated. 0.1 of the crude product was then placed aside for later TLC analysis.

During the third stage, the crude product was recrystallised from hexane. Care was taken not to use too much solvent, and because hexane is flammable and has a low boiling point, the hot plate settings were kept below maximum in order to minimise the risk of fire. The purified diphenylmethanol was then collected, and its mass was determined once dry. The percentage recovery of the recrystallisation and the overall yield of

the reaction were both calculated. 0.1g of the recrystallised product was kept aside for later TLC analysis. The melting point of the recrystallised material was now able to be determined at some point, or else during the next stage.

For the TLC analysis, solutions were prepared of benzophenone, crude diphenylmethanol, and recrystallised diphenylmethanol, by dissolving 0.1g of solid in 1 ml of dichloromethane. It was recommended to use vials instead of test tubes for this purpose. The eluant for developing the TLC was 1 mL of ethyl acetate dissolved in 5 mL of ligroin, and 12mL of this eluant was required. The TLC was spotted with the three solutions and the plate developed. The spots were then circled under the UV lamp, and the Rf values were calculated.