

Experiment 17

Phase Separation and Purification



The Task

The goal of this experiment is to use the separating funnel safely to separate two organic compounds.

Skills

At the end of the laboratory you should be able to:

- use a separating funnel,
- vacuum filter using a Büchner funnel,
- purify a solid material by recrystallisation.

Other outcomes

- Because of their different chemical properties, you will successfully separate benzoic acid and benzil using a separating funnel.

The Assessment

You will be assessed on all aspects of your ability to use a separating funnel correctly and safely. See Skill 5.

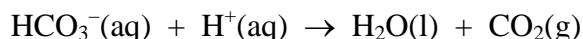
Introduction

The separation of compounds is one of the most common (and important) tasks in a chemistry lab. Whether the original source of material is from a natural source, an environmental sample or even the product of a synthetic procedure, we are usually faced with a mixture of compounds. We may only need one specific compound or we may wish to analyse all the different species present. In both cases the goal is to separate the different molecular species. All such separations depend on exploiting some physical difference between either the molecules in the mixture or some chemically modified versions of them. There is a huge array of separation techniques available and they make use of a range of different molecular properties, the most common being polarity, size, mass, crystal structure and charge (for ions).

One of the simplest methods makes use of the very different solubilities most compounds have in water compared to organic solvents. In this experiment you will use the fact that benzoic acid is soluble in organic solvents and that it can be easily converted to the water-soluble benzoate anion. To perform this separation you will use a sealable funnel specifically designed for the task.

The separating funnel

A separating funnel is used to separate two immiscible solvents that have been mixed together by shaking in the sealed apparatus (see Skill 5). During this process there is a build up of gas pressure in the separating funnel which **MUST** be released. The gas can be produced because one of the solvents (*e.g.* ether) is volatile. The action of shaking and holding the separating funnel in your warm hands can give the molecules enough kinetic energy to undergo a transition from the liquid to the gas state. Alternatively, a chemical reaction could occur in the separating funnel, generating a gas as the product. For example, a solution of sodium hydrogencarbonate is often used in the separating funnel to “mop up” any remaining acid from the reaction via the following reaction.



The carbon dioxide gas, $\text{CO}_2(\text{g})$, which is formed through this reaction can cause a sudden and dangerous build up of pressure within the separating funnel. If this happens the stopper can be blown out of the top and the contents of the separating funnel sprayed over you and your neighbours.

Safety

Chemical Hazard Identification

acetone - hazardous, highly flammable, irritating to eyes.

benzil - irritating to eyes, respiratory system and skin.

benzoic acid - Harmful if swallowed; irritating to the eyes.

diethyl ether - Extremely flammable, harmful if swallowed; vapours may cause drowsiness and dizziness.

ethanol - highly flammable, irritant.

hexane - highly flammable, irritant.

10 M hydrochloric acid - causes burns; irritating to respiratory system.

2 M sodium hydroxide - can cause severe skin and eye burns.

2 M hydrochloric acid – hazardous. Slightly corrosive, irritant.

brine (saturated sodium chloride solution) - not hazardous

anhydrous sodium sulfate - not hazardous

Risk Assessment and Control

Moderate risk.

Ether represents an extreme fire hazard. No naked flames are to be used in the laboratory for the duration of this experiment.

Pressure build up in the separating flask is a constant danger. Immediate and constant venting is essential. Re-read Skill 5 before you begin the experiment.

The recrystallisation of benzoic acid involves boiling water. Take care not to burn nor scald yourself.

Waste Disposal

Dispose of all your compounds in the appropriately marked containers in the fumehood. Waste ether is to go into the ether residues container in the fumehood. Waste NaOH and HCl solutions can be washed down the sink.

Experimental

This experiment is to be carried out in pairs.

Part A What solvents should we use?

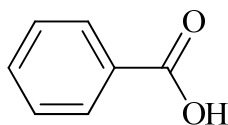
- (A1) Take 0.1 g of benzil in 6 separate semi-micro test tubes and test its solubility in the following: water, 2 M NaOH, 2 M HCl, diethyl ether, ethanol, hexane. Record all your results in your logbook.
- (A2) Repeat step (A1) using 0.1 g of benzoic acid instead of benzil.
- (A3) Test the miscibility of water with the following organic solvents: ethanol, hexane, ether. Record all your results in your logbook.

For your logbook

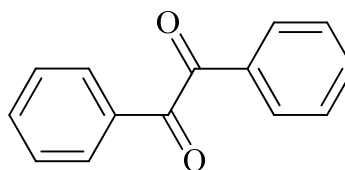
Which pair of solvents from the list given in (A1) will be suitable for separating a mixture of benzil and benzoic acid?

Part B The separation of benzil and benzoic acid

Now you will use the separating funnel to separate two different organic chemicals: benzoic acid and benzil.



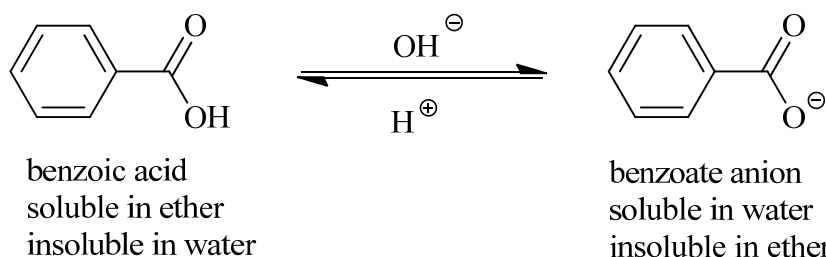
benzoic acid



benzil

In the separating funnel a compound is extracted from one solvent into another immiscible solvent by mixing the two solvents and allowing the solvents to separate. Separation is effected by running off the bottom (usually aqueous) layer via the tap at the bottom and then pouring the top (usually organic) layer out the top.

As you should have discovered for yourself in step (A2), we can manipulate some compounds (*e.g.* benzoic acid) so that they become soluble in the aqueous phase. In the experiment today, a mixture of benzoic acid and benzil is dissolved in ether. The benzoic acid is converted to the water-soluble benzoate anion by treating it with a strong base, *viz.* a solution of sodium hydroxide. Benzil does not react with sodium hydroxide, so it remains dissolved in the ether. After separation of the aqueous and organic layers, the benzoic acid is recovered from the aqueous solution by adding hydrochloric acid.



Do not begin this part of the experiment until you have been instructed in the use of the separating funnel by your demonstrator and read Skill 5.

- (B1) Check the tap of your 250 mL separating funnel to ensure that it doesn't leak and the tap moves smoothly. Set up the funnel in a retort ring attached to the stand on your bench (see Skill 5).
- (B2) Label two 100 mL conical flasks, aqueous layer and ether layer.
- (B3) Weigh out 2.0 g of the mixture (see Skill 3.1B) which consists of 50 % benzoic acid and 50 % benzil.
- (B4) In a 100 mL beaker dissolve the mixture in 40 mL of ether and transfer the solution to the separating funnel (*make sure the tap is closed*) using a glass funnel.
- (B5) Add 10 mL of 2 M NaOH.
- (B6) Stopper the separating funnel well, invert, DO NOT SHAKE, and immediately open the tap to release pressure which develops as the ether mixes with the alkaline solution.
- (B7) Close the tap, shake briefly and re-open the tap to relieve the pressure. Repeat this procedure until there is no more gas escaping and return the funnel to the stand, remove the stopper and allow the layers to separate.
- (B8) Use your dropper to add a drop of water and watch which layer it "goes to"; this is the aqueous layer. Does this agree with your results from step (A3)? In your logbook, draw a picture of the separating funnel, labelling the aqueous and ether layers and showing the species present in each layer.
- (B9) Run off the aqueous layer into the appropriately labelled conical flask.
- (B10) Repeat steps (B5), (B6), (B7) and (B9). Add 5 mL of water and repeat steps (B6), (B7) and (B9). All aqueous extracts should be run into the same flask in (B9).
- (B11) Set aside the combined aqueous layers.

You now need to assign tasks. One student in each pair should recover and purify the benzoic acid (Part C), the other should recover and purify the benzil (Part D).

Part C Recovery and purification of benzoic acid (one student of pair)

- (C1) Place ~ 60 mL of water in a 100 mL beaker (labelled with your name), add 2 boiling chips and heat to boiling on a hot plate on your bench. This is for your recrystallisation of benzoic acid later.
- (C2) Take the aqueous layer flask to the steam bath in the fumehood, add a few boiling chips and boil off any dissolved ether.
- (C3) Cool the solution and cautiously use a dropper to slowly add 10 M HCl with stirring, until the precipitation of white crystals appears complete. Check your solution is acidic to litmus paper - remove one drop on a glass rod and place on blue litmus. Add more 10 M HCl (dropwise) if the solution is still basic.

- (C4) Cool in ice if necessary and collect the crystals using a Büchner funnel (Skill 6.2).
- (C5) Transfer the crude benzoic acid to a clean 100 mL conical flask and recrystallise (Skill 9) from boiling water on the hot plate. (You will need about 30 mL of solvent.) When crystallisation appears complete, place the flask in ice for 5 minutes and then collect the crystals using a Hirsch funnel (Skill 6.2).
- (C6) Wash the crystals with a little cold water and dry at the pump. This can take a while when water has been used as the recrystallisation solvent.
- (C7) In your logbook record the weight of the crystals and describe what the crystals look like, including whether they are 'pure white' or have a 'yellow tinge'.

Part D Recovery and purification of benzil (other student of pair)

- (D1) The benzil remains in the ether layer. Wash the ether layer with 10 mL of brine (saturated NaCl solution). This reduces the amount of water dissolved in the ether. Remove the stopper and run off the lower layer into a conical flask for disposal down the sink later.
- (D2) Carefully pour the ether from the top of the separating funnel into the flask labelled ether layer, being careful that no droplets of water enter the flask.
- (D3) Add a spatulaful of anhydrous sodium sulfate (drying agent) and swirl. If your ether is really 'wet' you will need to add more sodium sulfate. If the ether is dry, you should see grains of sodium sulfate moving freely when you swirl the flask.
- (D4) Remove the sodium sulfate by gravity filtering the benzil solution through a fluted filter paper (Skill 6.1) into a clean and dry 100 mL conical flask.
- (D5) Add a few boiling chips and boil off the ether on the steam bath in the fumehood. You will end up with a yellowish sludge (your crude product). Remove the conical flask from the steam bath.
- (D6) Recrystallise the benzil (Skill 9) from ethanol (you will only need about 10 mL) on the steam bath. If crystallisation does not occur on cooling, consult your Demonstrator. After the crystals have formed, cool in ice for ~5 minutes and collect the crystals in a Hirsch funnel (Skill 6.2).
- (D7) Wash the crystals with a **small** amount of cold ethanol and dry at the pump.
- (D8) In your logbook record the weight of the crystals and describe what the crystals look like, including whether they are 'yellow' or have a 'white tinge'.

Cleaning up: Wash the glassware with ethanol to remove any crystals of benzil or benzoic acid, before placing it back into the cupboards.

Group Discussion

Answer these questions in your logbook, ready for group discussion.

What errors could be made using a separating funnel? How can you avoid them?

What did the recrystallised benzoic acid and benzil crystals look like? Do you think they were 'pure'? Why? How could you check?

If you started with 1 g of benzoic and benzil, how much of the purified compounds did you recover? Experimentally, where could you have lost it?