RECRYSTALLISATION OF BENZOIC ACID

EXPERIMENTAL TECHNIQUES REQUIRED

Recrystallisation (T 2), vacuum filtration (T 3)

OTHER DOCUMENTS : Report template

EXPERIMENTAL PROCEDURE

- **Treat ALL chemicals with caution. Avoid inhaling their vapours and avoid contact with eyes, skin and clothing.**
- **Work in a fumehood**
- **Heating apparatus gets hot ! Use caution and assume it is hot if in any doubt.**
- **The crude mixture is harmful if swallowed and avoid contact with your eyes**
- **The crude mixture is toxic to aquatic life (don't wash it down the sink)**
- **Steam can cause burns**

Heat 40-50ml of distilled water in a beaker with a stirrer bar on a hot plate - stirrer in order to have hotwater to use for the recrystallisation. Don't let it boil dry.

In a small Erlenmeyer flask, weigh the crude benzoic acid mixture (approx.. 0.5 g, accurately weighed) noting the appearance of the crude mixture. Dissolve the crude mixture in the minimum volume of hot water. In order to do this, add 10 mL of hot water to the Erlenmeyer and keeping it hot (boiling) to dissolve the crude benzoic acid. Check to see if any solid crude benzoic acid is visible. If there is undissolved benzoic acid, add another 1 ml of hot water and repeat the process. If you add too much hot water then you will need to heat the solution to allow some of the water to evaporate. However, if you over concentrate the solution, then your recrystallised product might be contaminated with the blue crystals.

Once all of the solids have dissolved, set the flask aside, cover it (with an upside down beaker or watch glass) and allow it to cool slowly and undisturbed for at least 20 mins. While it is cooling, collect and assemble the apparatus to set up a vacuum filtration using a Buchner funnel ready to filter (including the filter paper). *Get your TA to check your set up once you are done*. Prepare an ice-water cooling bath (ice in a large beaker that is large enough to put the Erlenmeyer in without risk of it tipping over). Have the ice bath ready to use to cool your recrystallising solution and use the cooling bath to chill about 8 mL of distilled water in a test tube.

After 10 mins of cooling, check the flask to see if any crystals are growing. If there are no crystals, then use a glass rod to *carefully* scratch the inside of the flask to help induce crystallisation.

Check with your TA if nothing happens. If crystals are present, then leave the flask to continue to cool to room temperature for another 10 mins.

Once the flask has cooled to room temperature, secure it in the ice-water bath (i.e. clamped to a retort stand) and leave it to cool for a further 20 mins.

For the vacuum filtration, turn on the vacuum and then wet the filter paper in the Buchner funnel with a little cold water. Collect the crystals of benzoic acid by vacuum filtration. The water should filter quickly, if not check the vacuum and the seal of the conical adaptor. Scrape all of the crystals out of the Erlenmeyer flask using a spatula (or maybe a stirring rod) and transfer them to the filter funnel. Rinse the Erlenmeyer flask with 1 or 2 mL of the ice-cold water (to get the last of the crystals) and transfer it to the filter funnel (and also to rinse the crystals). Allow the crystals to dry in the filter funnel by keeping the vacuum running and periodically gently stirring the crystals with a spatula. Once the crystals move freely and appear dry, remove the filter funnel and turn off the vacuum. Transfer your purified crystals to a preweighed watch glass to determine the weight of purified benzoic acid obtained the determine your % recovery based on the amount of crude benzoic acid mixture you had used. Record your observations of the purified benzoic acid crystals.

CLEAN UP

Organic liquids go in the organic waste container (red drum) in the waste fumehood.
Aqueous solutions go in the aqueous waste container (white drum) at the waste station.
Any solid chemical samples go into the solid waste container in the waste fumehood.
Wipe down your work area at your benchtop and fumehood.

REPORT

Before writing any Chem 351 laboratory report, we strongly recommend that you review section 8 in the introductory section of the student laboratory manual that discusses how to write reports and/or from "writing reports" on the course website. Students often don't get the grades they would like because they make errors that are addressed in that section of the manual. These are avoidable errors.

The "report" for this experiment is to be completed in the simple template provided including answering the questions. Remember that more is not necessarily better. It is important to be **accurate** and **concise** rather than verbose and vague. Proper English should be used and it should be written in your own words.