OCR (A) Chemistry A-level

PAG 6: Synthesis of an Organic Solid

6.3 Preparation of Methyl 3-nitrobenzoate

Method

Part 1: Preparation

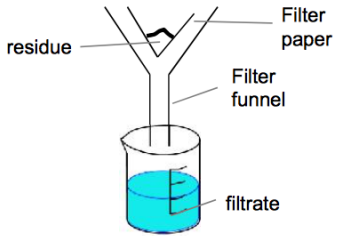
1. Mix 2.5 cm^3 of methyl benzoate with 5 cm^3 of concentrated sulfuric acid in a 50 cm^3 conical flask. Place the conical flask into an ice bath.
2. In a test tube, add 2 cm^3 of concentrated sulfuric acid and 2 cm^3 of concentrated nitric acid. Place the test tube into the ice bath.
3. Once cooled, use a glass dropping pipette to add the mixture from the test tube to the conical flask dropwise.
4. Stir continually, ensuring that the temperature remains below 10℃.
5. Leave the mixture at room temperature for about 15 minutes.
6. Add about 25 g of crushed ice to the conical flask and stir until it melts and a precipitate forms.
7. Filter the mixture under reduced pressure to obtain the crystals.
8. Wash the crystals with cold water.
9. Retain a few crystals for part 3.

Part 2: Recrystallisation

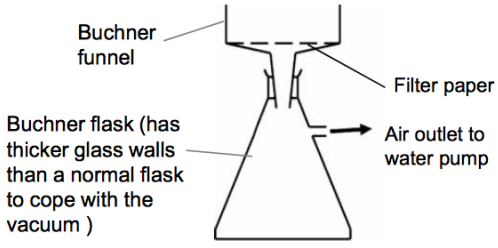
1. Heat about 10 cm^3 of ethanol to 60℃ using a water bath.
2. Dissolve the impure compound in the minimum volume of hot ethanol in a test tube.
3. Filter the hot solution to remove any insoluble impurities.
4. Leave the filtrate to cool to room temperature.
5. Use suction filtration to separate the crystals from the solution.
6. Wash the crystals with distilled water.
7. Leave the vacuum on for several minutes to dry the crystals.

When making an insoluble salt, normally the salt would be removed by **filtration,** washed with **distilled water to remove soluble impurities** and then **dried on filter paper**

Filtration



This is gravitational filtration. Use if small amounts of solid are formed.



This is vacuum filtration. The apparatus is connected to a water pump which will produce a vacuum. Use if larger amounts of solid are formed.

Ways in which product may be lost:

- Crystals lost when filtering or washing.
- Some product may stay in solution after recrystallization.
- Other side reactions may occur.

Part 3: Thin layer chromatography

1. Draw a line in pencil about 1 cm from the bottom of the TLC plate. Mark three equally spaced crosses along the line, to indicate the starting positions of the samples.

2. Use a capillary tube to spot samples from part 1 and part 2 and the pure methyl 3-nitrobenzoate on the TLC plate. Each spot should be as small as possible and the spot should be left to dry before the sample is spotted again on top.

3. Add about 10 cm^3 of solvent into a 100 cm^3 beaker and cover with a watch glass. The watch glass will prevent evaporation of the solvent.

4. Place the TLC plate into the beaker, making sure that the level of the solvent is below the pencil line. Make sure the watch glass is placed on top of the beaker.

5. When the solvent is about 1 cm from the top of the plate, remove the TLC plate and mark the solvent front with a pencil. Leave the plate to dry in a fume cupboard.

6. Place the plate in a beaker with iodine crystals and cover with a watch glass. Once the spots become visible mark them lightly with pencil.
7. Calculate the Rf values of the observed spots.

Note

- If the sample is too concentrated, then the spots would overlap.

Safety

- Methyl-3- nitrobenzoate - causes skin irritation and serious eye irritation.
- Iodine crystals - harmful if in contact with skin; harmful if inhaled.
- Methyl benzoate – harmful if swallowed.
- Ethanol - highly flammable.