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Hydrolysis of ethyl benzoate

*UNIT 3 PPA 3*

**Introduction**

Benzoic acid can be prepared by the alkaline hydrolysis of the ester, ethyl benzoate:



If sodium hydroxide is used, then the residual solution will contain sodium benzoate. Insoluble benzoic acid can be displaced from this solution by acidification. It can then be filtered off and purified by recrystallisation from water. The percentage yield of benzoic acid can be calculated and its melting point determined.

**Health and Safety**

Wear eye protection and if any chemical splashes on your skin wash it off immediately.

Ethyl benzoate is of no significant hazard.

2 mol l-1 sodium hydroxide is corrosive to the eyes and skin. Gloves and goggles should be worn.

5 mol l-1 hydrochloric acid is irritating to the eyes, lungs and skin and if swallowed. Wear gloves.

The product, benzoic acid is a skin, eye and respiratory irritant.

Ethanol is volatile, highly ﬂammable, irritating to the eyes and intoxicating if inhaled or ingested. But only a small amount of ethanol is produced in the reaction.

**Requirements**

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| ethyl benzoate | 2 mol l-1 sodium hydroxide |
| 5 mol l-1 hydrochloric acid | deionised water |
| l00 cm3 round-bottomed ﬂask | cork ring |
| condenser | heating mantle |
| anti-bumping granules | dropper |
| Buchner funnel and ﬂask blue litmus paper or pH paper | 100 cm3 measuring cylinder  |
| glass ﬁlter funnel | melting point apparatus |
| capillary tube | thermometer |
| water pump | balance (accurate to 0.01 g) |
| oven | hot plate |
| ﬁlter papers | 250 cm3 glass beakers |
| clock glass | glass stirring rod |

**Procedure**

1. Weigh the 100 cm3 round-bottomed ﬂask supported on a cork ring. To the ﬂask, add about 5 g of ethyl benzoate and reweigh the ﬂask and its contents.
2. To the ethyl benzoate, add approximately 50 cm3 of 2 mol l-1 sodium hydroxide and a few anti-bumping granules.
3. Set up the apparatus for heating under reﬂux. Using a heating mantle, reﬂux the reaction mixture until all oily drops of the ester have disappeared. This may take 45 - 60 minutes.
4. Allow the apparatus to cool and then transfer the reaction mixture to a 250 cm3beaker.
5. Slowly and with stirring, add 5 mol l-1 hydrochloric acid to the reaction mixture to precipitate out the benzoic acid. Continue adding the acid until no more precipitation takes place and the mixture tums acidic - test with blue litmus paper or pH paper. (About 30 cm3 of acid will be required)
6. Allow the mixture to cool to room temperature and filter off the precipitate at the water pump and wash the crude benzoic acid with a small volume of water.
7. Transfer the crude benzoic acid to a 250 cm3 beaker and recrystallise it from about 100 cm3 of water.
8. Filter off the crystals of benzoic acid at the water pump and wash them with a small volume of water. Allow air to be drawn through the crystals for a few minutes in order to partially dry them.
9. Weigh a clock glass and transfer the crystals to it. Dry the crystals in an oven at about 70°C and then reweigh the clock glass and crystals. (alternatively, the crystals of benzoic acid can be dried in the open air or in a desiccator with silica gel or anhydrous calcium chloride as desiccant.)
10. Determine the melting point of the benzoic acid.
11. Calculate the percentage yield of benzoic acid.

**Notes**

It is worthwhile emphasising to the students that reﬂuxing should continue until hydrolysis is complete.

If time permits, a second crop of crystals could be derived from the mother liquor.

**Technician guide**

Requirements per student (or group)

Reagents

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| ethyl benzoate (~5 g) | 2 mol l" sodium hydroxide (~50 cm3) (80 g sodium hydroxide per litre) |
| 5 mol 1-1 hydrochloric acid (~30 cm3) (430 cm’ concentrated hydrochloric acid per litre) | anti-bumping granules |
| blue litmus paper or pH paper | deionised water |

Apparatus

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| 100 cm3round-bottomed ﬂask (1) | cork ring (1) |
| access to balance (accurate to 0.01 g) | 100 cm3 measuring cylinder (1) |
| condenser (1) | heating mantle (1) |
| 250 cm3 glass beakers (3) | glass stirring rod (1) |
| Buchner funnel and ﬂask (1) | water pump (1) |
| access to hot plate | glass ﬁlter funnel (1) |
| clock glass (1) | access to oven |
| access to melting point apparatus | capillary tube (1) |
| 0 - 200°C thermometer (1) | ﬁlter papers to fit Buchner funnel (2) and |
| ﬁlter funnel (1) | dropper (1) |

**Notes**

If time permits, a second crop of crystals could be derived from the mother liquor.

The crystals of benzoic acid could equally well be dried in the open air or in a desiccator with silica gel or anhydrous calcium chloride as desiccant.