Oxford Cambridge and RSA

A Level Chemistry B (Salters)

H433/03 Practical skills in chemistry

Practical Insert

Wednesday 20 June 2018 – Morning

Time allowed: 1 hour 30 minutes

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INSTRUCTIONS

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INFORMATION

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Preparing an ester

An A level chemistry class is given a practical which requires them to prepare a sample of the ester, phenyl benzoate. They are also asked to calculate the percentage yield they achieve and to assess the purity of their product.

The students carry out the experiment as shown in the procedure below.

Procedure

Preparation of phenyl benzoate

1. Transfer about 5.0 g of solid phenol into a weighing bottle and weigh it to the nearest 0.01 g.
2. Pour 90 cm^3 of 2 M sodium hydroxide into a conical flask and add the phenol from the weighing bottle.
3. Reweigh the weighing bottle to the nearest 0.01 g.
4. In a fume cupboard pour 9 cm^3 of benzoyl chloride into the conical flask.
5. Insert the bung securely and shake the bottle for 15 minutes, carefully releasing the pressure every few minutes as the flask gets warm.
6. Cool the flask under cold, running tap-water.
7. Filter the crude product using a suction filtration apparatus.

   Use a spatula to break up the lumps of ester on the filter paper, being careful not to puncture the filter paper.
8. Pour more water over the crude ester to remove any remaining benzoyl chloride.

Recrystallisation

9. Transfer the crystals to a boiling tube and just cover them with ethanol.
10. Place the boiling tube in a water-bath or beaker of hot water, kept at about 60 °C and stir with a glass rod.
11. If some solid ester is still visible, add just enough ethanol to dissolve it completely after stirring.
12. In order to allow the separation of the ester as a solid rather than an oily liquid (phenyl benzoate has a low melting point) add more ethanol to double the volume of solution.
13. Place one drop of the solution onto a white tile and add one drop of neutral iron(III) chloride solution.
14. Cool the solution in an ice-water mixture until crystals appear.
15. Filter the crystals through the suction apparatus using a clean Buchner funnel and filter paper. To avoid losing any solid break the vacuum and use the filtrate to rinse the boiling tube into the funnel.
16. Using suction again rinse the crystals with about 1 cm^3 of cold ethanol and drain thoroughly.
17. Press the crystals between sheets of filter paper to remove excess solvent. Then put the crystals on another dry piece of filter paper and place in a warm oven for an hour.
18. Weigh the dry crystals in a pre-weighed specimen bottle and record the mass of your sample of phenyl benzoate.
19. Using melting point apparatus determine the melting point of your crystalline sample.

[Reference: Modified from 'Independent Learning Project for Advanced Chemistry; More functional groups – ILPAC unit 03', Inner London Education Authority, first published 1984 by John Murray (Publishers) Ltd]

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Results

Mass of weighing bottle and phenol / g	20.73
Mass of weighing bottle after emptying / g	15.82
Mass of specimen bottle / g	5.61
Mass of specimen bottle and phenyl benzoate / g	9.71
Melting point of product / °C	66–68

Other observations

Melting point of phenyl benzoate from data book = 70 °C

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