A level Chemistry B

H433/03 Practical skills in chemistry

Question Set 8

1 (a) (i) This question refers to the **Practical Insert** that is provided as an insert to this paper.

The equation for the reaction producing phenyl benzoate is as follows:

$C_6H_5COCl + C_6H_5OH \rightarrow C_6H_5COOC_6H_5 + HCl$

Draw a structural formula for phenyl benzoate, showing the bonding in the ester group. **[1]**

1 (ii) Use the student results to calculate the percentage yield of phenyl benzoate obtainedfrom the practical.

percentage yield = **[3]**

……%

1 (b) (i) In **step 8** of the procedure the water reacts with any remaining benzoyl chloride.

Write the equation for this reaction. **[1]**

1 (ii) Suggest and explain the reason for **step 13** in the procedure. **[1]**

1 (iii) Describe the practical procedure used to measure the melting point of an organic solid.You **do not** need to discuss the type of melting point apparatus you use **[3]**

1 (iv) What information can the students get from their melting point? **[1]**

1 (v) The recrystallisation procedure uses ethanol as the solvent.

Give the key properties needed by a solvent to be effective in recrystallisation. **[1]**

1 (c) The students carry out thin layer chromatography of the phenyl benzoate formed.
One student states that this will enable them to assess the purity of their product.

Comment on the validity of this statement.
You should describe any possible observations to back up your comments. **[4]**

Total Marks for Question Set 8 = 15

Practical Insert

Question Set No: 1

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 weighingbottle.
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 pressureevery 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 thefilter 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 aglass 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 benzoatehas 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) chloridesolution.
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 crystalson 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 ofphenyl 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]

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