

4.10 Key Steps in Laboratory Synthesis 2 Answers

1. a) The benzoic acid is referred to as “crude” because it has not been purified and will likely contain impurities.

b) The student should carry out the following steps to recrystallise the benzoic acid:

Dissolve the impure benzoic acid in a minimum volume of a hot solvent.

Hot filtration is carried out of the resulting mixture to remove any insoluble impurities.

The filtrate is then cooled slowly to allow crystals of the pure compound to form, leaving soluble impurities dissolved in the solvent.

Finally the product crystallises out and the impurities will remain in the solvent. The mixture can then be filtered and the benzoic acid crystals are washed and dried.

c)i) A sample of the solid benzoic acid is placed in a glass capillary tube by gently tapping one end. The capillary tube is then placed into the melting point apparatus along with a thermometer. The temperature is gently increased. The benzoic acid can be viewed through the eyepiece and when it melts the temperature is recorded.

ii) The melting point for benzoic acid is approximately 122°C. This means that the sample still contains impurities.

d)i) Mixed melting point is when a synthesised sample (in this case benzoic acid) is mixed together with a commercial sample which is pure. The mixed sample is placed in a glass capillary tube and the melting point taken.

ii) The second round of recrystallisation should increase the melting point closer to the actual value. Moreover, the melting point range should be narrower.

e) If a pure sample of benzoic acid is available, then a tlc analysis could be carried out. Both samples could be spotted on a tlc plate and compared.

2. a) Often, to observe the spots on a tlc plate a developing agent must be used or an ultraviolet lamp.

b) i) Methyl ethanoate: 0.12 Ethanol: 0.40

ii) Ethanol should be used. The spot with methyl ethanoate moved a very short distance.

c)i) The pure caffeine and the caffeine from the residue should be spotted onto a tlc plate. If both spots have the same R_f value then this will identify caffeine from the residue sample.

ii) If the tlc plate indicates that the residue sample has only one spot and the same R_f value as pure caffeine, then it can be concluded that it is pure.



3.a) $C_8H_9NO_2$

- b) Ethanol is a suitable solvent to recrystallise paracetamol because it is only partially soluble when cold but very soluble when warm.
- c) The melting point would be lower and have a broader range.
- d)i) Pure paracetamol
 - ii) 0.72 (range of 0.69-0.75 acceptable)
- e) The paracetamol appears to now be pure.

