

Purifying by recrystallisation – teacher notes

Introduction

Selecting a good solvent is the key to performing a successful recrystallisation. Ideally, the material to be recrystallised should be sparingly soluble at room temperature and yet quite soluble at its boiling point. Water can be used for recrystallising aspirin because it is cheap, readily available and safe. However, heating aspirin in water partially decomposes it although the quantity of crystals obtained may be satisfactory. The product can be tested with Fe^{3+} (aq) to see whether any improvement in purity can be detected.

Conclusions

- Aspirin crystallises as fine white or transparent needles.
- Some of the desired material is always lost along with the impurities in a crystallisation. The technique can work only if the impurities in the crude product make up a small fraction of the total mass or have very different solubilities.
- A pure substance forms crystals with a characteristic shape. Impurities disrupt the regular array of molecules in a crystal and so lead to irregularly shaped crystals.

Correct responses

Stages	Results
Shake your sample with the solvent and warm to dissolve.	The product dissolves only in the hot solvent. Soluble impurities also dissolve, but there should not be so many impurities that the solution is saturated. Insoluble impurities stay in suspension.
Filter the solution hot. Use a Buchner funnel and pump to make sure that the solution does not cool too much while it is being filtered. Throw away the residue.	Insoluble impurities stay on the filter paper, soluble impurities and the product stay in solution and are found in the filtrate.
Allow the solution to cool slowly. If no crystals appear, add a single crystal as a 'seed' or stir vigorously for a few minutes. (If still no crystals appear you have probably added too much solvent!)	The product becomes less soluble as the mixture cools and eventually crystallises. Soluble impurities are less concentrated so they stay in the solution. (NB if the mixture is cooled too quickly solvent can become trapped in the crystals and is difficult to remove.)
Filter the solution cold. Use a Buchner funnel and pump. Keep the residue.	The crystals are separated from the solvent which still contains soluble impurities, leaving the product contaminated only with solvent in which is dissolved a small amount of soluble impurity.
Wash the residue with a small amount of cold solvent. Why do you need to use the solvent cold?	Contaminated solvent passes through the funnel, leaving only product and pure solvent on the filter paper. Warm solvent would dissolve a significant amount of product and it would pass through the filter paper.
Dry the product on a watch glass, either at room temperature or in an oven.	Because the solvent is pure it leaves no residue apart from the product.

Further investigations

- Try further recrystallisation of the product to see if even better quality crystals are produced.
- If water was used as the solvent try recrystallising from ethyl ethanoate.
- Solubility depends on the polarity of the solvent and solute. Investigate the solubility of aspirin in different solvents, predicting the solubility based on the structure of the solvent molecules.

Answers

1. Most material is lost in the solvent.
2. It is mostly impurity that is lost.
3. The sample was contaminated with solvent.