

Purifying by recrystallisation – student sheet

Introduction

When an organic compound has been made it needs to be purified, particularly if it is a pharmaceutical chemical. This is because most organic reactions produce by-products but, even if the reaction is a 'clean' one, the purity standards for many products are so stringent that small amounts of other compounds have to be removed.

In the laboratory, this is often done by recrystallisation. The general method is to find a solvent that dissolves the product more readily at high temperature than at low temperature, make a hot solution, and allow to crystallise on cooling.

The crude product might contain;

- impurities which are insoluble in the solvent;
- impurities which are slightly soluble in the solvent; and
- impurities which dissolve readily in the solvent.

The solvent itself has also to be removed or it behaves as an impurity in its own right. It must not leave behind any residue.

One simple way to tell whether an organic compound is pure is to measure its melting (or boiling) point. A pure compound melts sharply: if impurities are present it melts slowly (over a range of temperature) and the melting point is lower than that of the pure compound.

In this activity, you are going to look at the stages involved in recrystallisation and draw up a procedure for carrying out the process. You may then use your plan to recrystallise a sample of the aspirin that you have made.

Below is a list of the stages in recrystallisation, but not necessarily in the correct order. Think about each of the types of impurity above and put these stages in the correct order. Then match each of them to the boxes which describe what has been achieved. You could arrange the results first or you could cut out a copy of the chart and rearrange it.

Stages	Results
Dry the product on a watch glass, either at room temperature or in an oven.	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.
Shake your sample with the solvent and warm to dissolve.	Insoluble impurities stay on the filter paper, soluble impurities and the product stay in solution and are found in the filtrate.
Wash the residue with a small amount of cold solvent. Why do you need to use the solvent cold?	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.

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!)	Because the solvent is pure it leaves no residue apart from the product.
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.	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.
Filter the solution cold. Use a Buchner funnel and pump. Keep the residue.	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.)

When you have done this activity you should try out your method using half of your sample of aspirin. Water is a good solvent to use but you get better results using ethyl ethanoate. You might like to organise your group to compare the two solvents.

Weigh your sample before you start and again afterwards to find out how much you lose in the recrystallisation.

Questions

1. Where do you think most material is lost?
2. Do you think it is mostly product, mostly impurity or some of each?
3. What conclusion would you come to if your sample weighed more after recrystallisation?

In the next activity you measure the melting point of your sample before and after recrystallisation.