Lesson 10: Key Steps in Laboratory Synthesis 2

*Read through the lesson notes. You can write them out, print them or save them.

*Once you have tried to understand the lesson answer the questions that follow and self-evaluate your work by checking the answers.

Learning Intention

-Learn about the laboratory technique recrystallisation.

-Learn about methods of identifying products, these include melting point analysis and tlc (thin layer chromatography).

Background

This is the second lesson in key steps in laboratory synthesis. It concentrates on what a chemist will do in the laboratory once they have synthesised and isolated a product. It is essential to purify the product first and then ensure it is the actual desired product through identification.

Purification: Recrystallisation

When a product has been synthesised, it is common that it will contain impurities that will show up on spectra or they will affect the results of physical tests carried out. To purify a product the technique of recrystallisation is regularly carried out in the laboratory.

There are four main points to recrystallisation.

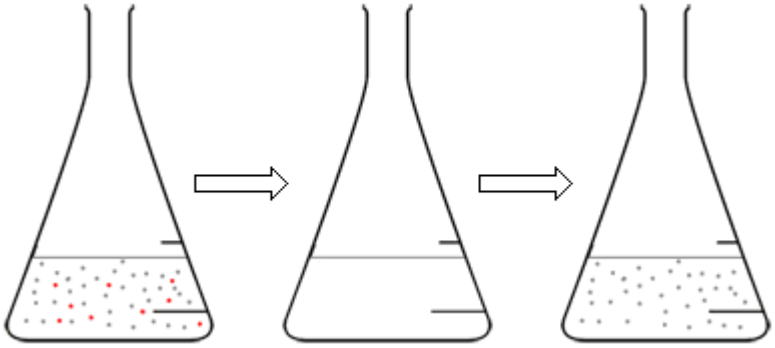
1. The impure solid is gently dissolved in a minimum volume of a hot solvent.

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

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

4. Finally the product crystallises out and the impurities remain in the solvent. The mixture is filtered and the crystals are washed and dried.

When choosing a suitable solvent for recrystallisation it should be one in which the product is only partially soluble when the solvent is cold but very soluble in the solvent when hot.



Product with impurities (in red).

Solution heated to dissolve both product and impurities.

When the solution cools down, the product comes "crashing out" and leaves behind the impurities.

A fluted filter paper is used as it provides a larger surface area and makes for a faster filtration than the usual filter paper cone.

It is good practice to put the filter paper and glass funnel in an oven before carrying out the filtration. This reduces the risk of crystals forming on the filter paper and the stem of the funnel.

<u>Identification</u>

1. Melting point analysis

   A small sample of the purified product is placed inside a thin glass capillary tube which is sealed at one end. This is then placed in the melting point apparatus and viewed through an eyepiece. When the solid turns to a liquid, the temperature of the thermometer is noted and recorded.

Melting point apparatus with eyepiece and thermometer.

Glass capillary tube with product.

The melting point of a solid product can be determined and compared with the literature value for the substance in question. If these are in close agreement, then the likely identity of the compound can be confirmed.

If the synthesised product is pure then the melting point will have a narrow range (maybe just 2°C) and accurately agree with the literature value. However, if there are impurities present then the melting point will be lower and have a broader range due to disruption in intermolecular bonding of the crystal lattice.

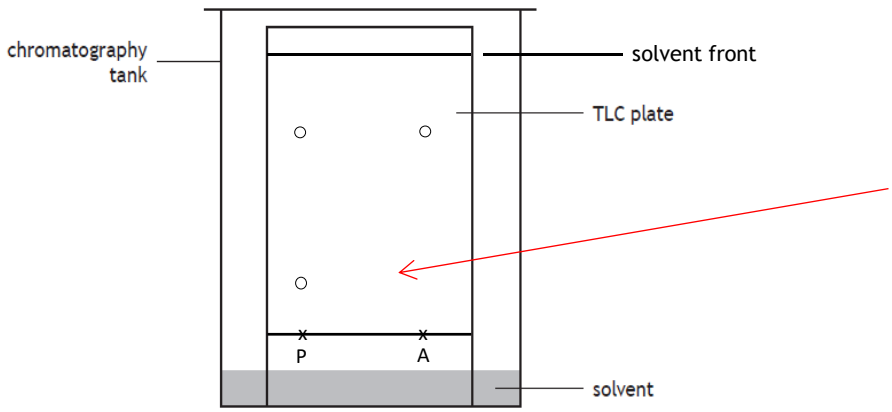
On some occaions, chemists carry out a mixed melting point analysis. For this analysis, a pure sample (if available) and a sample of the synthesised product are mixed and placed together in the glass capillary tube. The melting point of the mixture is determined in the same way.

2. TLC (thin layer chromatography)

Chromatography is a technique used to separate the components present within a mixture. It separates substances by making use of differences in their polarity or molecular size. TLC uses a fine film of silica or aluminium oxide spread over glass or plastic.

A sample solution is spotted on a TLC plate and then placed in a tlc chamber with a small volume of suitable solvent, which acts as the mobile phase. It is important that the volume of solvent just covers the bottom of the chamber and that it is below the level of the spot. The solvent then travels up the plate and the components of the sample separate out. Often, a pure substance will show up as only one spot on the developed chromatogram. This allows TLC to be used to assess the purity of a product prepared in the lab.

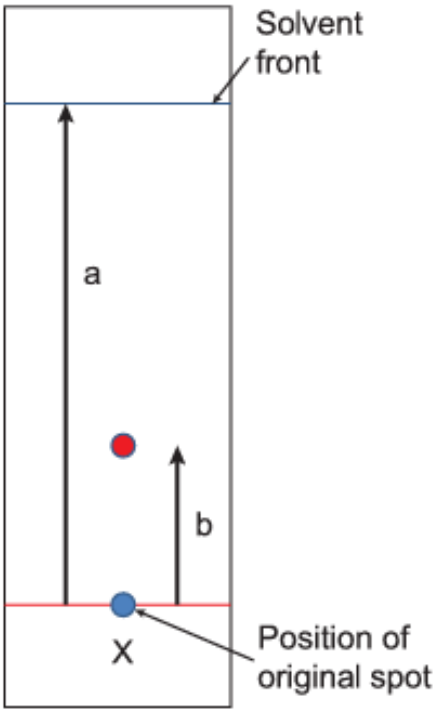
The spots are often not in the visible part of the electromagnetic spectrum and therefore need a developing agent or an ultraviolet lamp to be observed.



In this example, a student's product, P, is compared against a pure sample A, (known as a co-spot).

Note that the student's product has a slight impurity.

Retardation factor (R_f) values (distance travelled by compound/distance travelled by solvent) can be calculated and under similar conditions a compound will always have the same R_f value within experimental error.
R_f is measured by calculating the distance travelled by the substance divided by the distance travelled by the solvent front.



R_f of sample X is given by:

$$R_f = \frac{\text{distance travelled by sample}}{\text{distance travelled by solvent}}$$

$$R_f = b/a$$

This lesson gives an insight into the setting up of laboratory equipment. To help visualise these concepts better, the video clips below will facilitate your understanding of the main points to the lesson.

https://www.youtube.com/watch?v=j4p6KvuqsJ4
https://www.youtube.com/watch?v=GZjgrvujBmc
https://www.youtube.com/watch?v=qJLvB6NFnoA
https://www.youtube.com/watch?v=sh-96_KfqgY
https://www.youtube.com/watch?v=lj5OWzhZSac

Suggested Further Reading – Not mandatory.

→ Read Scholar Heriot-Watt/ Researching Chemistry Section 6.5, 6.7 and 6.8.

→ Read Bright Red text book pages 91, 94 and 95.

TASK

→ Answer the questions from Sheet 4.10 and check the answers when you have completed them.

4.10 Key Steps in Laboratory Synthesis 2

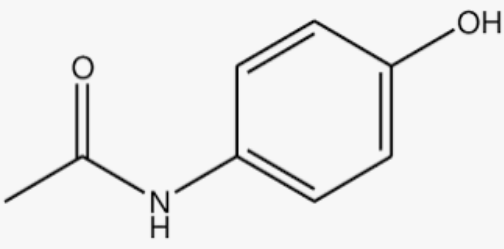
1. One of the steps from an instruction booklet for the synthesis of benzoic acid suggests that the "crude benzoic acid can be recrystallised from about 100cm^3 of water."
   a) Why is the benzoic acid referred to as "crude?"
   b) Explain accurately how a student could recrystallise the "crude" benzoic acid.
   c) The student dried the recrystallised sample of benzoic acid and tested its purity by carrying out a melting point analysis. They obtained a melting point range of 114-118°C.
      i) Explain how the student would prepare the sample and how they would use the melting point apparatus for the analysis.
      ii) Search Google for the literature value for the melting point of benzoic acid and comment on the purity of the sample of benzoic acid obtained by the student.
   d) The student decided to recrystallise the benzoic acid for a second time and then carried out a mixed melting point analysis.
      i) What is meant by mixed melting point analysis?
      ii) Comment on what effect the second process of recrystallisation should have on the melting point range and value of the sample.
   e) Explain which other type of analysis the student could carry out to determine the purity of the benzoic acid sample.

2. An Advanced Higher Chemistry project involves the extraction of caffeine from different brands of tea bags. A student wanted to analyse a residue that had been extracted by using thin layer chromatography.
   They set up two trials to determine which solvent was best to use for the mobile phase. They used a pure sample of caffeine on tlc plates as a control.
   The results from the trial are shown below.

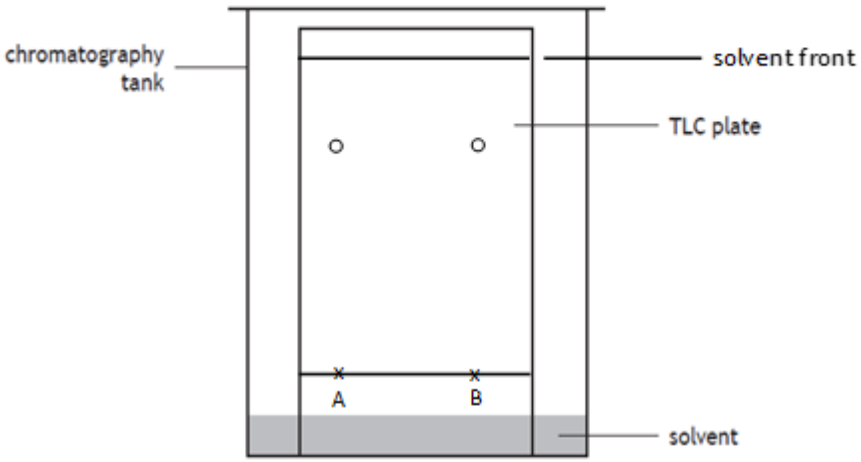
Solvent used	Distance to solvent front/cm^3	Distnace to spot/cm^3
Methyl ethanoate	6.5	0.8
Ethanol	5.7	2.3

   a) The student initially didn't observe any spots on the the tlc plates. Explain what they would have to do to observe the spots.
   b) i) Calculate the Rf value for caffeine using both solvents.
      ii) Which solvent should be used for the chromatography?
   c) How would thin layer chromatography be used to
      i) identify caffeine in the residue;
      ii) decide whether the sample is pure

3. 4-acetamidophenol is commonly known as paracetamol and can be readily synthesised in an organic laboratory. A sample of the synthesised paracetamol was recrystallised using ethanol. The purity of a dried sample was analysed using melting point analysis and thin layer chromatography.The extended structural formula of paracetamol is given below.



a) Give the molecular formula of paracetamol.
b) Explain why ethanol is a suitable solvent for the recrystallisation of paracetamol.
c) If there were still impurities present in the paracetamol, explain what effect would this have on the melting point.
d) TLC was used to confirm the purity of the recrystallised paracetamol. A sample was dissolved in a small volume of solvent and spotted onto a TLC plate. The following chromatogram was obtained.



B: Recrystallised sample of paracetamol

i) State the name of the substance spotted at A on the tlc plate.
ii) Calculate the Rf value of the substance spotted at A on the tlc plate.

e) Based on the results obtained from the tlc analysis, comment on the purity of the paracetamol.