

## Laboratory Chemistry - Separation & Purification Techniques

After working through this Factsheet you will:

- Have been introduced to a range of practical techniques;
- Be able to give a short method describing each technique;
- Understand how solid and liquid products can be isolated and purified.

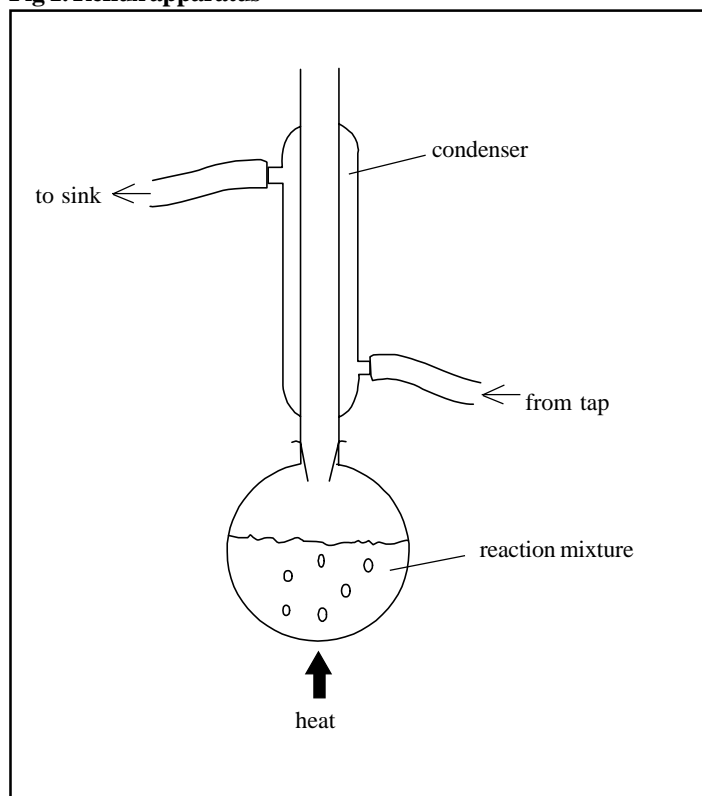
**Exam Hint:** - Candidates should have a good knowledge of these practical techniques, and be able to describe them concisely (with the aid of diagrams if necessary). They are especially important when preparing for a practical exam or practical assessment of the "planning" skill, but may also be required in the written papers.

It is impossible to generalise about methods of preparation of crude chemical products, as every compound is prepared in a slightly different manner. Candidates should be aware, however, that examiners will ask about methods of reactions covered elsewhere in the AS and A2 courses. Candidates should be able to apply their existing knowledge to practical situations, and will be given background information in the exam or assessment question.

One reaction condition worth mentioning here, however, is that of heating under **reflux**, as it is very common in organic preparations (Fig 1).

Reflux apparatus are necessary when a reactant has a low boiling point, or the reaction is slow at room temperature. The condenser prevents the escape of any volatile reagent or product.

Fig 1. Reflux apparatus



This Factsheet mainly focuses on the practical techniques required once the initial reaction has been carried out, and the crude product has been formed in the reaction mixture.

These techniques are more general, as in most cases the crude product must be:

- Isolated - from the reaction mixture.
- Purified - for use or identification.

The practical techniques outlined in this Factsheet have been placed into two groups - those associated with:

1. isolation and purification of a **solid** product
2. isolation and purification of a **liquid** product.

### 1. Isolation and Purification of a Solid Product

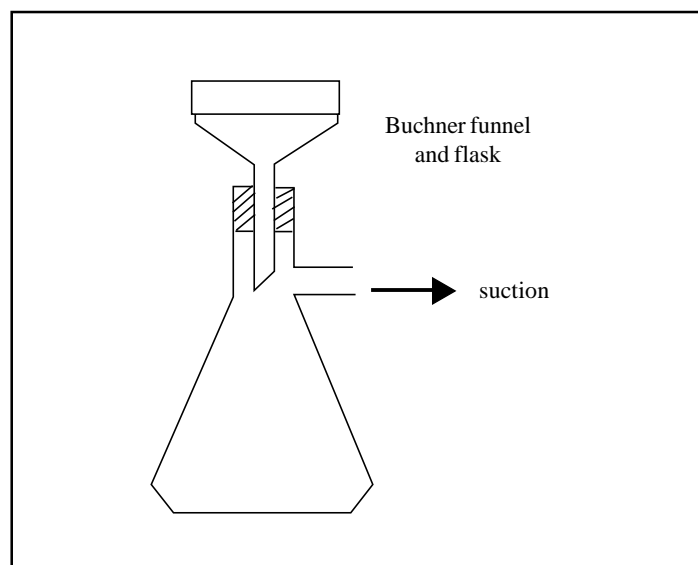
Once a solid product has been formed, the method, or plan, should commonly include three further steps:

- a) Filtration
- b) Recrystallisation
- c) Melting point determination.

#### a) Filtration

To isolate a crude solid product from a liquid, use **suction filtration**, using a Buchner funnel (Fig 2).

Fig 2. Suction filtration



A small amount of cold solvent should be used to wet the filter paper prior to filtration, and to remove any solid remains from the reaction vessel - in an effort to save as much of the solid product as possible.

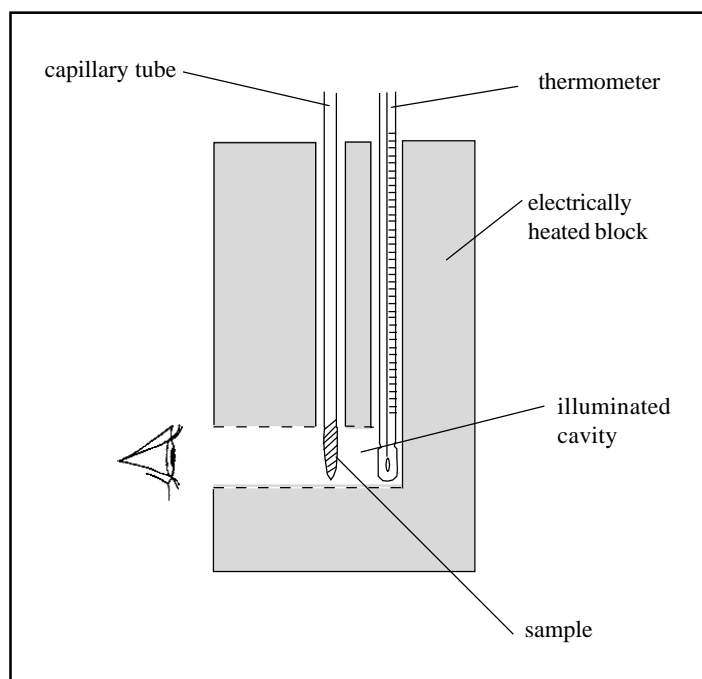
**b) Recrystallisation** - To purify the solid product

A suitable solvent for recrystallisation will often be evident (e.g. the solvent from which the crude product was initially crystallised), and should be a solvent which will not react with the solid. The solubility of the solid should be high near the boiling point and low near room temperature. Candidates should **learn** the method of recrystallisation.

- Dissolve the solid in the minimum volume of hot solvent.
- Quickly filter the hot solution using preheated filter funnel and fluted filter paper.
- Collect the filtrate and allow to cool and the solid to recrystallise.
- Use suction filtration (Buchner funnel) to collect the solid.
- Wash the solid with a small amount of cold solvent.
- Dry the solid product.

**c) Melting Point Determination**

The melting point of a solid is used to judge the purity of the product. A solid should have a sharp melting point, and recrystallisation should be repeated until this is obtained. Melting point should be determined using melting point apparatus (Fig 3).

**Fig 3. Melting point apparatus**

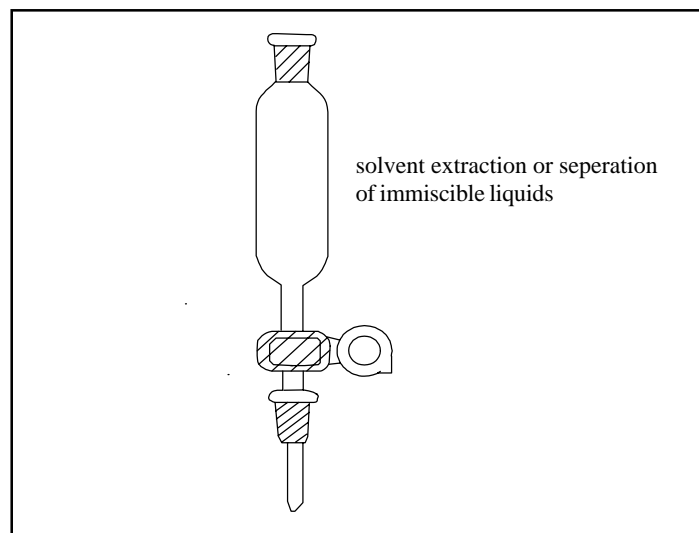
The sample is placed in a capillary tube, and the temperature increased slowly until it melts.

**Melting points can also be used for identification purposes.**

**2. Isolation and Purification of a Liquid Product**

There is slightly more to consider when planning how to extract a pure liquid product, as a liquid may be miscible or immiscible, acidic, or may need drying.

**a) Separating Funnel** - For use when separating immiscible liquids (Fig 4).



If, after carrying out a reaction, candidates are left with a liquid product which is immiscible with the rest of the reaction mixture, they must be able to find out which layer (upper or lower) contains the product. This can be discovered by examining liquid density data - obviously the more dense liquid will be the lower layer.

The two layers, which meet at an **interface**, are physically separated by running the lower layer out of the separating funnel (using the tap) and retaining the upper layer in the funnel.

**b) Neutralising Acidic Liquid Products**

Often acid conditions are required when preparing organic liquids, and the acid should be neutralised by the addition of a solid base such as sodium carbonate ( $\text{Na}_2\text{CO}_3$ ), or sodium hydrogen carbonate ( $\text{NaHCO}_3$ ) solution.

The use of an indicator is not required as it is clear when the neutralisation is complete as no further carbon dioxide gas is evolved on the addition of the base.

Care must be taken if sodium carbonate (or sodium hydrogen carbonate) is added to a liquid and a stopper placed over the vessel so it can be shaken, as the pressure of the carbon dioxide gas formed must be periodically released.

Another by-product of the neutralisation reaction will be water, so an aqueous layer may be formed. This can be removed using a separating funnel and drying agent.

**Example - Method for the preparation of Benzenecarboxylic acid.**

Candidates would be given the background information in terms of the chemical reactions involved, and should be able to calculate reasonable quantities of reactants. Focus here on steps 3, 4, 5 and 6 as they put the work covered so far in this Factsheet into a practical chemistry context.

- (1) Add 40cm<sup>3</sup> of dilute sodium hydroxide solution to 1cm<sup>3</sup> of ethylbenzenecarboxylate in a round-bottomed flask. Reflux for 30 minutes.
- (2) Transfer the mixture to a beaker and add dilute sulphuric acid until the solution is just acidic. Use a glass rod and indicator paper to do this.
- (3) Use suction filtration (Buchner funnel and flask) to filter off crystals of benzenecarboxylic acid.
- (4) Recrystallise the compound by dissolving the crystals in the minimum amount of hot water and filtering the hot solution using preheated filter funnel and fluted filter paper. Allow the filtrate to cool so that crystals form.
- (5) Use suction filtration to collect the crystals, wash with cold water and let them dry.
- (6) When dry, weigh the solid product to calculate the yield, and determine the melting point.

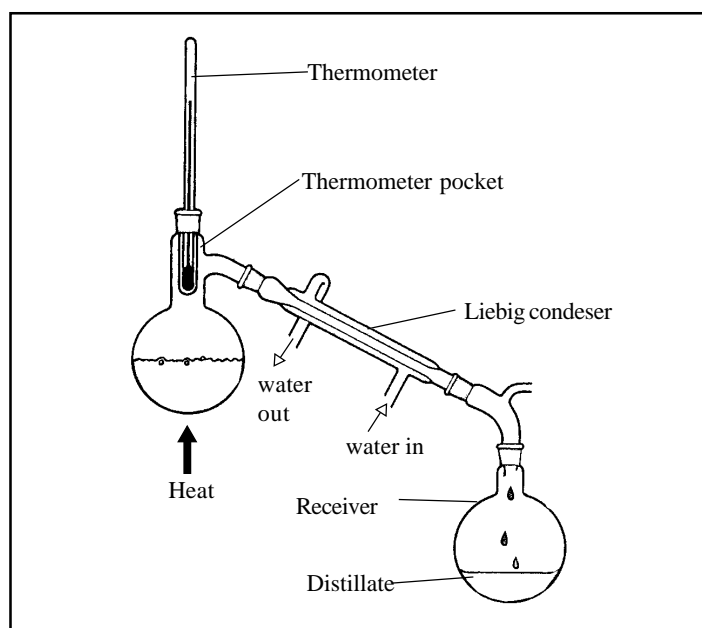
**c) Drying of Liquid Products - the Removal of Water**

The anhydrous solids calcium chloride or sodium sulphate can be added to the liquid product if it is necessary to dry it. After addition and agitation, the mixture should be allowed to stand for a few minutes and then filtered. The dry liquid can then be collected as the filtrate.

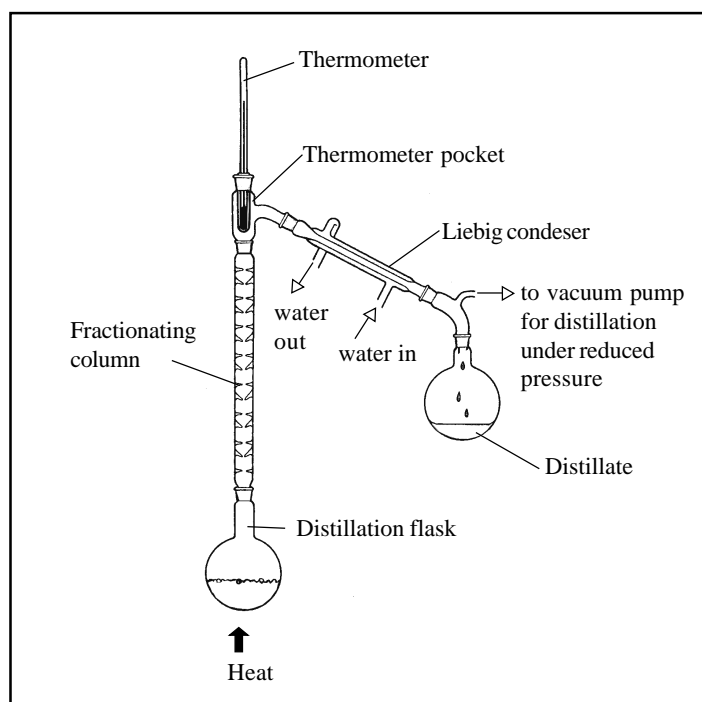
**Distillation**

Distillation can be used when the boiling point of the product is significantly different to that of the rest of the reaction mixture.

Distillation is used to purify a liquid by boiling it and then condensing it away from its impurities (Fig 5).

**Fig 5. Distillation apparatus****Fractional Distillation**

Fractional distillation is most commonly used to separate two liquids (if the boiling points are fairly close). Pure samples of each liquid can usually be obtained, unless the boiling points are too close (Fig 6).

**Fig 6. Fractional distillation apparatus**

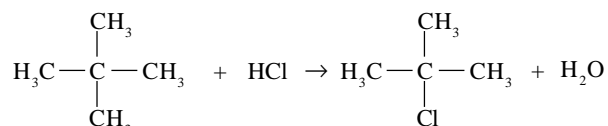
Fractional distillation will be discussed in more depth in future Factsheets.

For either type of distillation, knowledge of the boiling point of the liquid product is important.

**Preparation of a halogenoalkane**

2-chloro-2-methylpropane can be prepared from 2-methylpropan-2-ol and hydrochloric acid. The reaction will take place at room temperature as tertiary alcohols undergo substitution readily.

Boiling point of product (2-chloro-2-methylpropane) is 50 °C.



2-chloro-2-methylpropane and 2-methylpropan-2-ol are flammable, and both volatile liquids at room temperature. Concentrated hydrochloric acid is very corrosive.

Given this background information, candidates should be able to suggest a reasonable procedure plan, with safety precautions.

**Procedure**

1. Measure 10cm<sup>3</sup> 2-methylpropan-2-ol into a measuring cylinder.
2. Pour the 2-methylpropan-2-ol into a 50cm<sup>3</sup> separating funnel, and reweigh the measuring cylinder to calculate the exact mass of liquid poured.
3. Add 20cm<sup>3</sup> concentrated hydrochloric acid (excess) a few cm<sup>3</sup> at a time, taking care to mix the reactants after each addition by inverting the stoppered funnel. Ensure that any build up of gas pressure is released.
4. Leave the reaction mixture in a fume cupboard for 15 minutes, gently agitating the mixture at intervals.
5. Allow the 2 layers in the separating funnel to separate, then run off and discard the lower aqueous layer.
6. Add sodium hydrogen carbonate solution 2cm<sup>3</sup> at a time to neutralise the remaining organic layer. Shake the funnel carefully, being sure to release the build up of gas pressure. Continue the addition until the excess acid is neutralised.
7. Allow layers to separate and discard the lower aqueous layer.
8. Run the organic product into a small conical flask and add 3 spatulas of anhydrous sodium sulphate to dry it. Stopper and swirl the flask for 5 minutes.
9. Decant the liquid into a small pear-shaped flask.
10. Distil the product, collecting the fraction in the range 47 to 53 °C into a pre-weighed flask.
11. Find the mass of the purified product.

**Safety Precautions**

- Keep flammable liquids away from flames.
- Carry out acid addition in fume cupboard.
- Keep stoppers on bottles as much as possible.
- Wear gloves and safety spectacles.

**Exam Hint:** - Take note of the style of language used when writing out a method - it needs to be clear, accurate and concise. Name the apparatus, calculate quantities to be used, and use diagrams if appropriate.

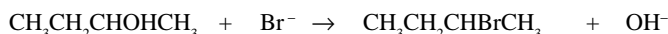
**Practice Question**

This is an example of a practical assessment task which. It is possible that candidates would be given some general advice as to what areas to revise and read about prior to the assessment, but notes and books would not be allowed whilst answering.

**Planning the preparation of 2-bromobutane**

2-bromobutane is a liquid (density =  $1.3\text{gcm}^{-3}$ ,  $M_r = 137$ ) which is immiscible with water.

It is prepared from butan-2-ol (density =  $0.8\text{gcm}^{-3}$ ,  $M_r = 74$ ), by refluxing it with solid sodium bromide, water and concentrated sulphuric acid.



You are asked to plan a safe procedure for making  $20\text{ cm}^3$  of pure 2-bromobutane, assuming a 75% yield.

Butan-2-ol dissolves in concentrated hydrochloric acid.

The plan should include:

(a) The exact volume of butan-2-ol required (show your calculations).

(b) A description of the safe procedure of the preparation.

Include:

- Diagrams of apparatus
- Which layers are discarded and kept when immiscible liquids are produced.

(c) A description of all the steps in the purification process.

(d) A description of the final distillation of the pure product. (N.B. there is no need to re-draw distillation apparatus).

(e) All safety factors should be considered.

Data:

Densities:	Butan-2-ol	= $0.8\text{ gcm}^{-3}$
	2-bromobutane	= $1.3\text{ gcm}^{-3}$
	Water	= $1.0\text{ gcm}^{-3}$
	Conc. Hydrochloric acid	= $1.2\text{ gcm}^{-3}$
	Sulphuric acid and water mix	= $1.4\text{ gcm}^{-3}$

Boiling points:	2-bromobutane	= $91\text{ }^\circ\text{C}$
	2-chlorobutane	= $78\text{ }^\circ\text{C}$

**Answer**

- (a) Conversion of  $20\text{ cm}^3$  2-bromobutane to grams. ( $20 \times 1.3 = 26\text{g}$ ) 1  
 Conversion of grams 2-bromobutane to moles. ( $26/137 = 0.190$ ) 1  
 Recognise molar ratio 1:1, deducing moles of butan-2-ol. ( $0.190$ ) 1  
 Use 75% purity to calculate number of moles of butan-2-ol required initially. ( $0.190/0.75 = 0.253$  moles) 1  
 Deduce mass of butan-2-ol required initially ( $0.253 \times 74 = 18.7\text{g}$ ) 1  
 Converts grams to volume ( $\text{cm}^3$ ) of butan-2-ol required ( $18.7/0.8 = 23.4\text{ cm}^3$ ) 1
- (b) Need for cooling / slow addition of conc. sulphuric acid. 1  
 Reflux (for at least 30 minutes). 1  
 Diagram of reflux apparatus. (-1 for each error) 3
- Distillation 1  
 Made clear aim is to distil off upper organic layer. 1  
 Diagram of distillation apparatus. (-1 for each error) 3
- (c) Remove and discard upper aqueous layer. 1  
 Wash with concentrated hydrochloric acid to remove unreacted butan-2-ol. 1  
 Collect lower layer. 1
- Wash with an appropriate quantity of sodium hydrogen carbonate solution ( $2\text{cm}^3$  at a time) to remove excess acid. 1  
 Remove stopper to ease pressure as gas is produced. 1  
 Collect lower organic layer. 1
- Dry using appropriate drying agent. 1  
 Filter or decant to remove desiccant. 1
- (d) Distil to further purify. 1  
 Collection of distillate in temperature range ( $88\text{-}94\text{ }^\circ\text{C}$ ). 1
- (e) Identification of hazards 2  
 e.g. corrosive nature of acid, exothermic reaction of concentrated acid and water, 'holding up' of vapour in reflux condenser.
- Precautions 2  
 e.g. gloves and goggles, gentle heating during reflux, no naked flame near flammable solvents, careful addition of chemicals.
- Total = 30 marks.

**Acknowledgements:** This Factsheet was researched and written by Kieron Heath. Curriculum Press, Unit 305B, The Big Peg, 120 Vyse Street, Birmingham, B18 6NF. ChemistryFactsheets may be copied free of charge by teaching staff or students, provided that their school is a registered subscriber. No part of these Factsheets may be reproduced, stored in a retrieval system, or transmitted, in any other form or by any other means, without the prior permission of the publisher. ISSN 1351-5136