Chem Factsheet

January 2003 Number 48

Laboratory Chemistry - Organic Techniques

Before working through this Factsheet you should:
- Have some practical experience in organic chemistry;
- Undersatnd the Organic Chemistry covered so far at AS and A2 (covered in Factsheets 15, 16, 17, 27, 31, 32, 33, 34, 35 and 39);
- Understand the laboratory techniques for separating and purifying (covered in Factsheet 30).

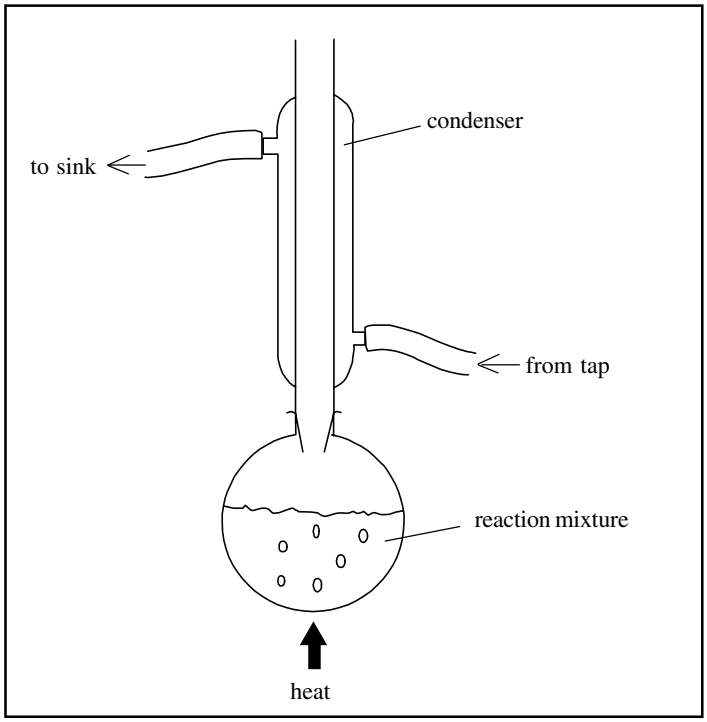
After working through this Factsheet you will:
- Have met and revised common practical techniques in organic chemistry;
- Be able to represent the relevant equipment with simple diagrams;
- Be able to assess simple organic practicals in terms of safety.

As several of these techniques were discussed in detail in Factsheet 30, this Factsheet is designed to be more of a focused revision aid.

It is necessary for candidates to display a familiarity with a variety of practical techniques in organic chemistry when preparing for both written exams and practical exams or assessment.

1. Heating under reflux

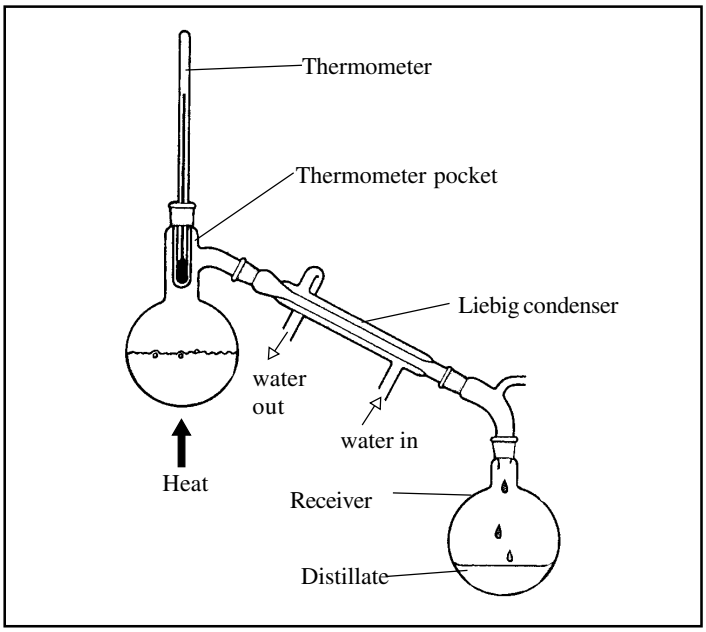
Reflux apparatus are necessary when a reactant has a low boiling point, or the reaction is slow at room temperature. The condenser prevents the escape of any of the volatile reagent or product.



2. Distillation

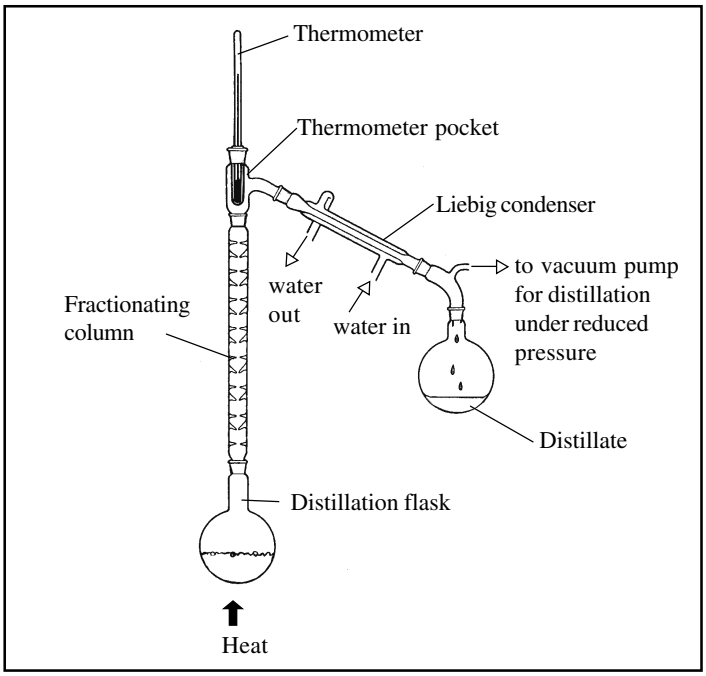
Distillation can be used to isolate a liquid product if the boiling point of the product is significantly different to that of the rest of the reaction mixture.

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



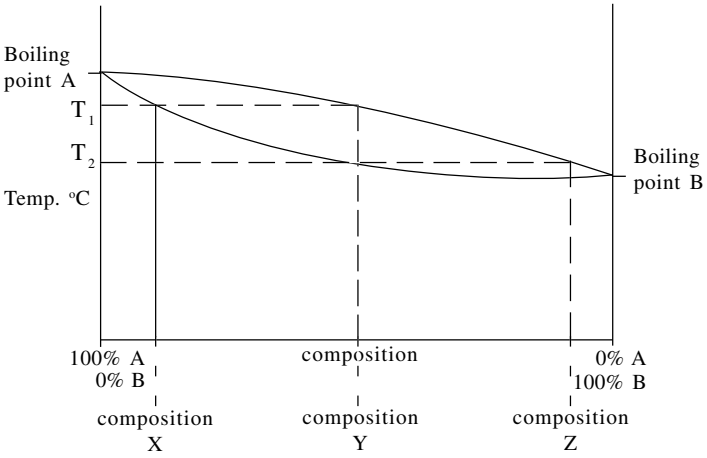
3. Fractional Distillation

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



Candidates need to be familiar with the following types of graphs plotting boiling points against composition.

Chemical A must be separated from chemical B. Chemical A has a higher boiling point than chemical B, so chemical B is a more volatile liquid.



Consider a mixture of two chemicals A and B, of composition X. If it is heated it will boil at T_1 °C to give a vapour of composition Y.

Note that Y is richer in the more volatile component than X.

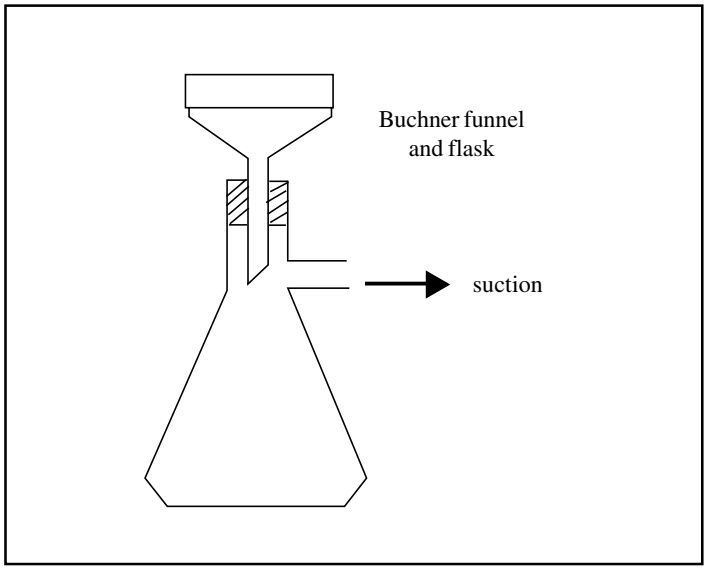
If Y is condensed and reboiled it will boil at a temperature T_2°C, giving a vapour of composition Z, where Z is richer in the more volatile component B than Y was.

Eventually pure B will be distilled off from the top of the fractionating column and pure A will be left in the flask.

How the vapours are continuously condensed and reboiled will be discussed in a later Factsheet, which will deal with the workings of fractional distillation in even more detail.

4. Filtration under reduced pressure

To isolate a solid product from a liquid, suction filtration using a Buchner (or Hirsch) Funnel is an effective method.



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 flask.

5. 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.

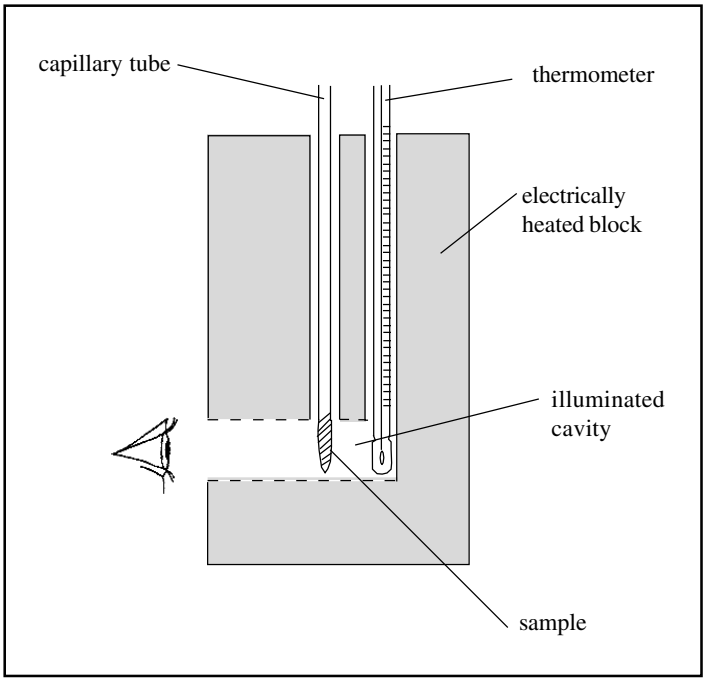
Candidates should **learn** the method of recrystallisation.

- 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.

6. 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.



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.

7. Boiling point determination

Boiling points can also be used for identification purposes, and to check the purity of a product. Boiling points are most commonly measured in the process of distillation (see section 2).

8. Selecting an appropriate heat source

There are a variety of heat sources available in the laboratory, and heat is often required for organic reactions to increase the rate of reaction or to provide the activation energy to initiate the reaction.

Two things must be considered when selecting the type of heat source:
(a) The temperature or strength of heating required.
(b) Safety.

Consider the following options:

Bunsen burner

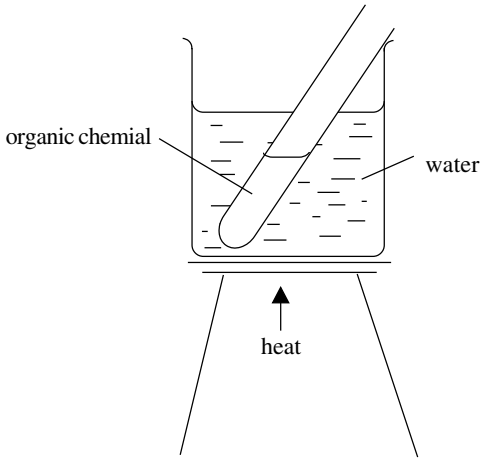
Direct, variable heat and accessible, but **danger** - do not use with flammable reagents, solvents or products.

Electric heating mantle

No naked flame (so can be used with flammable chemicals), controllable temperature, but a little cumbersome. Electricity, and necessary wiring, obviously required.

Water bath

Useful when gentle heating required, as maximum temperature is that of boiling water. Some water baths are electrically heated, whilst a simple water bath is a beaker over a Bunsen flame, so candidates should be aware if any flammable vapours may be produced.



Oil bath

Same principle as water bath, but higher temperatures can be reached. Take some time to reach required temperatures, and some time to cool down again afterwards.

Exam Hint: *- A note about safety during organic practicals. Candidates should be able to make simple risk assessments when it comes to practical work. A common type of exam question will ask for safety precautions during a specified procedure. For success in the exams and for your own personal safety, knowledge of safe procedure is important.*

*It is **assumed** that students wear eye protection during practical work, so the answer "wear goggles" will not suffice in response to a question about safety.*

Consider the following:

- *Fume hoods should be used for all reactions involving toxic, irritant or carcinogenic chemicals.*
- *No naked flames should be used with flammable chemicals.*
- *The use of gloves is required when dealing with corrosive or irritant chemicals such as concentrated acids.*

Use these ideas and common sense when answering questions about safety. When carrying out practical procedures, be careful to follow the prescribed method and pay attention to the risk assessment and safety notes.

Acknowledgements:
This Factsheet was researched and written by Kieron Heath
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