Organic Chemistry

a selection of typical applications

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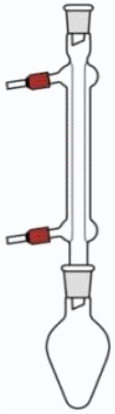
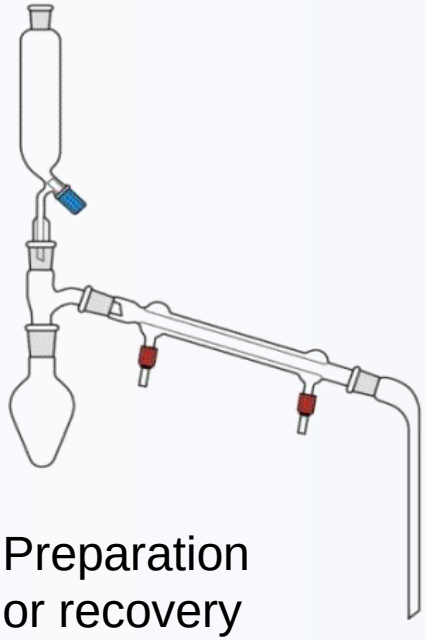
Definition

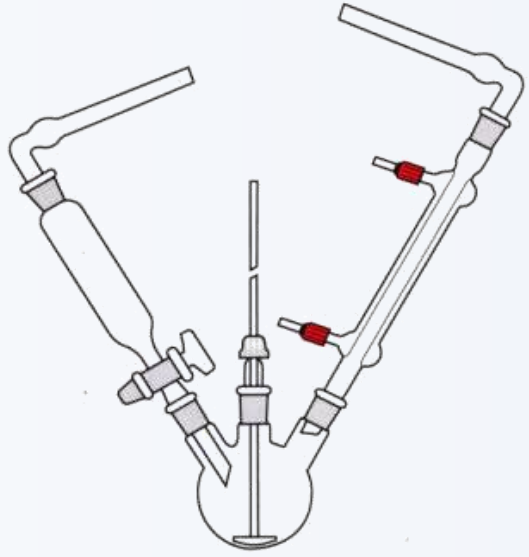
- ***Quickfit;*** *a system of individual components produced from borosilicate glass, that feature interchangeable ground glass joints which can be combined together to create assemblies for chemistry experiments.*



Reflux



Preparation
or recovery



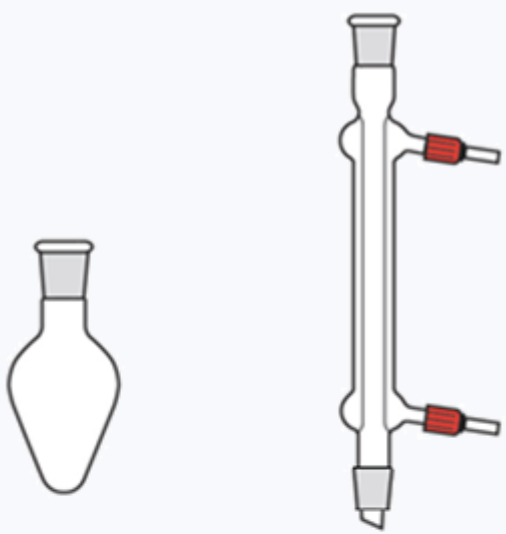
Reflux with stirring
and drying

Organic Chemistry Techniques

Reflux	**Recrystallisation**
Reflux with addition	**Weighing**
Distillation	**Filtration**
Vacuum distillation	Gravity
Fractional distillation	Büchner
Steam distillation	**Chromatography**
Distillation of a volatile product	Column
Extraction	Thin Layer Chromatography
Liquid-Liquid	**Dean-Stark apparatus**
Solid-Liquid	**Standard solutions**
Rotary Evaporation	**Titration**

Reflux

- Reflux is a distillation technique involving the condensation of vapours and the return of condensate to the system from which it originated
- It is used in industrial and laboratory distillations. It is also used in chemistry to supply energy to reactions over a long period of time
- The reaction mixture is contained in the flask. Sufficient agitation may be provided in the process of brisk boiling. It is recommended that the flask should be not more than half full
- The assembly is small enough and simple enough to be lifted by hand and agitation achieved by swirling



Pear shape Flask

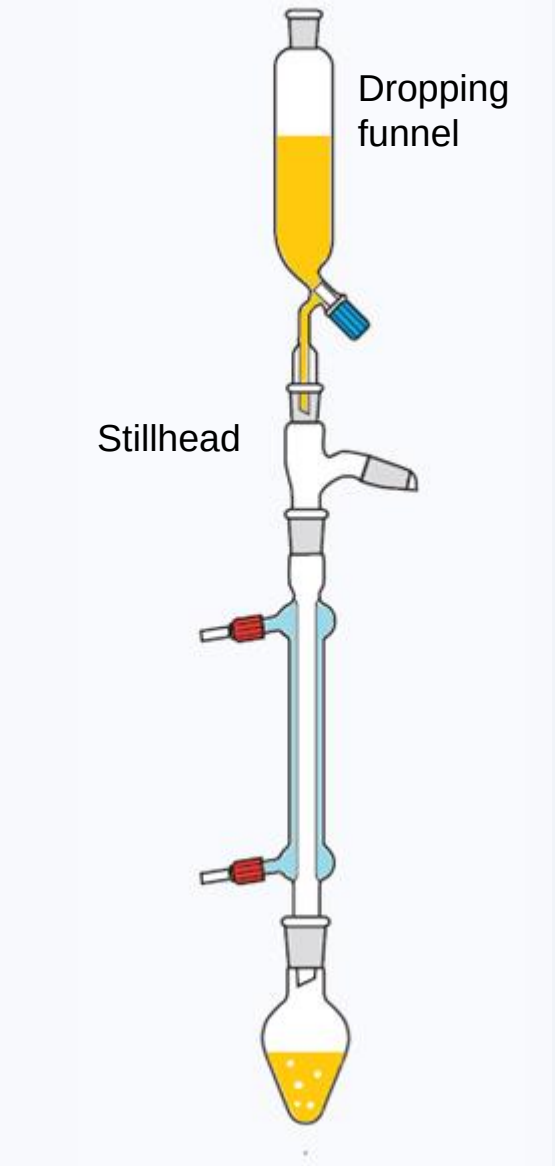
Liebig Condenser

What you need

- Flask – pear or round bottom, capacity should be approximately double the flask contents.
- Condenser – Liebig, coil or Allihn in vertical alignment
- Heat source – Bunsen burner, water bath, heating mantle or hot plate

Reflux with Addition

- A reactant is added to the refluxing reaction mixture in a controlled way via either a traditional dropping funnel and a venting piece, such as a stillhead for example or a pressure equalising dropping funnel
- This technique is used to prevent exothermic reactions from getting out of control

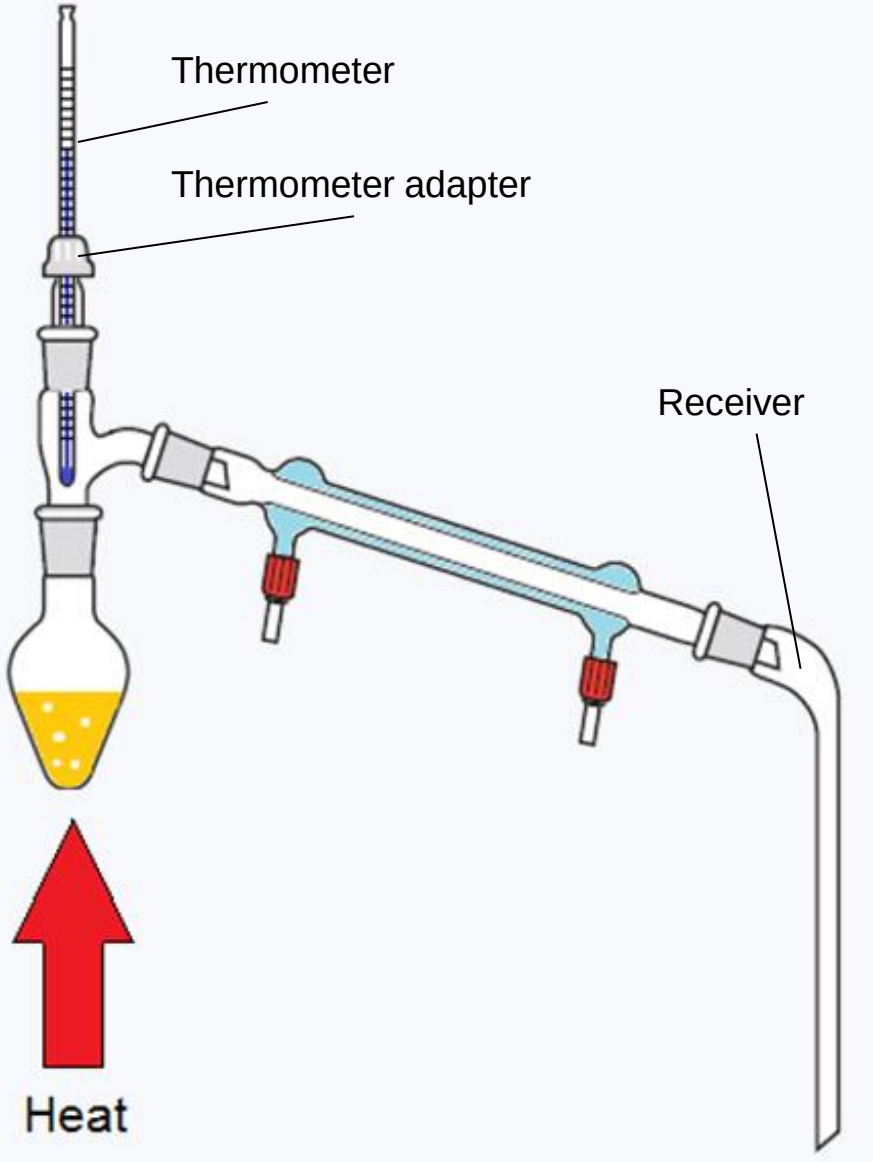


What you need

- Stillhead
- Dropping funnel

Distillation

- Distillation is the process of separating components of liquid mixtures through vaporisation and condensation, based on different volatility or boiling points of components in the mixture
- Distillation is a physical separation process, and not a chemical reaction
- This technique is used for purification
- This is a simple distillation assembly



What you need

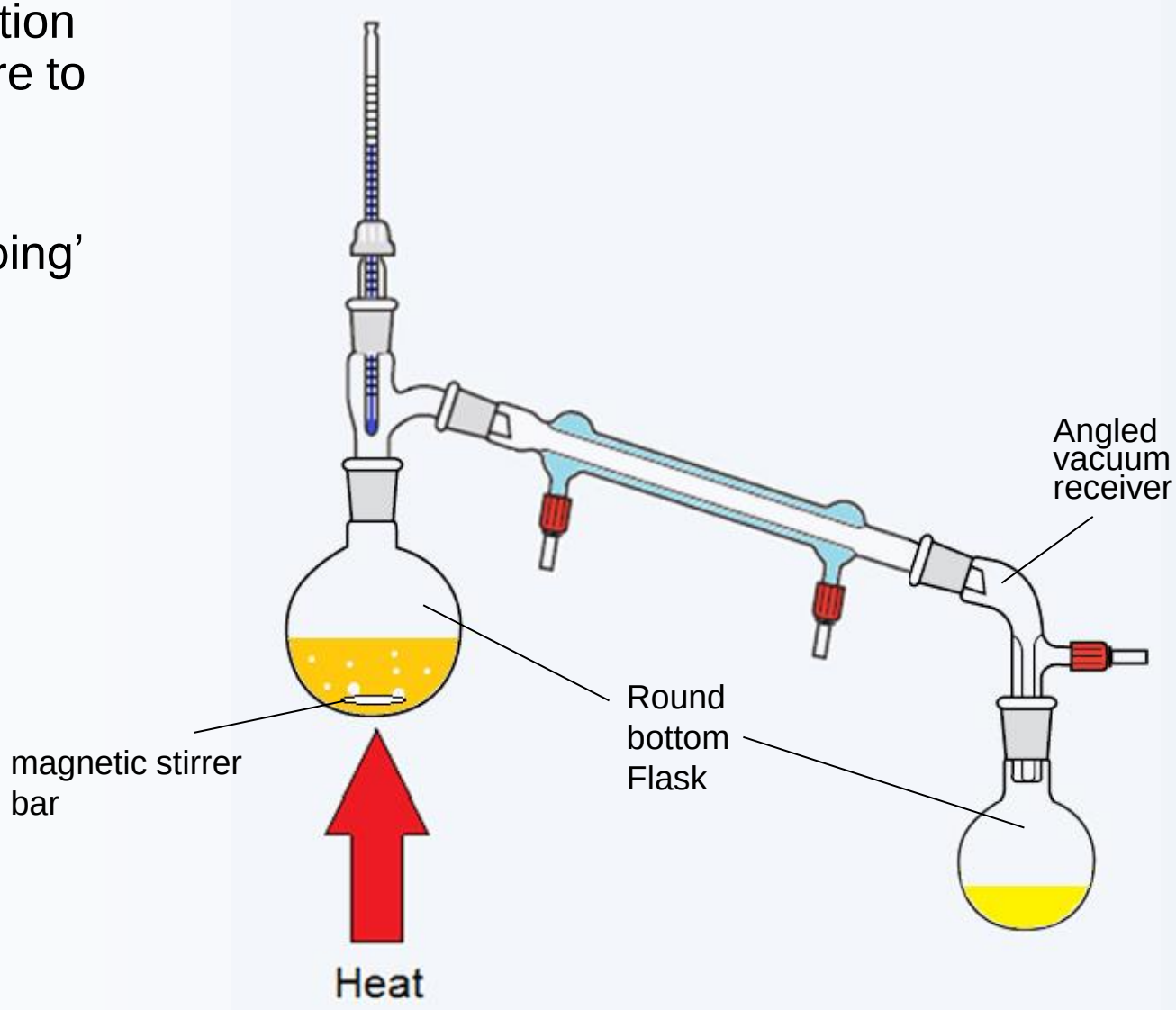
- Flask – pear or round bottom, capacity should be approximately double the flask contents.
- Condenser – Liebig or Davis in horizontal alignment
- Stillhead
- Receiver
- Thermometer and thermometer adapter
- Heat source – Bunsen burner, water bath, heating mantle or hot plate

Vacuum Distillation

- Vacuum distillation is a distillation carried out at reduced pressure to lower boiling points
- Using a magnetic stirrer bar prevents the liquid from ‘bumping’ when under vacuum

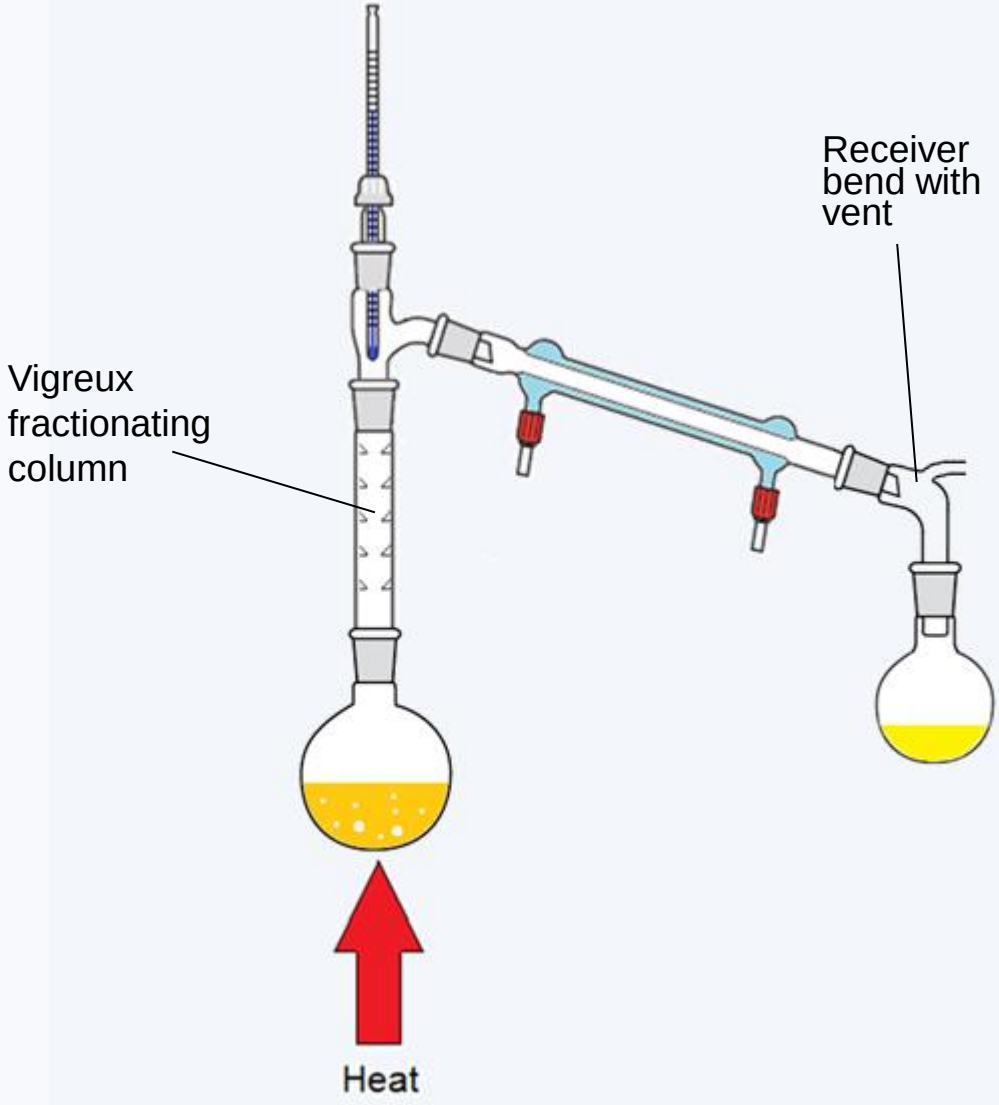
What you need

- Flask – pear or round bottom, capacity should be approximately double the flask contents.
- Condenser – Liebig
- Stillhead
- Angled vacuum receiver
- Thermometer and thermometer adapter
- Heat source – Bunsen burner, water bath, heating mantle or hot plate



Fractional Distillation

- Fractional distillation is used to separate compounds with boiling points that are close together

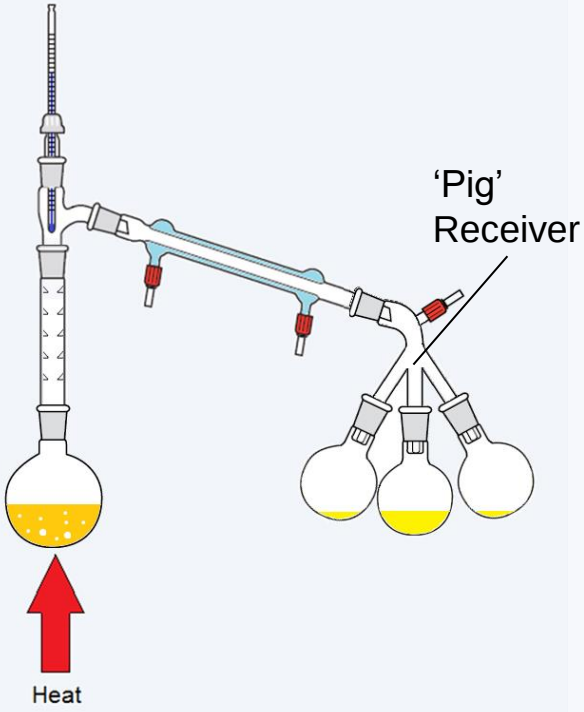


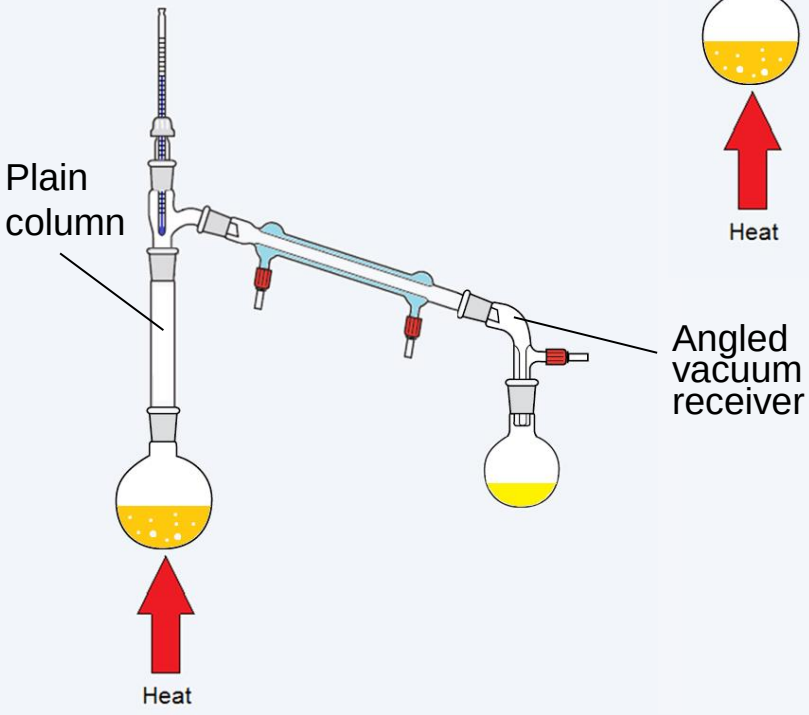
What you need

- Flasks – pear or round bottom, capacity should be approximately double the flask contents.
- Condenser – Liebig or Davis in horizontal alignment
- Stillhead
- Vigreux fractionating column
- Receiver bend with vent
- Thermometer and thermometer adapter
- Heat source – Bunsen burner, water bath, heating mantle or hot plate

Fractional Distillation

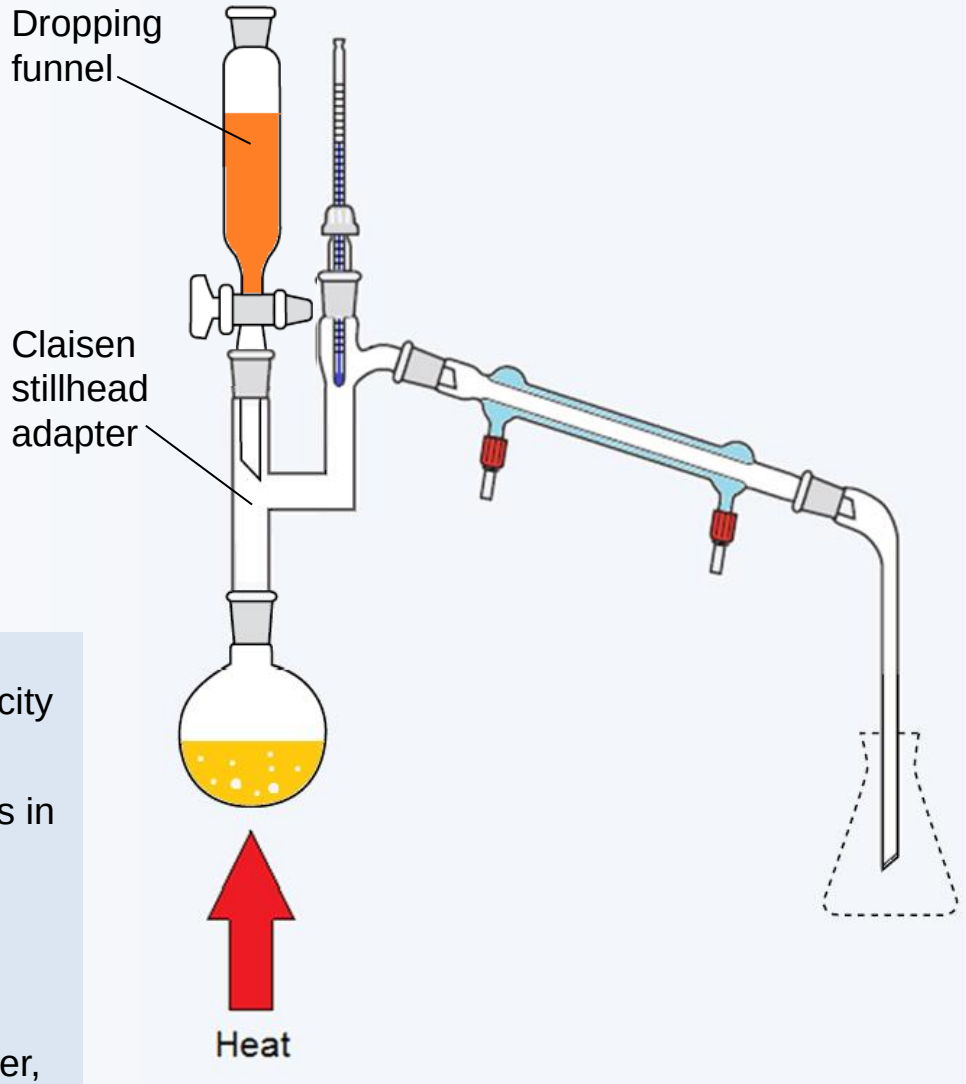
- An alternative to using a Vigreux fractionating column is to use a plain column or open column
- Angled vacuum receivers can be exchanged for a ‘Pig’ receiver or a four flask connector which are used to collect several fractions without interrupting the distillation
- The ‘Pig’ when positioned can be rotated to fill each of the flasks in turn
- An angled vacuum receiver can also be used in the same as a receiver bend with vent
- It is important never to distil from a closed system, this may cause a dangerous build up of pressure





Steam Distillation

- The extraction of a crude mixture containing water-insoluble material (such as natural products) can be achieved by the co-distillation with steam
- The dropping funnel is filled with water and added to the distillation to maintain the level of water in the distillation flask
- The distillate is collected in a conical flask and the product can be separated from the water using a separating funnel or by solvent extraction

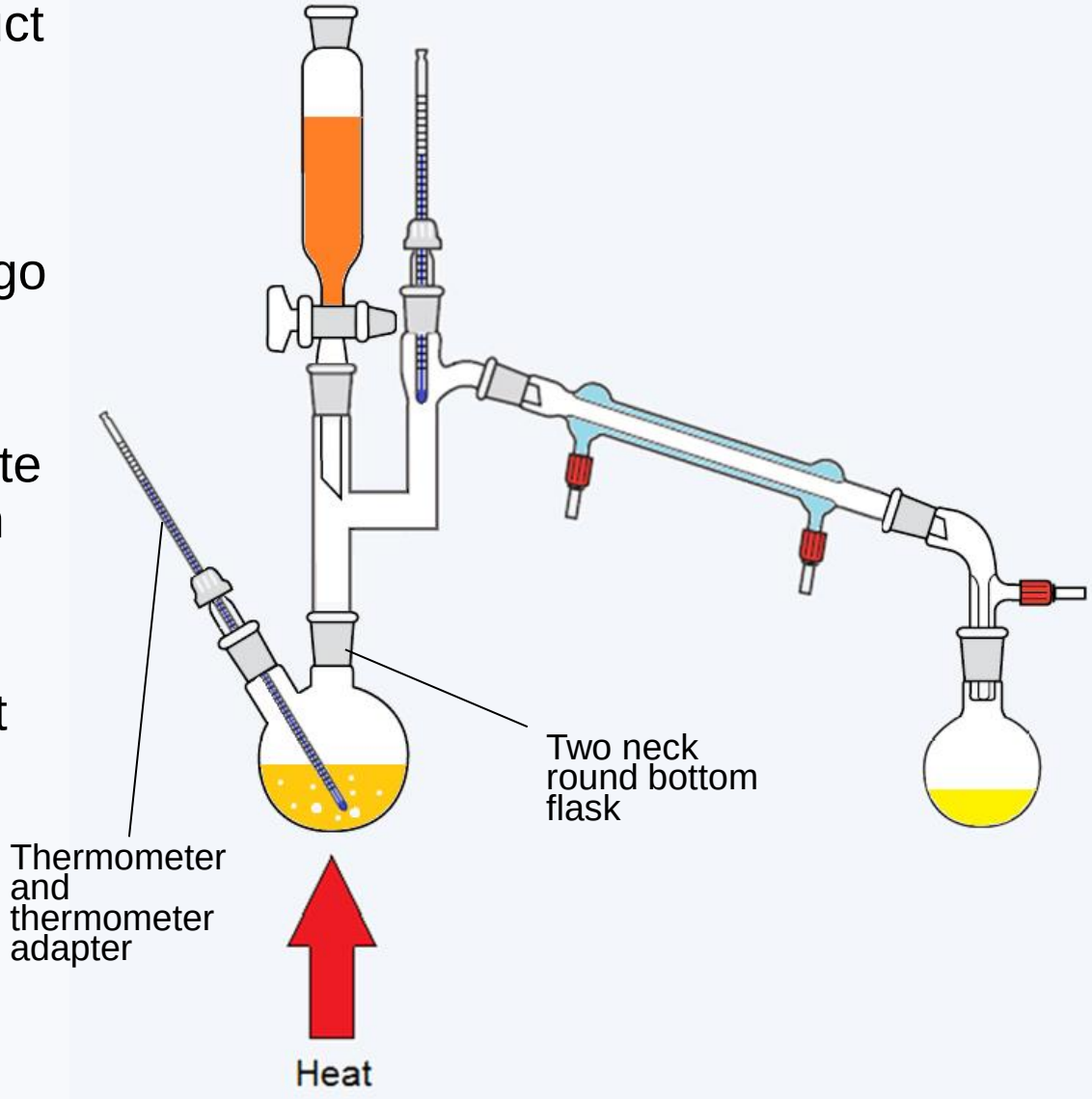


What you need

- Flask – round bottom, capacity should be approximately double the flask contents.
- Condenser – Liebig or Davis in horizontal alignment
- Claisen stillhead adapter
- Receiver
- Thermometer and thermometer adapter
- Heat source – Bunsen burner, water bath, heating mantle or hot plate

Distillation of a Volatile Product

- A technique to remove a volatile product from a reaction mixture to prevent further reaction taking place
- There are many reactions that yield products which themselves can undergo further reaction
- One such example is the oxidation of primary alcohols with acidified chromate (VI) which will yield an aldehyde which can be oxidised further to a carboxylic acid
- The aldehyde has a lower boiling point than the alcohol so it can be removed by distillation as soon as it is formed



Extraction

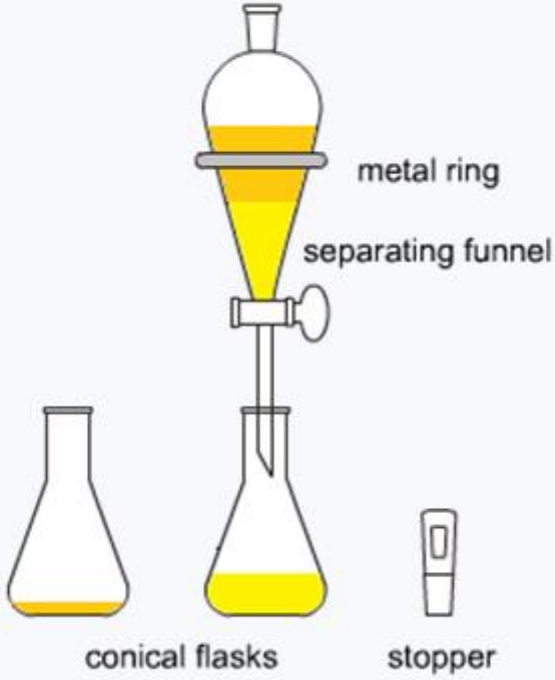
- Extraction is a separation process consisting in the separation of a substance from a matrix
- There are two types of extraction:
  - Liquid-liquid extraction
  - Solid-liquid extraction

Extraction: Liquid-Liquid

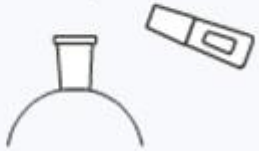
- This technique uses two solvents which are immiscible, for example an organic solvent such as dichloromethane can be used to extract an organic compound from an aqueous solution leaving water soluble impurities behind
- A variation of this is acid - base extraction where acidic or basic compounds are extracted out of organic solutions using basic or acidic aqueous solutions

When extracting, the flask is stoppered, inverted and shaken holding the flask so that the stopper is held in place. The tap is opened occasionally (with the flask inverted) to relieve any pressure build up.

The separated layers are kept in two conical flasks.



It is important to remove the stopper before allowing the contents of the funnel to be poured out.



An extracting solvent is chosen so that it is immiscible with the holding liquid and one that the compound has a greater solubility in.

What you need

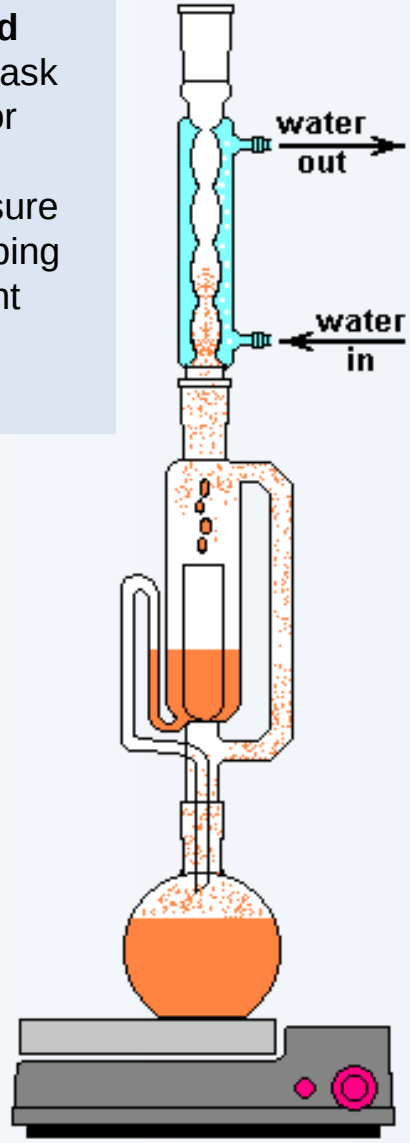
- Conical flasks
- Separating funnel
- Stopper
- Retort stand
- Metal ring to support funnel

Extraction: Solid-Liquid

- Solid-liquid – Soxhlet Extraction
  - Can be used when a compound of low solubility such as a lipid, needs to be extracted from a solid
  - The technique places a Soxhlet extractor in-between a flask and a condenser
  - Typically a porous, cellulose thimble contains the solid material within the extraction chamber
  - The flask is heated and the vapour from the refluxing solvent evaporates, condenses and then collects in the extraction chamber, repeatedly washing the solid, extracting the desired compound into the flask
  - The solvent in the flask is then evaporated and the mass of the remaining lipid is measured – a rotary evaporator can be used to remove the solvent

What you need

- Round bottom flask
- Soxhlet extractor
- Condenser
- Optional - Pressure equalising dropping funnel for solvent recovery

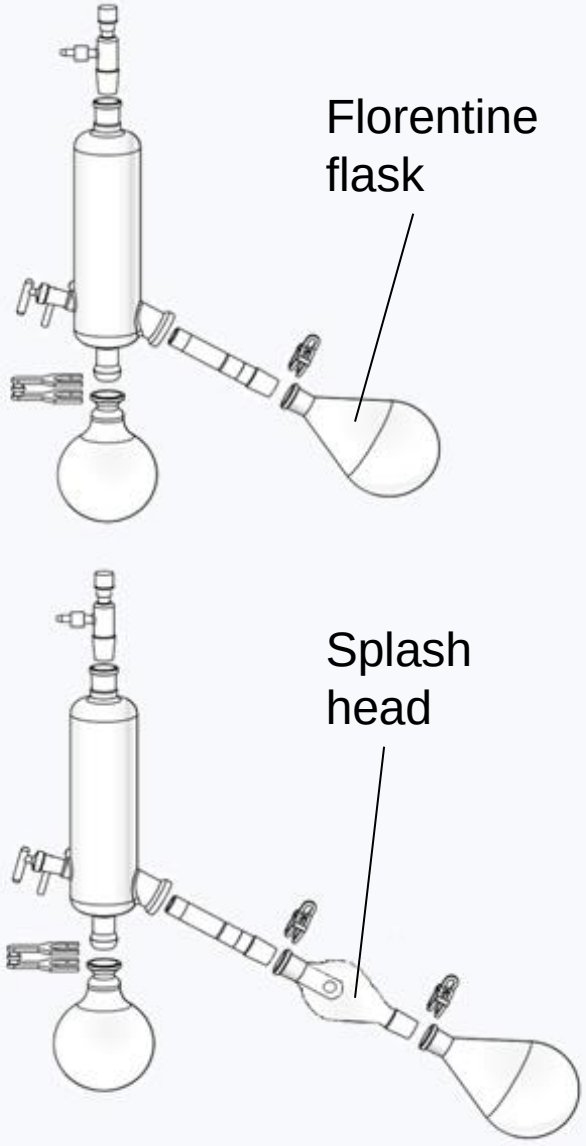


Rotary Evaporation

- A rotary evaporator is used to remove large amounts of solvent from solutions at a reduced pressure
- This is often done to isolate a product from a chromatographic separation or a solvent extraction.
- Rotary evaporators feature a thermostatically controlled water bath and a vacuum to create conditions to evaporate solvents within a system of reduced pressure
- A key disadvantage in rotary evaporations is the potential of some sample types to foam or bump, which can result in loss of a portion of the material intended to be retained. Even professionals experience periodic mishaps during evaporation, especially bumping
- Rotary evaporators can also be equipped with further special traps called splash heads, that are best suited to particular difficult sample types, including those with the tendency to foam or bump.

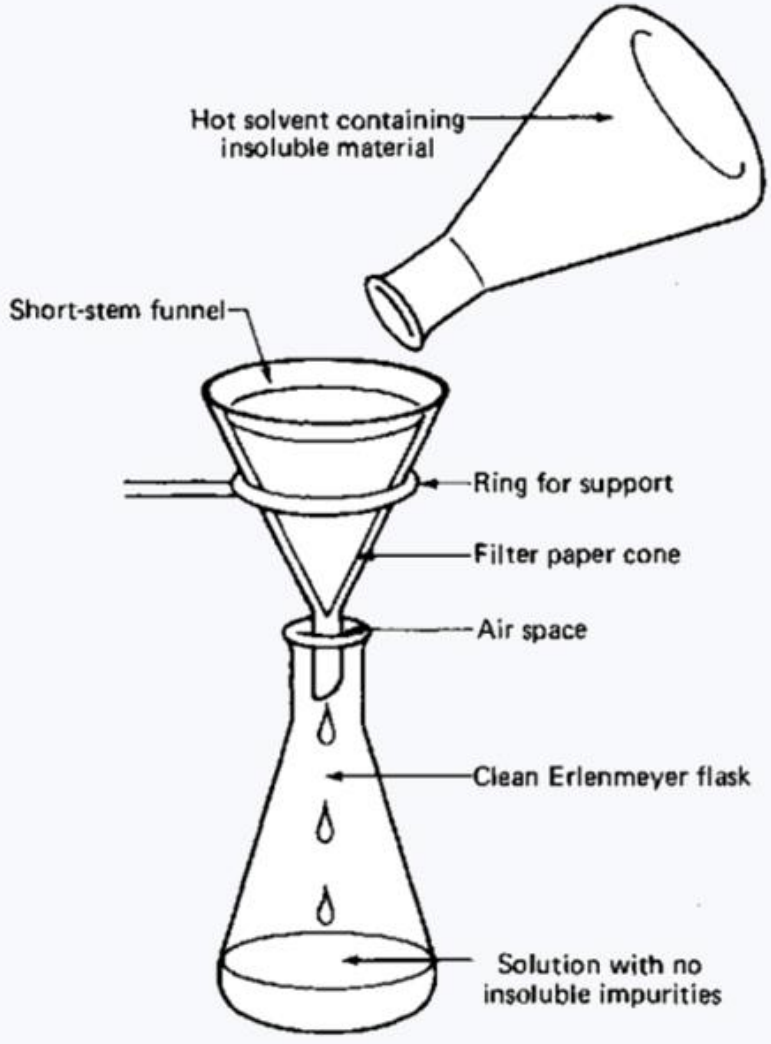
Rotary Evaporation

- SciLabware produces evaporation flasks and accessories that are compatible with a wide selection of rotary evaporator manufactures
- Evaporation flasks
  - Florentine flasks
  - Round bottom flasks
- Splash head
  - Anti-climb
  - With sinter
- Splash heads reduces entrainment of raw liquid into the vapour stream, protecting the condenser against liquid ingress from liquids that have tendency to foam or bump



Recrystallisation

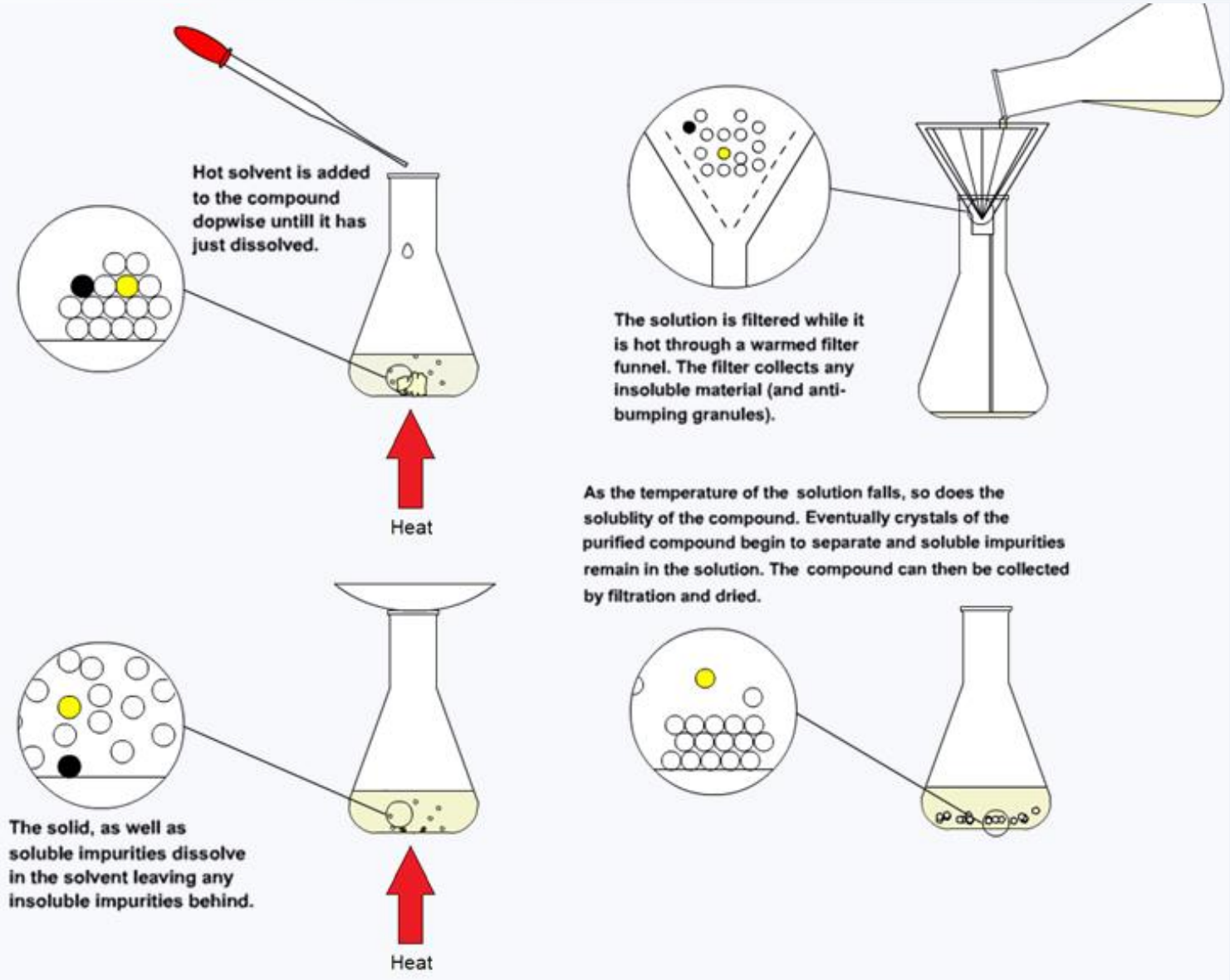
- Reactions often yield impure products which need to be purified by recrystallisation.
- The impurities present can be side products of the reaction, unreacted starting materials as well as insoluble impurities such as anti-bumping granules
- By dissolving both impurities and a compound in a minimum amount of suitable hot solvent
- Immediate filtration of the hot mixture removes any insoluble impurities
- The filtrate solution is then to cool allowing crystals of the purified product to form
- The last step is the collection of the product by filtration



Recrystallisation

What you need

- Pasteur pipette
- Watch glass
- Conical Erlenmeyer flasks
- Plain stem filter funnel
- Filter paper
- Bucher funnel
- Bucher flask

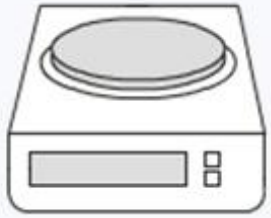


Weighing

- There are two types of laboratory balance
  - General purpose top-pan balance
  - Analytical balance
- Weighing is done to ensure the correct amount of a reactant is added to a reaction, for the preparation of standard solutions or the weighing of a product to calculate a yield
- There is also a branch of analytical chemistry called 'Gravimetric Analysis' where precipitates are accurately weighed as a means to determine concentrations

What you need

- Weighing bottles
- Weighing scoops
- Scoops
- Watch glasses
- Tweezers
- Funnels



The 'tare' button is used to bring the balance to zero once the empty weighing container is placed on the pan.

A general top-pan balance will be able to weigh to the nearest 0.01 g.

general purpose top-pan balance

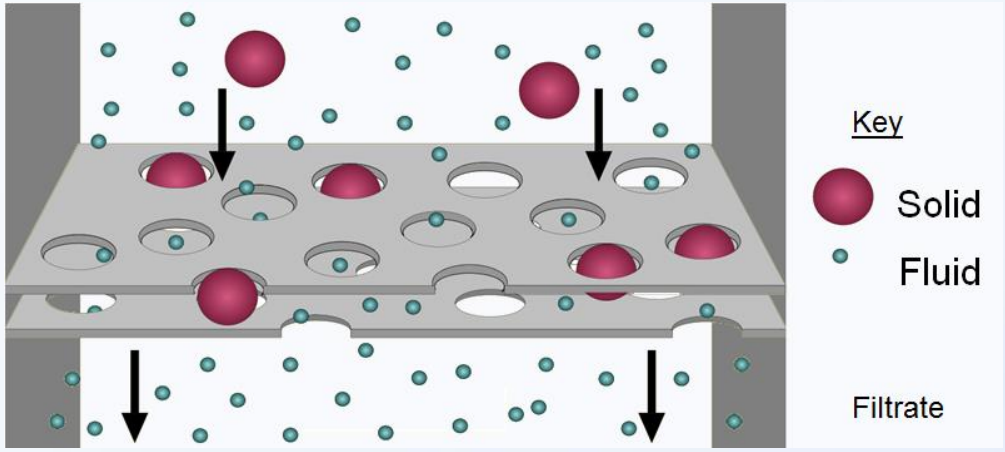
The doors of the balance must always be closed when taking measurements.

An analytical balance is designed for accurate analytical work and it should be capable of weighing to the nearest 0.0001g.

analytical balance

Filtration

- Filtration is the mechanical or physical operation used for the separation of solids from fluids by interposing a medium through which only the fluid can pass.
- The fluid that passes through is called the filtrate.
- If the solid is to be discarded it is done by gravity filtration
- If the solid is to be collected it is done under a reduced pressure using a Büchner Funnel and Büchner Flask



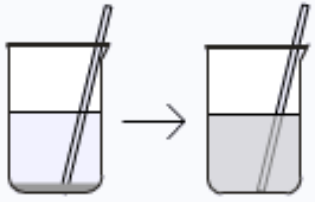
Filtration: Gravity Filtration

- This is the simplest kind of filtration when the solution to be filtered is poured through a filter paper in a filter funnel
- The filtration of hot solutions through a heated funnel and fluted filter paper is often carried out as part of a recrystallisation
- Gravity filtration is generally carried out to remove impurities rather than to isolate solids

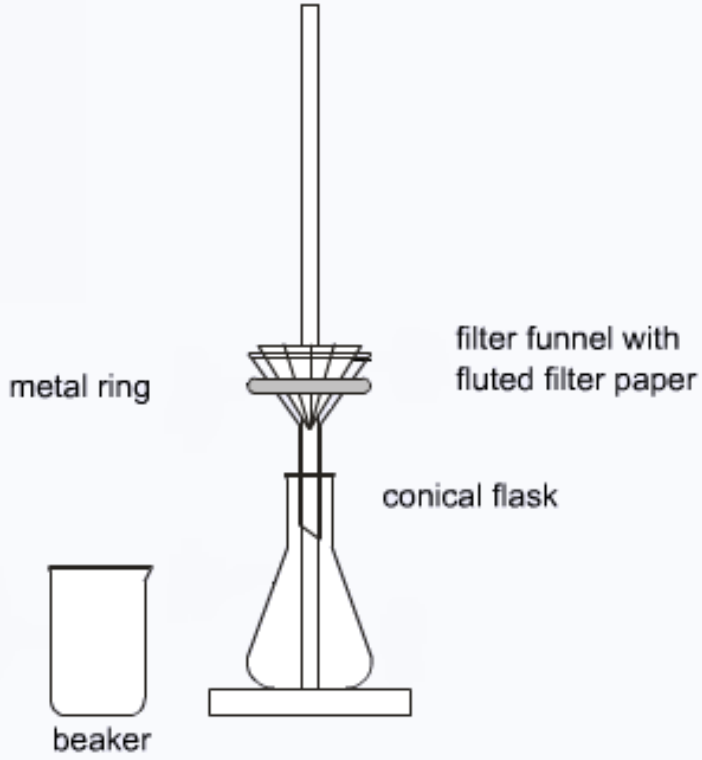
What you need

- Beakers
- Stirring rod
- Conical flask
- Filter funnel
- Filter papers
- Retort stand
- Metal ring to support funnel

If the filtration is part of a recrystallisation process it may be necessary to do a 'hot filtration' to prevent crystals forming in the funnel. To do this it is necessary to heat the funnel, the flask and the filter paper in an oven before the filtration.

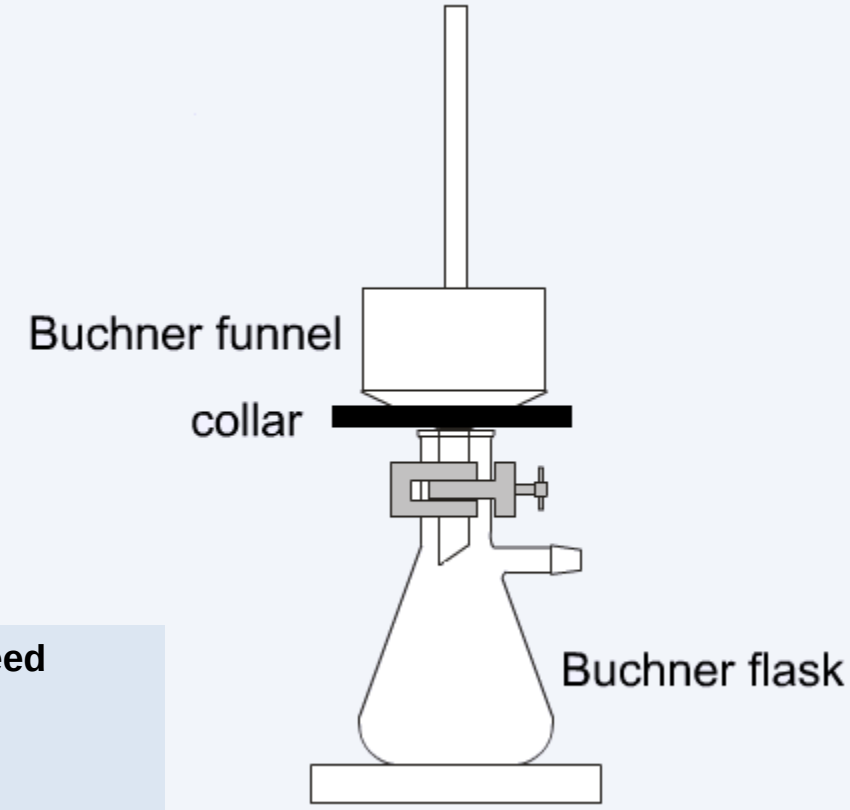


When filtering solids, it is best not to let them settle. The mixture will be much easier to pour if the solids are agitated with a stirring rod before they are transferred to the funnel.



Filtration: Büchner Filtration

- When a solid needs to be isolated from a solution it is normally done at a reduced pressure using a Büchner flask and Büchner funnel

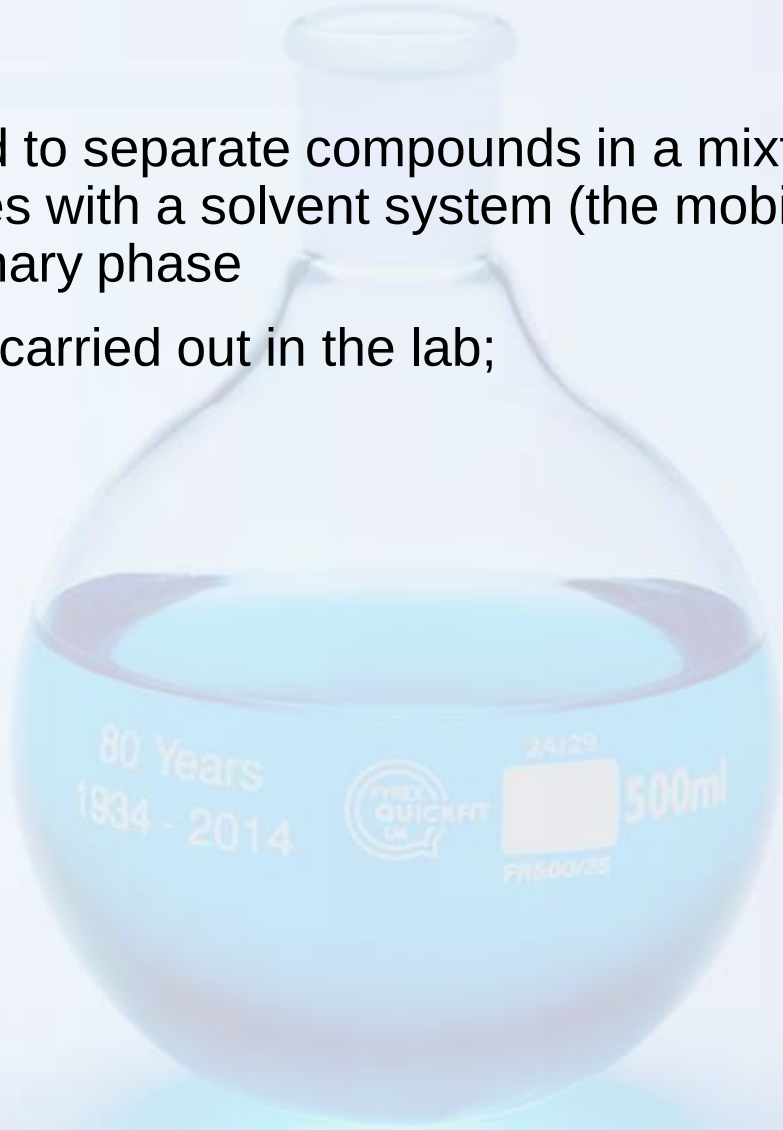


What you need

- Beakers
- Stirring rod
- Büchner flask
- Büchner funnel
- collar
- Filter papers
- Retort stand
- Metal clamp

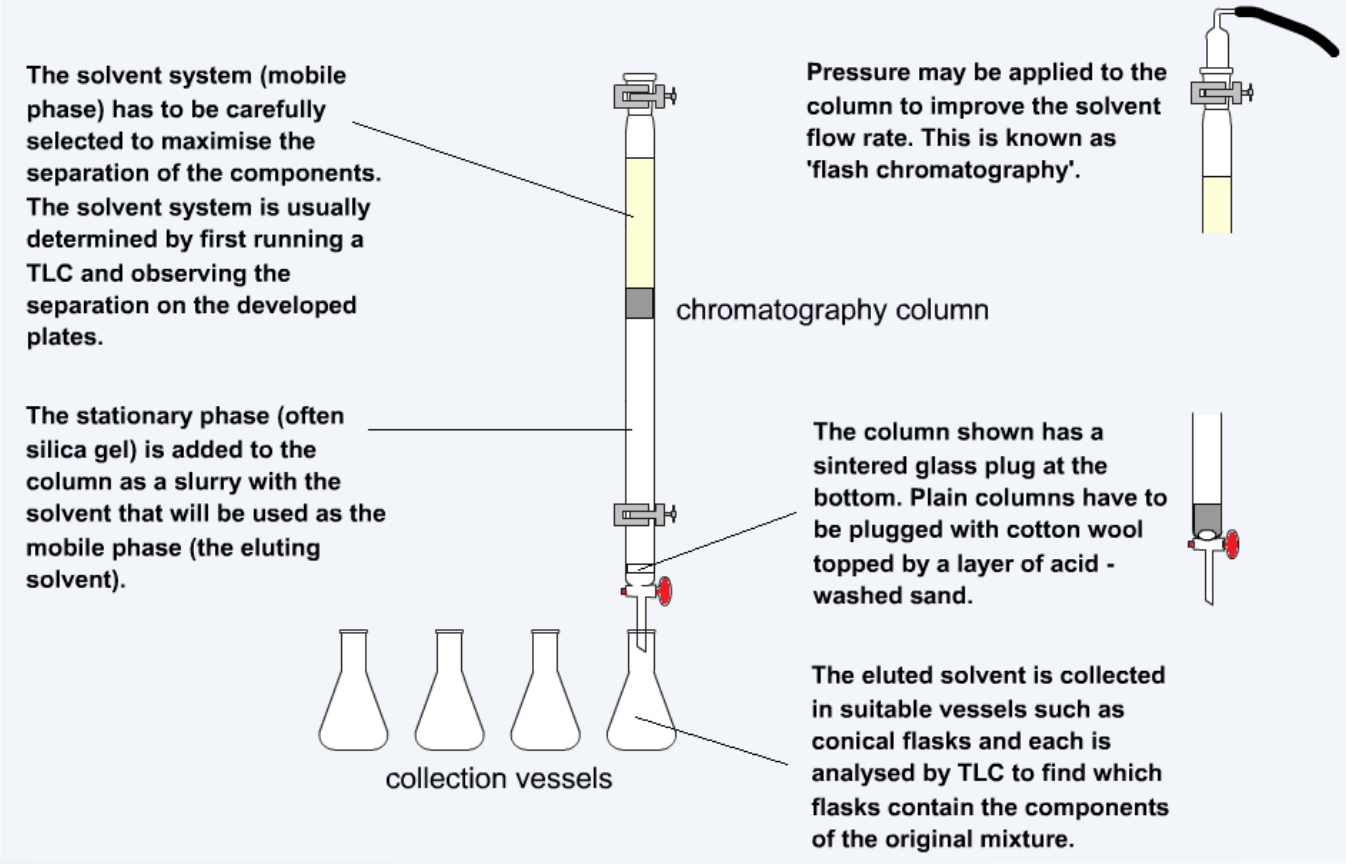
Chromatography

- Chromatography is a versatile technique used to separate compounds in a mixture. It relies on differences in interaction of molecules with a solvent system (the mobile phase) and a solid or gel known as the stationary phase
- There are two main types of chromatography carried out in the lab;
  - Column Chromatography
  - Thin Layer Chromatography (TLC)



Column Chromatography

- The separation of mixtures produced in reactions is often carried out by supporting the stationary phase in a column and allowing the solvent to move the mixtures through whilst collecting fractions of the emerging solvent. This can be carried out by allowing the solvent to flow under gravity or under a moderate pressure to increase the solvent flow rate ('Flash Chromatography')
- The fractions are usually analysed by TLC in order to identify which contain the components of the mixture

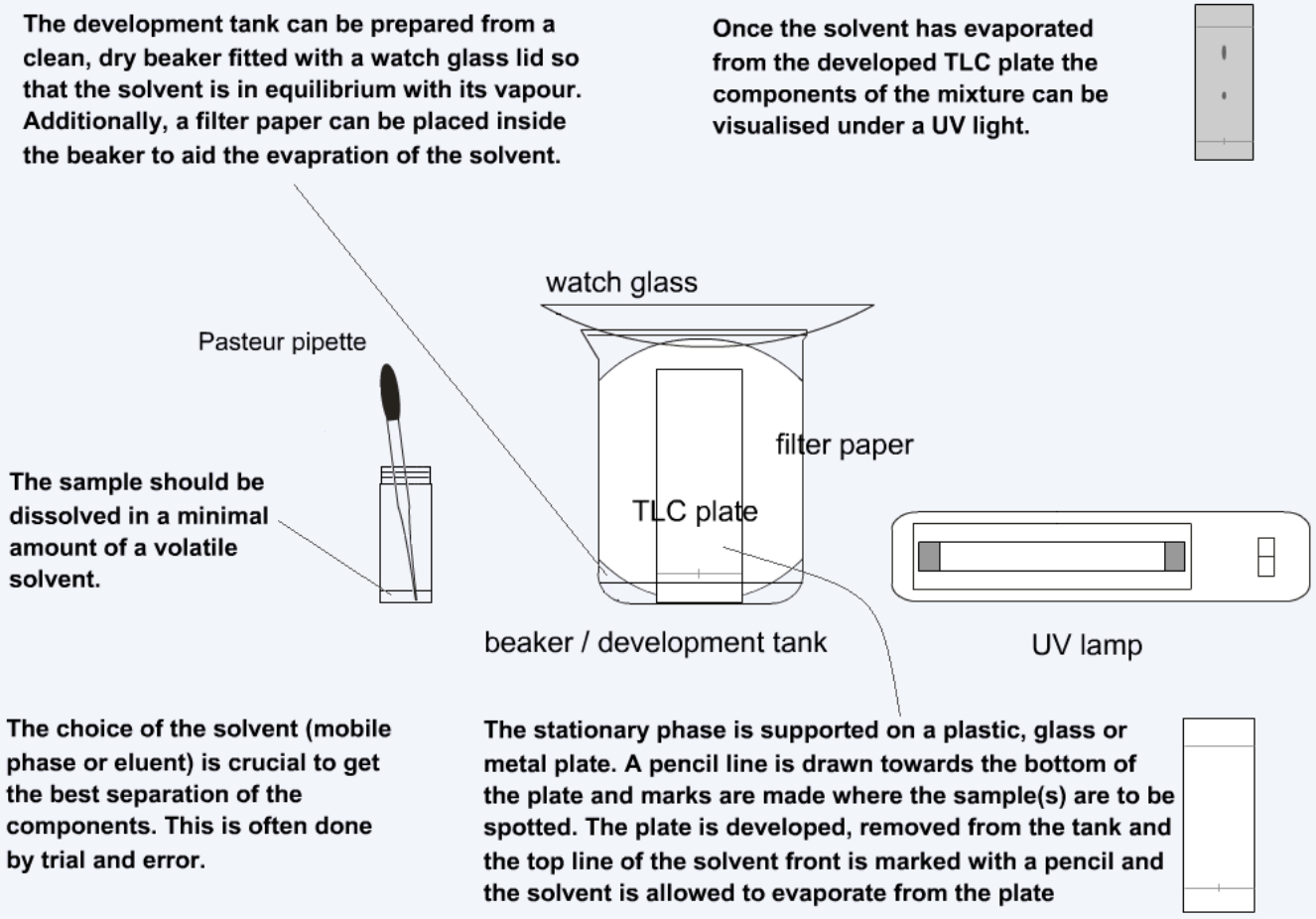


Thin Layer Chromatography

- The technique of Thin Layer Chromatography (TLC) is normally used as an analytical method to follow the progress of a reaction, to analyse mixtures or to establish conditions for a preparative separation of compounds using column chromatography
- The stationary phase (often silica) is coated on plastic or aluminium plates
- The mixture is spotted on the plate and solvent is allowed to run up the plate and separate the compounds

The development tank can be prepared from a clean, dry beaker fitted with a watch glass lid so that the solvent is in equilibrium with its vapour. Additionally, a filter paper can be placed inside the beaker to aid the evapration of the solvent.

Once the solvent has evaporated from the developed TLC plate the components of the mixture can be visualised under a UV light.



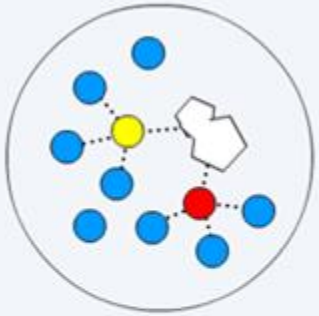
The sample should be dissolved in a minimal amount of a volatile solvent.

The choice of the solvent (mobile phase or eluent) is crucial to get the best separation of the components. This is often done by trial and error.

The stationary phase is supported on a plastic, glass or metal plate. A pencil line is drawn towards the bottom of the plate and marks are made where the sample(s) are to be spotted. The plate is developed, removed from the tank and the top line of the solvent front is marked with a pencil and the solvent is allowed to evaporate from the plate

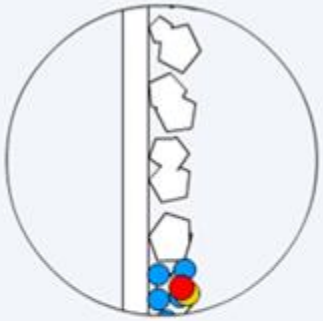
Thin Layer Chromatography

1.

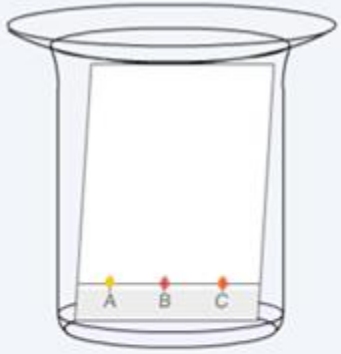


Thin Layer Chromatography (TLC) is used to analyse reaction mixtures and establish solvent systems to be used for column chromatography. Like column chromatography, TLC relies on the difference in affinities for various compounds for the stationary phase (often silica) and the solvent used as the mobile phase.

2.



TLC plate is positioned inside the development tank



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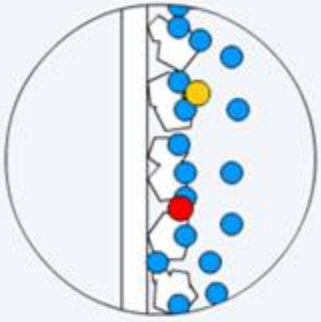
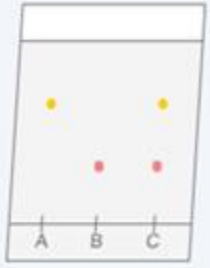


As the mobile phase (solvent) moves up the plate each component is carried at a different rate.



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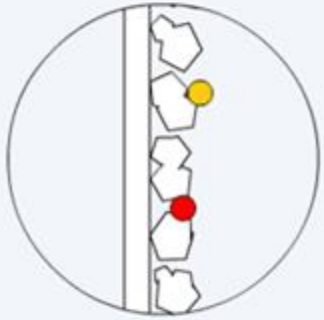
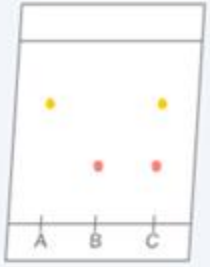


When the solvent reaches the top end of the plate, the plate is removed from the beaker and the solvent allowed to evaporate.



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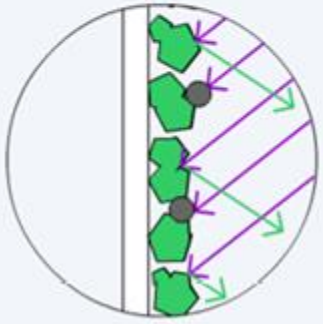
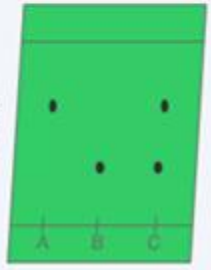
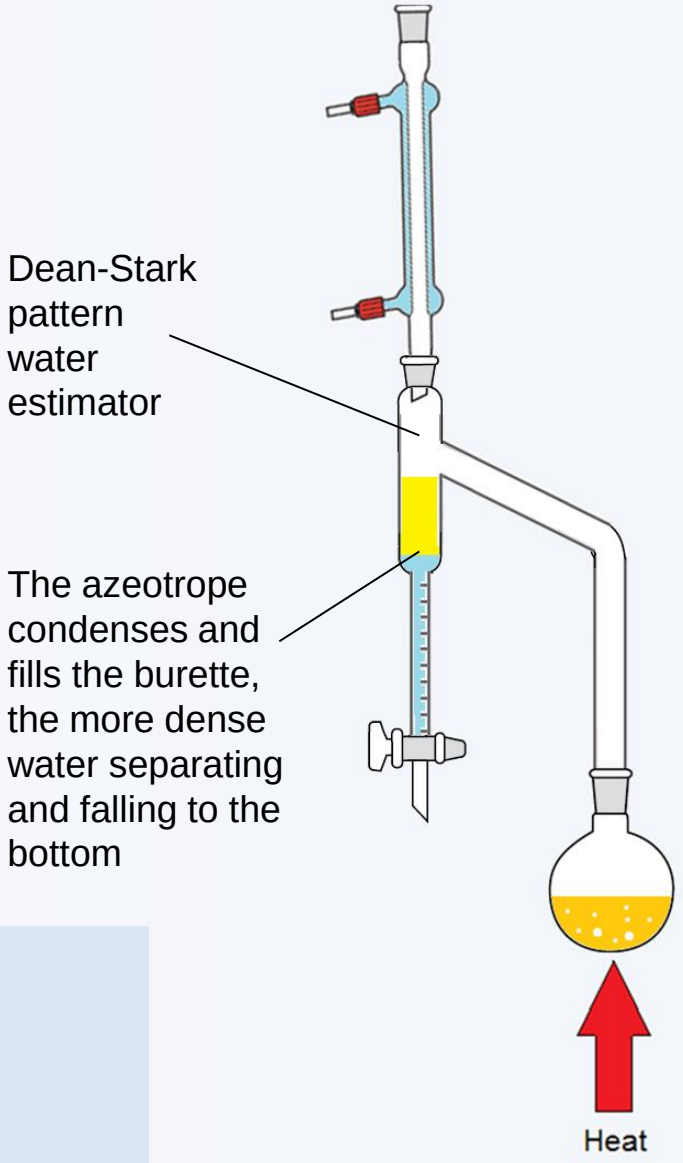


Because the plates are treated with a fluorescing agent, observing the plate under UV light shows the separated components as dark spots.



Dean-Stark Apparatus

- There are many equilibrium reactions that yield water as a co-product where removal of the water as it is produced is necessary to drive the reaction to completion and this is done using a Dean-Stark pattern water estimator.
- The reaction is carried out under reflux in a solvent which is less dense than water, both immiscible with it and forms an azeotrope.
- The apparatus allows the water to be separated from the condensed azeotrope preventing it from returning to the reaction mixture.



What you need

- Round bottom flask
- Dean-Stark pattern water estimator
- Condenser

Standard Solutions

- A standard solution is a solution of accurately known concentration prepared from a primary standard (a compound which is stable, of high purity, highly soluble in water and of a high molar mass to allow for accurate weighing) that is weighed accurately and made up to a fixed volume.

Distilled water in a wash bottle is used to make up volumes of standard solutions as well as help to transfer solutions and solids for dilution.

weighing bottle

wash bottle

beaker / stirring rod

volumetric flask

funnel

Solutions should be made up to volume by adding distilled water from a wash bottle or Pasteur pipette so that the meniscus sits on the graduation.

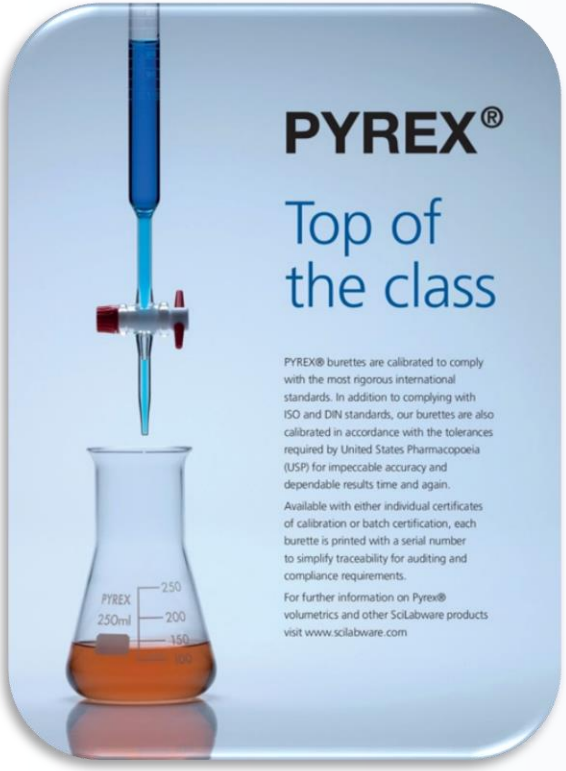
Solutions to be diluted in volumetric flasks should always be added with the aid of a funnel. Once the liquid has been transfered, the residual solution in the funnel should be washed into the flask using a wash bottle.

Solids should always be 'weighed by difference' using weighing bottles or dishes on an accurate balance.

Solids should be disolved in distilled water in a beaker prior to being transferred to a volumetric flask.

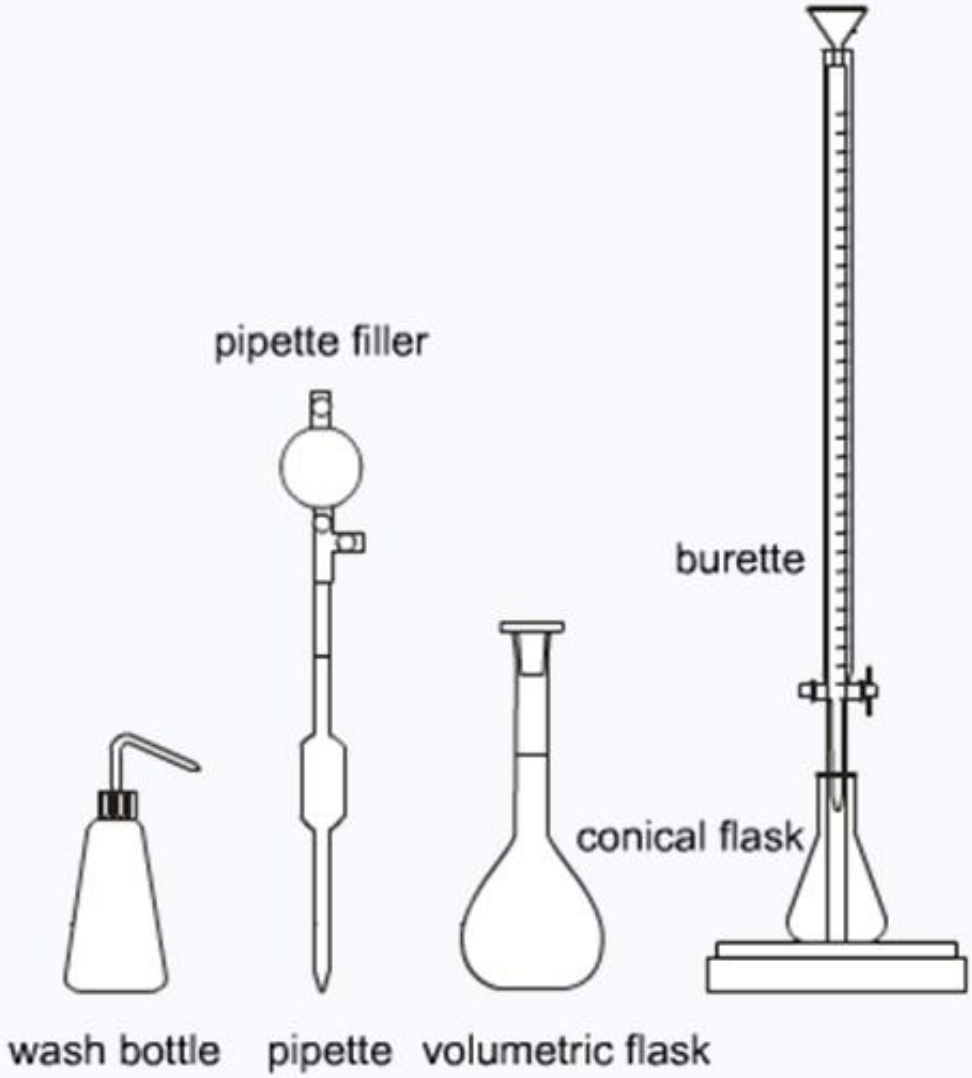
Titration

- The addition of one reagent (the titrant) from a burette to another reagent until an end-point is reached is known as a titration. These have to be done with great care and precision to establish reliable and accurate results.



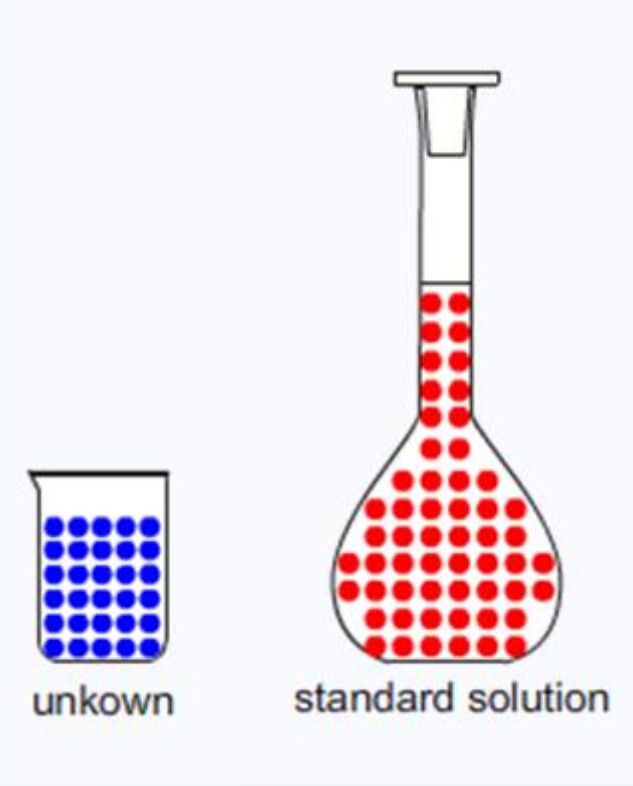
What you need

- Burette
- Pipette
- Pipette filler
- Wash bottle
- Volumetric flask
- Conical flask
- Funnel – optional



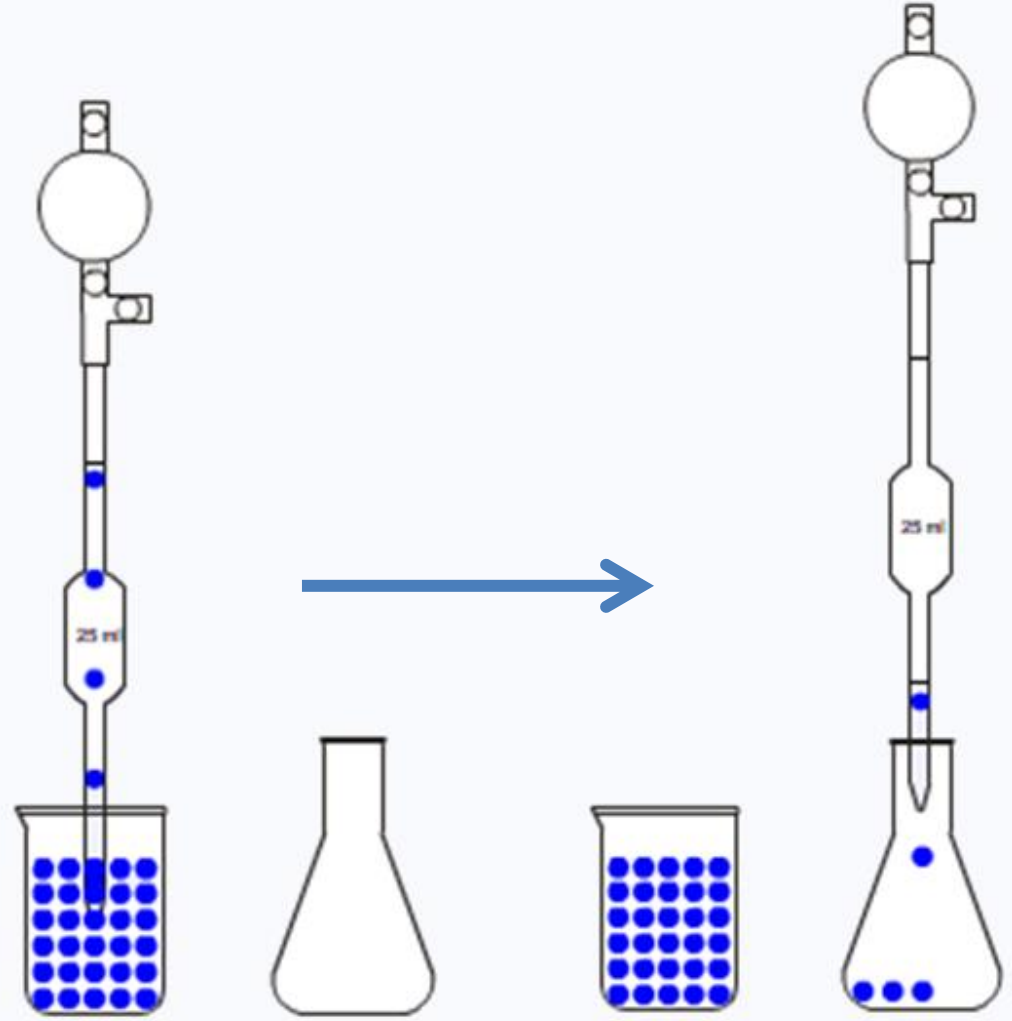
Titration: Application

- Titrations are performed to determine the concentration of a solution by reacting it with an accurately prepared standard solution.
- The most common titration is that of an acid with a base, for which an indicator is necessary.



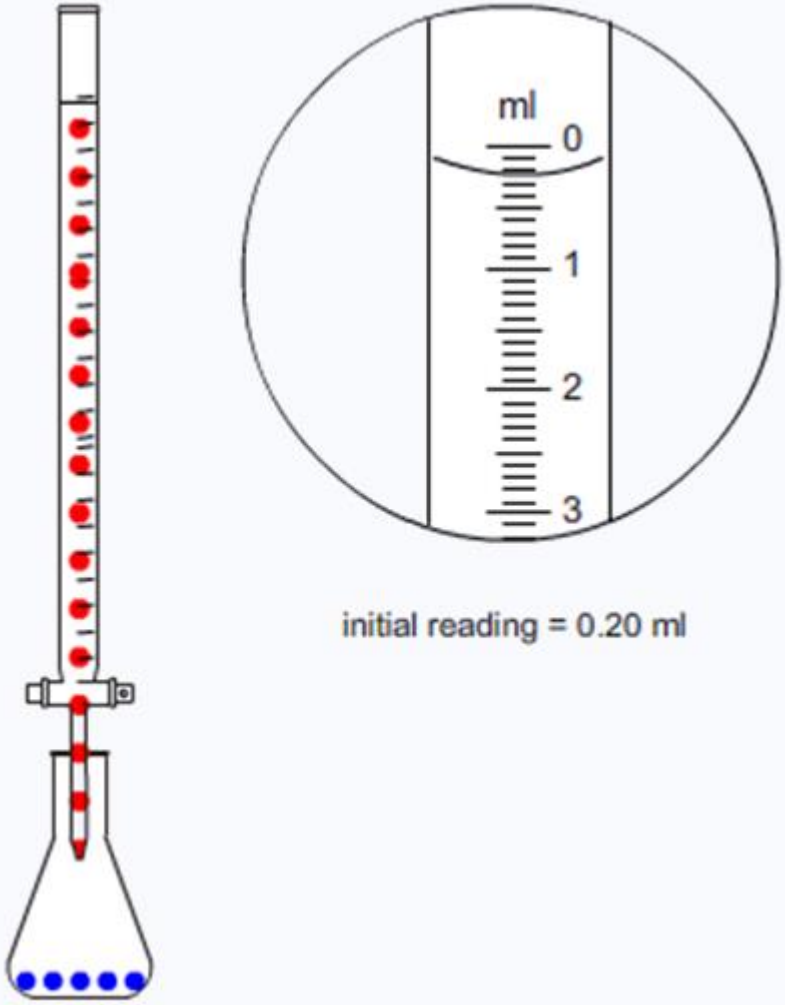
Titration: Application

- A sample of the unknown solution is transferred to a conical flask using volumetric pipette.
- This sample is known as the ‘aliquot’.
- At this stage a suitable indicator is added



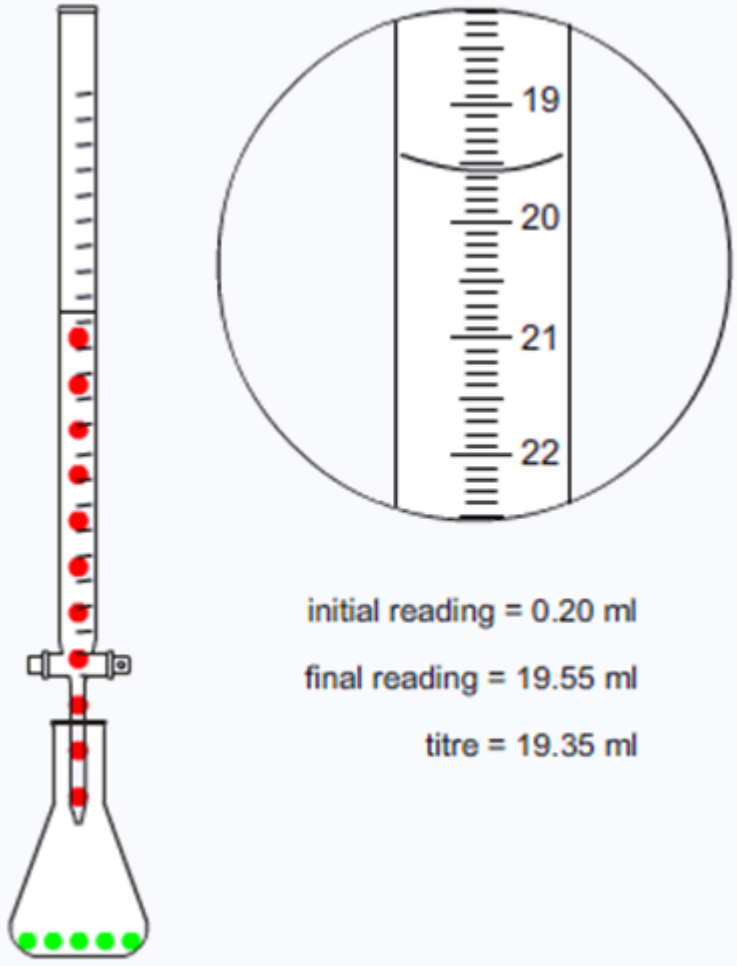
Titration: Application

- The standard solution is added to the burette, the initial reading is taken and then the titration can be carried out.



Titration: Application

- The solution with unknown concentration is titrated with the standard solution until an ‘end – point’ is reached. The final volume is recorded and the initial reading subtracted from it to give the ‘titre’.



Titration of an Acid with a Base

using phenolphthalein indicator

- Figure 1 – Start point
  - Begin the titration.
  - Phenolphthalein indicator is clear in acidic and neutral conditions becoming pink almost purple when alkaline or basic.
- Figure 2 – Slow down
  - During the titration a slight colour may be observed before disappearing, the titration should then be slowed down.
- Figure 3 – End point
  - When the indicator changes colour, this is often described as the ***end point*** of the titration, the acid and alkali are now mixed in exactly the right proportions to "neutralise" each other.
- Figure 4 – Too much base added
  - If the solution had turned pink this is an indication that too much alkali has been added.
  - Too far, start again