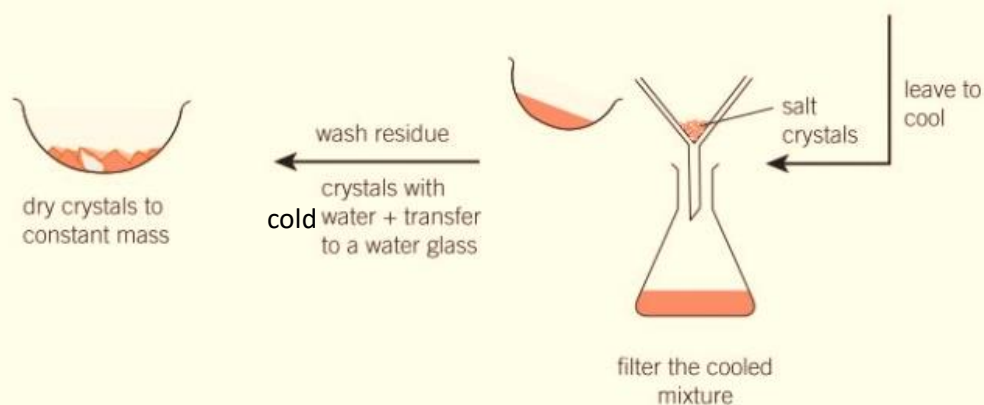
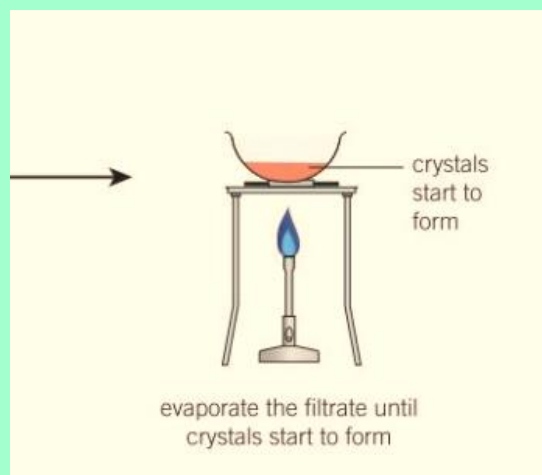
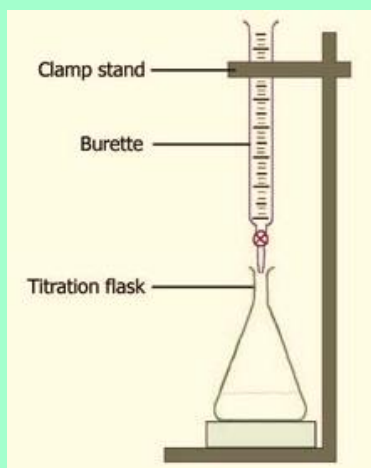


EXPERIMENTAL PROCEDURES - AS CHEMISTRY

Making water-soluble salts using an Acid + Alkali (soluble base):

Soluble salts can be made by reacting an acid and soluble bases (alkalis) or by reacting an acid with an insoluble base.

- 1) Add just enough acid to the alkalis to make a neutral solution – this can be done by periodically checking with a small sample of universal indicator paper or if both solutions are of a known concentration – use an acid-base titration:
 - a. Transfer 25.0cm³ of the alkaline solution to clean conical flask using a pipette (a safety pipette filler is used to draw solution into the pipette).
 - b. Using a burette, add the correct amount of acid to neutralise the alkali. Do not add any indicator.
- 2) Transfer the neutralised solution to a clean evaporating basin and heat over a Bunsen burner to evaporate the water. Heating should not be too vigorous as this can result in spitting.
- 3) Once crystals of solid begin to form, stop heating. Leave the mixture to cool.
- 4) Filter the mixture and wash the solid residue with cold distilled water (only a small amount).
- 5) Transfer the residue to a watch glass and heat in an oven to dry the solid. Ensure the heat is below the melting point of the solid.
- 6) At regular intervals remove the watch glass and solid, cool in a desiccator, and weigh. Once the solid has dried to a constant mass, stop heating the solid salt and leave to cool in a desiccator.

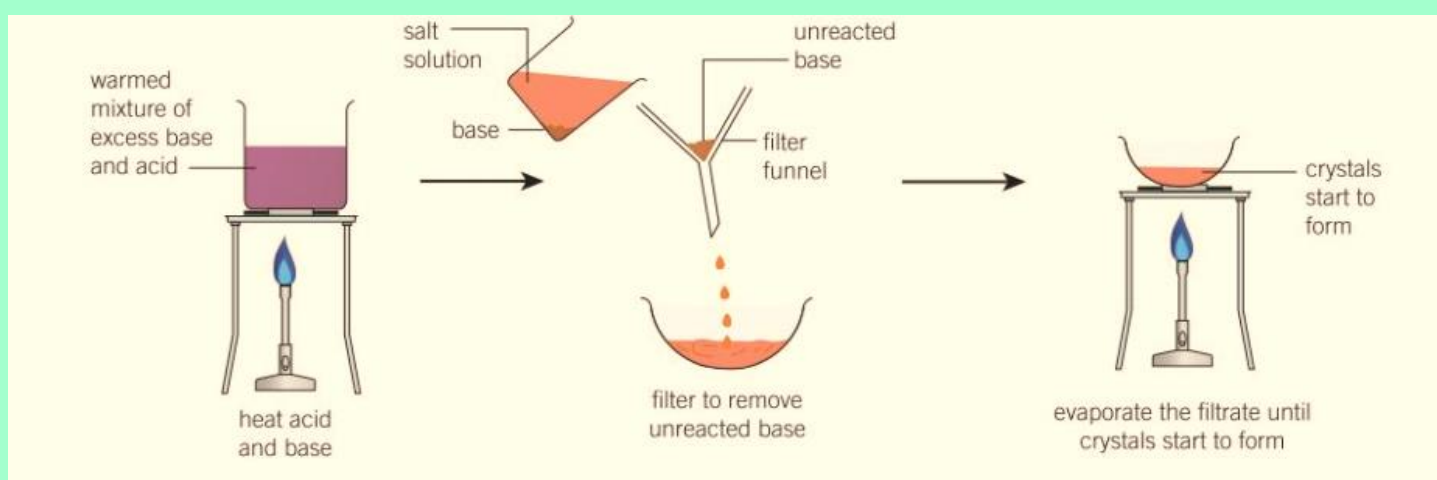


summary of the preparation of an inorganic salt by reacting an molecule base cool an acid

▲ **Figure 7** Summary of the preparation of an inorganic salt by reaction of a soluble base with an acid

Making water-soluble inorganic salts using an Acid + Insoluble base:

- 1) In a beaker, warm excess insoluble base in dilute acid.
- 2) Continue to warm (but not boil) until the solution is neutral, universal indicator paper is used to check this. Add more solid base if needed.
- 3) Filter off the excess base and transfer the filtrate to a clean, dry evaporating basin.
- 4) Heat the evaporating basin until salt crystals begin to appear on the sides of the basin.
- 5) Cool the basin and contents
- 6) Filter the mixture, and discard the filtrate solution.
- 7) Wash the residue with cold distilled water.
- 8) Transfer the residue to a watch glass and heat in an oven to dry the solid. Ensure the heat is below the melting point of the solid.
- 9) At regular intervals remove the watch glass and solid, cool in a desiccator, and weigh. Once the solid has dried to a constant mass, stop heating the solid salt and leave to cool in a desiccator.



▲ **Figure 7** Summary of the preparation of an inorganic salt by reaction of an insoluble base with an acid

Making water-insoluble salts using inorganic salt solutions:

Reacting two soluble salt solutions will form another soluble salt and an insoluble salt (precipitate).

1. Add equal volumes of the desired salt solutions in a beaker and stir to form a precipitate of the insoluble salt.
2. Filter the precipitate, discarding the filtrate.
3. Wash the precipitate several times with cold distilled water.
4. Transfer the residue to a clean watch glass and place in a drying oven to dry out. Ensure the oven is set to a temperature below the melting point of the salt.
5. At regular intervals remove the watch glass and solid, cool in a desiccator and weigh. Once the solid has dried to a constant mass, stop heating and leave to cool in the desiccator.

Acid-base titration (alkalis of known concentration) :

1. Rinse a burette with distilled water and then the acid solution to be held within it. Fill the burette with an acid solution of known concentration, running a little through the burette into a waste beaker to fill the tip.
2. Record the initial reading to the nearest 0.05cm^3
3. Fill a clean pipette
4. Transfer 25.0cm^3 of the alkaline solution to clean 250cm^3 conical flask using a 25.0cm^3 pipette (a safety pipette filler is used to draw solution into the pipette). The pipette should be rinsed with distilled water and a sample of the alkaline solution prior to this.
5. Add 2 to 3 drops of suitable indicator to the conical flask and swirl gently.
6. Run the acid from the burette into the flask. Swirl the flask continually and watch for the first hint of the solution changing colour. This titration should be done as a rough trial run to give an indication of the amount of acid required.
7. Record the final reading on the burette and calculate the titre (final – initial = vol. acid). Discard this solution and rinse the conical flask with distilled water.
8. Refill the burette and record the initial reading.
9. Repeat steps 4 to 7 until there are three concordant results (within $\pm 0.10\text{cm}^3$).
10. Calculate the average titre of acid to be used in calculations.

For finding the concentration of **an unknown alkali** (an acid of known concentration). The same steps are involved, only the acid is placed in the flask and the alkalis in the burette.

What indicator is suitable?

Phenolphthalein Changes colour sharply. It is colourless in acidic solution and pink in alkaline solution.

Used for weak acid, strong base

Methyl orange is red/orange in acidic solution and yellow in alkaline solution.

Used for strong acid, weak base

Both are suitable for titrating strong acid with a strong base. However, both indicators are poor at indicating an endpoint when a weak acid is titrated with a weak base. An indicator should never be used when doing such titrations.

Titration	Phenolphthalein	Methyl orange
strong acid + weak base	Yes	Yes
weak acid + strong base	Yes	Yes
strong acid + strong base	Yes	Yes

- ❖ NaOH/KOH are strong bases but NaHCO₃ is a weak base
- ❖ HCL / H₂SO₄ / H₃PO₄ are strong acids a strong acid but HSO₃ is a weak acid

The enthalpy change for neutralisation:

1. Use a measuring cylinder to add a known volume and known concentration of acid to an insulated vessel such as a polystyrene cup.
2. Record the initial temperature of the acid using a thermometer.
3. Use another measuring cylinder to add a known volume and known concentration of alkalis to the vessel.
4. Top the vessel with a lid with a hole in it for the thermometer.
5. Record the changes in temperature - while continuously stirring the mixture - every 30 seconds until there are no further changes in temperature.
6. Calculate the maximum increase in temperature (Max temp. – Initial temp.)

Alternatively, a solid basic compound could have been added to the acid solution. In this case, it is added in excess.

We usually assume the density of the solution is 1g/cm³ in exams.

Therefore:

Volume of the total solution (acid + alkali) = mass of solution

If a solid base is used then we ignore the mass of the solid added and instead use the volume of the initial solution (the acid) = mass of solution

e.g. If 2g of CaCO₃ are added to 25cm³ of acid, the mass used in calculation is 25g not (25+2)g

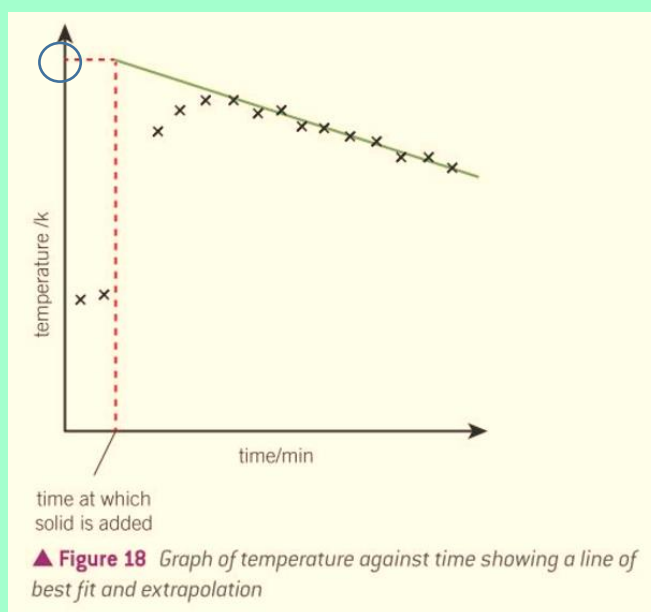
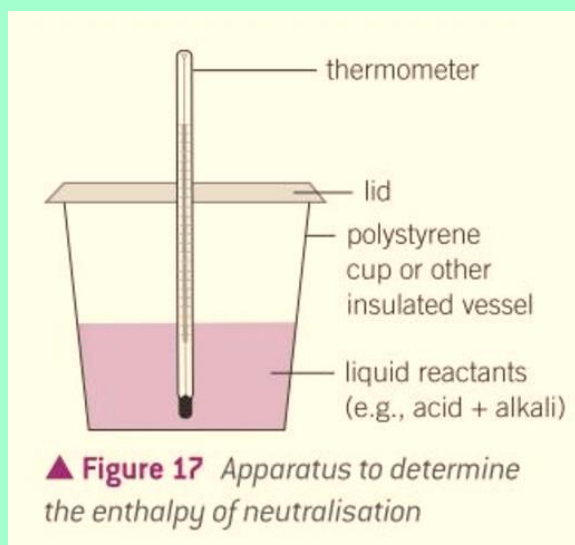
Enthalpy of neutralisation is always for one mole of water produced so....

$$E(\text{KJ}) = mc\Delta T = (\text{mass of solution} \times 4.18 \times \Delta T) / 1000$$

$$\Delta H (\text{KJ mol}^{-1}) = E/n = \text{energy (KJ)} / \text{moles of water produced}$$

(This scales the enthalpy of reaction to enthalpy of neutralisation and so makes it for one mole of water formed)

We use the stoichiometric values in the equation to find how many moles of water formed.



Notice that if we are asked to draw a line of best fit for the data we don't draw a curve. Instead, we ignore the initial readings and draw a line for the straight portion and extrapolate it back. The circle shows the point at which the maximum temperature is. This is only obtained by extrapolation and is the highest point on the line of best fit immediately after the reagents are mixed.

The values of temperature recorded are too low since the reaction does not occur instantaneously but over a period of time.

1. Throughout the reaction, heat is released and some is lost from the reaction vessel to the surroundings and air particles.
2. Heat may also be lost through evaporation if a lid is not used.
3. Side reactions may occur if there are impurities.

The enthalpy of neutralisation is:

The enthalpy change that occurs when one mole of H^+ ions reacts with 1 mole of OH^- ions under standard conditions to form 1 mole of H_2O and in solutions containing 1 mol dm^{-3} .

Given that all acid base reactions have the same ionic equation: $H^+ + OH^- \rightarrow H_2O$ the standard enthalpy change for neutralisation will always be the same ($\sim 58\text{ kJ mol}^{-1}$)

