

Chemistry 3B

Sulphate solubility

If a solution of any sulphate is added to a solution of a group 2 metal compound then a precipitate is likely.

| Group 2 ion in solution | Effect of adding a sulphate solution |
|-------------------------|--|
| Mg ²⁺ | No precipitate, MgSO ₄ is soluble |
| Ca ²⁺ | White precipitate of CaSO ₄ |
| Sr ²⁺ | White precipitate of SrSO ₄ |
| Ba ²⁺ | White precipitate of BaSO ₄ |

Hydroxide solubility

If sodium hydroxide is added to a solution of a group 2 compound then a precipitate is likely.

| Group 2 ion in solution | Effect of adding a hydroxide solution |
|-------------------------|--|
| Mg ²⁺ | Faint white precipitate of Mg(OH) ₂ |
| Ca ²⁺ | Faint white precipitate of Ca(OH) ₂ |
| Sr ²⁺ | Faint white precipitate of Sr(OH) ₂ on standing |
| Ba ²⁺ | No precipitate, Ba(OH) ₂ is soluble |

Heating carbonates and nitrates

| Substance | Effect of heat |
|------------------------------------|--|
| Lithium and all group 2 carbonates | Carbon dioxide detected |
| Sodium and potassium carbonates | No effect (except water of crystallisation may be given off) |
| Sodium and potassium nitrates | Oxygen only gas evolved |
| Lithium and all group 2 nitrates | Nitrogen dioxide and oxygen evolved |

Action of heat on compounds

- Carbonates - Carbon dioxide is given off.
- Hydrogencarbonates - Carbon dioxide and water formed.
- Group 1 nitrates - Nitrite and oxygen formed.
- Group 2 nitrates - Oxide, brown fumes of nitrogen dioxide and oxygen formed.

Distinguishing between hydrocarbons

- Alkane: Burn /oxidise/combust them. They will burn with a yellow flame and form CO₂ and H₂O (limited supply of CO).

- Alkene: A yellow, sootier flame is produced (due to the extra carbon and higher ratio of carbon:hydrogen).

Identifying some functional groups

- Alkene - Add to orange bromine water. The alkene will decolourise it.
- Halogenalkane - Heat with sodium hydroxide solution. Acidify with dilute nitric acid and then test with silver nitrate solution as with inorganic halides.
- Alcohols or carboxylic acids containing C-OH - In a dry test tube (i.e. dry alcohol), add PCl₅. Misty fumes of HCl are produced, which turns blue litmus paper red.

Distinguishing between different classes of alcohol

Primary - Add PCl₅. Warm it with aqueous potassium dichromate (K₂Cr₂O₇) and dilute H₂SO₄. Misty fumes are given off and the colour changed from orange --> aldehyde.

Secondary - Misty fumes, changes to green --> ketone.

Tertiary - Misty fumes, no colour change.

Specification

f) interpret quantitative and qualitative results.

g) devise and plan simple experiments based on the chemistry and techniques summarised in a to e above. Normally the last question on the paper. It will ask you to identify certain compounds from four. E.g. titrations, how to make a standard solution, how to titrate, identify QCO₃ when heating it where Q is a group 2 metal ion.

h) evaluate error in quantitative experiments see Appendix I for material available to assist centres in teaching this area
 Percentage error = $\frac{\text{absolute uncertainty}}{\text{actual value}} \times 100\%$

j) comment on safety aspects of experiments based on supplied data or recall of the chemistry of the compounds listed in Units 1 and 2. Safety considerations should relate to specific experiments not be of a general nature it will be assumed that students wear eye protection during all practical work.

Halogens are toxic and harmful by inhalation, although iodine is much less so than chlorine or bromine, because it is a solid. Chlorine and bromine must always be used in a fume cupboard. Liquid bromine causes serious burns and must be handled with gloves.

Ammonia is toxic. Concentrated ammonia solutions should be handled in the fume cupboard.

Concentrated mineral acids are corrosive. If spilt on the hands, washing with plenty of water is usually enough, but advice must be sought. Acid in the eye requires immediate attention and prompt professional medical attention.

Barium chloride solution and chromates and dichromates are extremely poisonous and so should be used in the fume cupboard/should not be inhaled.

Sodium or potassium hydroxide or concentrated ammonia in the eye is extremely serious and must always receive professional and immediate attention. Sodium hydroxide and other alkali metal hydroxides are amongst the most damaging of all common substances to skin and other tissue. Wear gloves, goggles and an apron when handling these solutions in high concentrations.

General safety

- Toxic/carcinogenic – use gloves, fume cupboard
- Flammable – Water baths, no naked flames.
- Harmful gases – Use fume cupboard
- Corrosive – wear goggles/gloves
- Spillage of concentrated acid – wash with plenty of water.

tests

| Ion | Formula | Test | Observations |
|-------------------|--------------------|---|---|
| Carbonate | CO_3^{2-} | Test 1 Heat the solid in a test tube with a Bunsen burner. It should decompose producing the oxide and carbon dioxide. E.g. $\text{CuCO}_{3(s)} \longrightarrow \text{CuO}_{(s)} + \text{CO}_{2(g)}$ Test for the gas using limewater solution. | Limewater should turn from colourless to cloudy in the presence of carbon dioxide due to precipitation of calcium carbonate. |
| | | Test 2 Add dilute HCl to the solid. Test for the gas evolved using limewater solution. | Vigorous effervescence. Limewater should turn from colourless to cloudy in the presence of carbon dioxide due to precipitation of calcium carbonate. |
| Hydrogencarbonate | HCO_3^- | Test Add calcium chloride to a hydrogencarbonate solution. | No precipitate forms since calcium hydrogencarbonate is soluble. |
| Sulphate (VI) | SO_4^{2-} | Test Add barium chloride solution acidified with dilute HCl to the test solution. | White precipitate of barium sulphate forms. |
| Sulphite | SO_3^{2-} | Test Warm the sulphite with dilute HCl. Test for gases using acidified potassium dichromate(VI) solution (or paper) | The solution turns green. |
| Chloride | Cl^- | Test 1 Add concentrated sulphuric acid to the solid chloride. | White steamy acidic fumes are seen - HCl fumes. |
| | | Test 2 Add dilute nitric acid to a solution of a chloride to acidify the solution. This eliminates any carbonates or sulphites. Add silver chloride to the solution. Add dilute ammonia solution. | White precipitate of AgCl forms. Solid dissolves. |
| Bromide | Br^- | Test 1 Add concentrated sulphuric acid to the solid bromide. | Steamy brownish acidic fumes are seen. |
| | | Test 2' Add dilute nitric acid to a solution of a bromide to acidify the solution. This eliminates any carbonates or sulphites. Add silver chloride to the solution. Add concentrated ammonia solution. | Cream precipitate of AgBr forms. Solid dissolves. |
| Iodide | I^- | Test 1 Add concentrated sulphuric acid to the solid iodide. | Purple acidic fumes are seen. The mixture turns to a brown slurry. |

| | | | |
|------------------|------------------|---|---|
| | | Test 2 Add dilute nitric acid to a solution of a iodide to acidify the solution. This eliminates any carbonates or sulphites. Add silver chloride to the solution. Add concentrated ammonia solution. | Yellow precipitate of AgI forms. Solid is insoluble. |
| Nitrate | NO_3^- | Test 1 Heat solid nitrate. If group 1 solid (not Li) then will decompose to give the nitrite and oxygen. All other solid nitrates decompose to give the metal oxide, nitrogen dioxide and oxygen. | Oxygen gas is evolved that will relight a glowing splint. Brown gas is seen (NO_2). Oxygen gas is also evolved and will relight a glowing splint. |
| | | Test 2 Boil nitrate solution with aluminium/Devarda's alloy, in sodium hydroxide solution. Test vapour with red litmus paper. | Litmus paper turns blue in the presence of ammonia. |
| Ammonium | NH_4^+ | Test Warm ammonium compound with NaOH. Test vapours immediately using damp red litmus paper. | NH_3 turns the litmus paper blue. |
| Lithium | Li^+ | Test Dip nichrome wire in HCl. Dip wire in solid. Heat wire in centre of flame. Observe colour of flame. | A carmine red flame is seen. |
| Sodium | Na^+ | Test Dip nichrome wire in HCl. Dip wire in solid. Heat wire in centre of flame. Observe colour of flame. | A yellow flame is seen. |
| Potassium | K^+ | Test Dip nichrome wire in HCl. Dip wire in solid. Heat wire in centre of flame. Observe colour of flame. | A lilac flame is seen. |
| Magnesium | Mg^{2+} | Test Add NaOH solution to the magnesium solid. | A white solid forms which is insoluble in excess NaOH(aq). This is $\text{Mg}(\text{OH})_2(\text{s})$ |
| Calcium | Ca^{2+} | Test | A brick red flame is seen. |

| | | | |
|------------------|------------------|---|------------------------------|
| | | Dip nichrome wire in HCl. Dip wire in solid. Heat wire in centre of flame. Observe colour of flame. | |
| Strontium | Sr ²⁺ | Test Dip nichrome wire in HCl. Dip wire in solid. Heat wire in centre of flame. Observe colour of flame. | A crimson red flame is seen. |
| Barium | Ba ²⁺ | Test Dip nichrome wire in HCl. Dip wire in solid. Heat wire in centre of flame. Observe colour of flame. | A apple green flame is seen. |

Tests

| Name | Formula | Test | Observations |
|-----------------------|-----------------|--|--|
| Hydrogen | H ₂ | Ignite gas. | Squeaky pop is heard. |
| Oxygen | O ₂ | Place a glowing splint in a sample of the gas. | The glowing splint relights. |
| Carbon dioxide | CO ₂ | Bubble gas through limewater (saturated solution of calcium hydroxide) | A solution turns from colourless to cloudy. A white (milky) precipitate of calcium carbonate forms which is sparingly soluble. |
| Ammonia | NH ₃ | Test for gas using damp red litmus paper. | Litmus paper turns blue. |
| Chlorine | Cl ₂ | Test 1 Test for gas using damp litmus paper (red or blue) | Chlorine bleaches the litmus paper very quickly. |
| | | Test 2 Test for gas using moist starch-iodide paper. | The paper turns blue-black. |
| | | Test 3 Pass gas through a solution of a bromide. | The solution turns from colourless to orange. |
| | | Test 4 Pass gas through a solution of an iodide. | The solution turns from colourless to brown (possibly with a black precipitate, iodine). |

| | | | |
|-------------------------|-----------------|--|---|
| Nitrogen dioxide | NO ₂ | Not many tests for this gas. | The gas is brown. |
| Sulphur dioxide | SO ₂ | Test 1 Bubble gas through a solution of potassium dichromate (VI) dissolved in sulphuric acid. | The solution turns from orange to green. |
| | | Test 2 Bubble gas through a solution of potassium manganate (VII) dissolved in sulphuric acid. | The solution turns from purple to colourless. |

Volumetric analysis

Volumetric analysis (titration) involves the reaction between two solutions. For one solution, both the volume and the concentration are known; for the other, the volume only is known. Apparatus used includes a burette, a pipette and a volumetric flask.

What is a standard solution?

A solution for which concentration is accurately known. The concentration may have been found by a previous titration or by weighing the solute and making a solution of known volume. Such a solution is a primary standard solution.

How is a 250cm³ standard solution prepared?

- Make sure that the balance is clean and dry. Wipe it with a damp cloth.
- Place the weighing bottle on the pan and tare the balance (i.e. re-zero it)
- Take the bottle off the balance and add solid to it. This ensures that no spillages fall on the pan.
- When you have the required amount, write its value down immediately.
- Replace on balance, and if the required amount is added, withdraw the mass.
- Wash out a 250cm³ volumetric flask three times using pure water.
- Transfer the solid to a 250cm³ volumetric flask using a funnel, and wash out the weighing bottle into the flask through the funnel.
- Add about 100cm³ of distilled water to the flask.
- Stir the solution using a glass rod.
- Wash all remaining apparatus including the glass rod, funnel and transfer the rest of this to the flask.
- Make up to 250cm³ with distilled water so that the bottom of the meniscus just touches the 250cm³ mark.
- Stopper the flask.
- Shake the flask vigorously and/or invert the flask 5 or 6 times to dissolve the solid.
- Concentration of solution = mass of solid used/molar mass of solid x 1000/250 (units moldm⁻³)

Using the pipette

- A glass bulb pipette will deliver the volume stated on it within acceptable limits only.
- Using a pipette filler, draw a little of the solution to be used into the pipette and use this to rinse the pipette.
- Fill the pipette to about 2-3cm³ above the mark. Pipette fillers are difficult to adjust accurately, so quickly remove the filler and close the pipette with your forefinger (not thumb). Release the solution until the bottom of the meniscus is on the mark.
- Immediately transfer the pipette to the conical flask in which you will do the titration, and allow the solution to dispense under gravity.

Using the burette

- Making sure that the tap is shut, add about 10-15cm³ of the appropriate solution to the burette and rinse it out, not forgetting to open the tap and rinse the jet.

- Close the tap and fill the burette. A small funnel should be used to add the solution but be careful not to overfill the funnel.
- Remove the funnel, because titrating with a funnel in the burette can lead to serious error if a drop of liquid in the funnel stem falls into the burette during the titration.
- Bring the meniscus on to the scale by opening the tap to allow solution to pass through the burette. There is no particular reason to bring the meniscus exactly to the zero mark.
- Make sure that the burette is full to the tip of the jet.
- After a suitable indicator has been added to the solution in the conical flask, swirl the flask under the burette with one hand whilst adjusting the burette tap with your other hand.
- Add the solution in the burette to the conical flask slowly, swirling the flask all the time.
- As the endpoint is approached, the indicator will change colour more slowly. The titrant should be added drop by drop near to the endpoint.
- Repeat the titration until you have three concordant titres, i.e. volumes that are similar. This means within 0.2cm³ or better if you have been careful. Taking the mean of three titres that differ by 1cm³ or more is no guarantee of an accurate answer.

Common indicators

- Methyl orange - yellow in alkali, red in acid (orange at end point)
- Phenolphthalein - pink in alkali, colourless in acid.

Enthalpy change measurements

- Weigh a spirit lamp (containing a liquid alcohol) using a balance accurate to 3 decimal places. Record the mass measured.
- Use a measuring cylinder to put 100 cm³ of distilled water into a small beaker and clamp this at a fixed height above the spirit lamp (about 2 cm).
- Record the initial temperature of the water using a thermometer.
- Light the lamp using a burning splint.
- Heat the water using the spirit lamp until the temperature has gone up by about 10°C. Stir the water with the thermometer the whole time.
- Put a cap on the spirit to stop the alcohol burning. The lid stops also stops further evaporation of the liquid alcohol.
- Reweigh the spirit lamp and record the mass.
- Calculate the enthalpy change

Possible sources of error

- There may be heat loss due to the apparatus used and heat may have dissipated through the insulating material --> should use a polystyrene cup and insulation like a lid.
- The specific heat capacity and density of water are used (and not of HCl).
- The masses of solid added to the acid are ignored.
- It is assumed that the specific heat capacity of the polystyrene cup is negligible.
- Some heat is lost when the hydrogen or carbon dioxide are evolved in the reactions.

Many organic reactions are slow and require prolonged heating

- To achieve this without loss of liquid, reaction mixtures are heated in a flask carrying a vertical condenser.
- This is heating under reflux; the solvent is condensed and returned to the flask, so the mixture can be heated as long as desired.

- To heat the round bottomed flask, either use a water bath, an oil bath or a heated plate mantle. A Bunsen burner isn't really suitable.

Simple distillation

To separate a volatile solvent from a mixture

- Simple distillation is used where a volatile component has to be separated from a mixture, the other components of the mixture being very much volatile or non-volatile.
- The mixture is heated.
- The fraction that boils is collected within the temperature range of the fraction. (normally 1 or 2 degrees before the boiling temperature)
- The condenser cools the fraction so it distils and is collected in the receiving flask.

Fractional distillation

To separate mixtures of volatile liquids.

- Re-crystallisation - Used to purify a solid material by removing both soluble and insoluble impurities. The choice of solvent is important. The substance must be easily soluble in the boiling solvent and much less soluble at room temperature. This ensures the smallest possible loss of material, although some loss is inevitable with this technique.

Re-crystallisation method

1. Dissolve the solid in the minimum amount of boiling solvent. This ensures that the solution is saturated with respect to the main solute but not with respect to the impurities, which are present in much smaller amounts.
2. Filter the hot mixture through a preheated filter funnel. This removes insoluble impurities. The hot funnel is necessary to prevent the solute crystallising and blocking the funnel. Filtration under vacuum using a Buchner funnel is often preferred, because it is fast.
3. Cool the hot filtrate, either to room temperature or, if necessary, in a bath of iced water. Rapid cooling gives small crystals, slow cooling large ones. The large crystals are often less pure.
4. Filter the cold mixture using a Buchner funnel.
5. Wash the crystals with a small amount of cold solvent. This removes any impurity remaining on the surface of the crystals. A small amount of cold solvent is used so that the crystals aren't washed away / don't dissolve.
6. Suck the crystals as dry as possible on the filter.
7. Transfer the crystals to a desiccator to dry. Drying between filter paper is sometimes recommended, but it is a very poor method.

Melting point determination

This is used to determine the purity of the re-crystallisation solid. Place small amount of the solid in the sealed end of a capillary tube. Place in the melting point apparatus. A sharp melting point over a small range shows purity, when compared with the set-book value of a higher melting point, that indicates an impure solid.

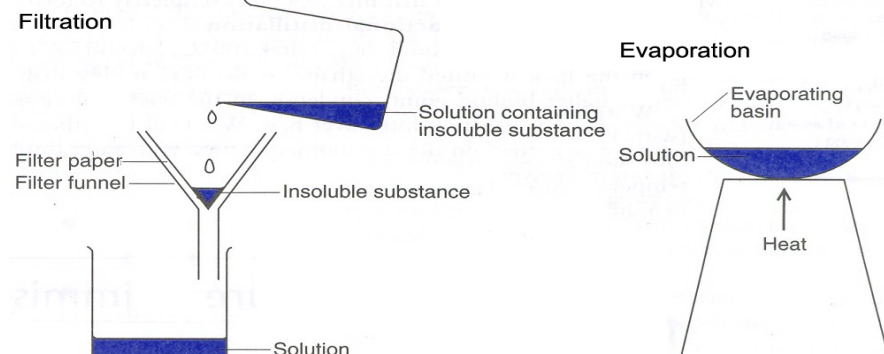
Organic tests

- Collect 10 cm³ of the samples.
- Test the samples in the following order
- Alkenes – bromine water --> decolourises --> alkene

- Alcohols – Spatula of solid PCl_5 . Test fumes with damp litmus paper --> litmus red? White fumes near ammonia? --> alcohol
- Halogenoalkane – Add NaOH , ethanol as solvent. Shake and warm for 3 minutes. Cool and add nitric acid + silver nitrate. --> white = chloride; cream = bromide; yellow = iodide. --> Confirm with ammonia
- Alkane, the substance left is the alkane.

Techniques

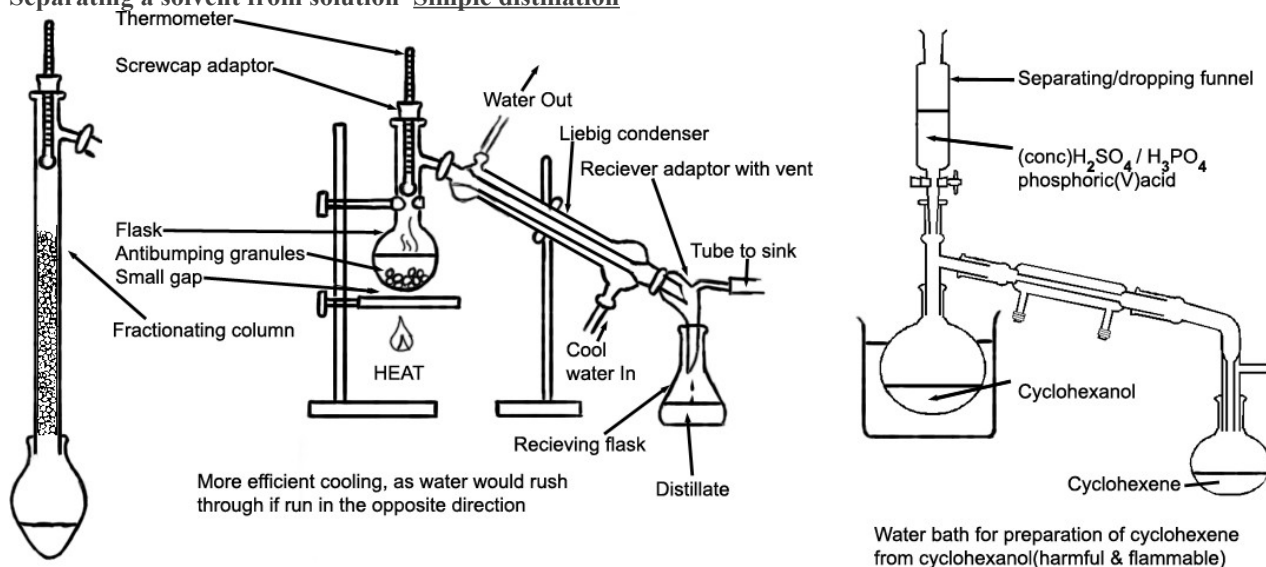
Separating insoluble impurities from a soluble substance (Removing sand and impurities from salt solution)



Separating a mixture of immiscible liquids (Separating a mixture of water and hexane)

Water and hexane are immiscible forming 2 separate layers and are separated using a **separating funnel**

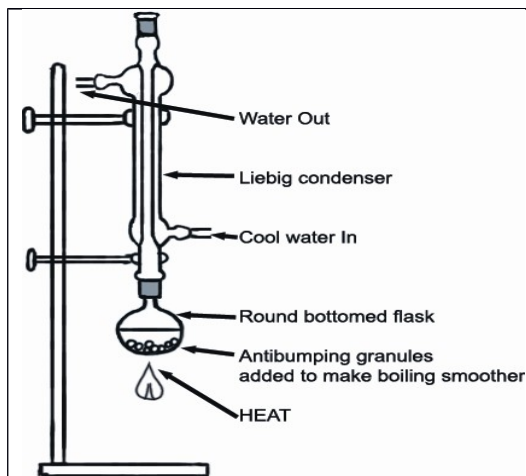
Separating a solvent from solution Simple distillation



Separating a liquid from a mixture of miscible liquids

Fractional distillation Separates mixtures of miscible liquids with different Bt's, using a fractionating column increasing efficiency of redistillation process, packed with inert material(glass beads) increasing surface area where vapour may condense.

- When mixture is boiled vapours of most volatile component(lowest Bt) rises into the vertical column where they condense to liquids.
- As they descend they are reheated to Bt by the hotter rising vapours of the next component.
- Boiling condensing process occurs repeatedly inside the column so there is a temperature gradient.
- Vapours of the more volatile components reach the top of the column and enter the condenser for collection



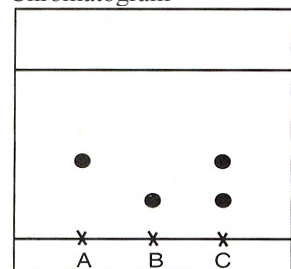
Boiling under reflux Where reagents volatile

- condenses vapours and returns reagents to flask, prevents loss of reactants/products, prolonged heating for slow reactions
- For preparation of aldehyde/carboxylic acid from alcohol

- (1) Reason for heating the mixture but then taking the flame away
(1) provide E_a , exothermic/prevent reaction getting out of control

Separating mixtures of similar compounds in solution (Separating dyes present in a sample of ink)

Chromatogram



x Original position of each substance
● Final position of each substance

Chromatography

Different components of the dye spread out at different rates
Using a square sheet of filter paper, spots of dye solutions are put along the baseline
The filter paper is coiled into a cylinder and placed in a tank containing a small volume of solvent
The lid is replaced on the tank, solvent rises up the filter paper
When the solvent nearly reaches the top of the filter paper, the filter paper is removed and position of solvent marked.

Dyes A & B are either pure substances or a mixture of dyes not separated with the solvent used
Dye C is composed of A & B as the spots correspond
Colourless substances can be separated and seen by spraying/dipping the filter paper into a **locating agent** which colours the spots produced

Separating a solid which sublims, from a solid which doesn't sublime

Given a mixture of Ammonium chloride(sublimes) and sodium chloride(doesn't sublime)

Heat the mixture. Ammonium chloride turns directly to vapour but the sodium chloride remains unchanged

When the vapour is cooled solid ammonium chloride collects free from sodium chloride

A pure substance has a definite M_t , presence of impurities causes the substance to melt over a range of temperatures

Best method of separation of (1) Oil and water (2) Alcohol and water (3) Nitrogen from liquid air

(1) Separating funnel(2) Fractional distillation (3) Fractional distillation

| Mixture | Compound |
|--|--|
| <ul style="list-style-type: none"> - Proportions of the different elements can be varied - Properties are those of the elements making it up - Elements can be separated by simple methods - No energy gained or lost when the mixture is made | <ul style="list-style-type: none"> - Different elements have to be present in fixed proportions - Properties different from properties of elements making it up - Difficult to separate into the elements which make it up - Energy usually given out/taken in when compound is formed |

Sub-atomic particles

Protons, neutrons and electrons which makes up the atom

Particle An atom, molecule, ion, electron or any identifiable particle **RTP** Room temperature and pressure

Electron A negatively charged particle, with negligible mass occupying the outer regions of all atoms

Immiscible Unable to mix, dissolve in each other, to form a homogenous mixture **Miscible** Soluble in each other

(aq) Substance dissolved in water to form an aqueous solution

State symbols Physical state of the reactants at RT **Aqueous(aq)**

Solvent Substance in which other substances are dissolved

Solute Substance dissolved in another substance(solvent)to form a solution

Chemical species Collection of particles **Distilled water** Water that has been purified by distillation

| PARTICLES | STRUCTURES | Ion |
|-----------|---|--|
| Atoms | Molecules (found in solids, liquids and gases) | When number of protons and electrons are different |
| Ions | Giant structures (solids at room temperature) | Atom The smallest part of an element that can exist on its own |
| | Free ions (found in molten salts and solutions) | Molecule 2 or more atoms bonded together |
| | | Element A pure substance which can't be split up by chemical reaction |
| | | Compound Combination of elements in fixed proportions via synthesis |
| | | In formation of a compound from ions the charges balance out |

Physical properties: M_t , B_t , hardness

• Compounds ending in **-ate** **-ite** contain oxygen, greater proportion of oxygen in the compound ending in **-ate**

- | | | | |
|-----------------|--------------------------|-----------------|--------------------------|
| Sodium sulphate | Na_2SO_4 | Sodium sulphite | Na_2SO_3 |
|-----------------|--------------------------|-----------------|--------------------------|
- Compounds with prefix **per**- contain extra oxygen

| | | | |
|--------------|-----------------------|-----------------|-------------------------|
| Sodium oxide | Na_2O | Sodium peroxide | Na_2O_2 |
|--------------|-----------------------|-----------------|-------------------------|
 - Compounds with prefix **thio**- contain a sulphur atom in place of an oxygen atom

| | | | |
|-----------------|--------------------------|---------------------|-----------------------------------|
| Sodium sulphate | Na_2SO_4 | Sodium thiosulphate | $\text{Na}_2\text{S}_2\text{O}_3$ |
|-----------------|--------------------------|---------------------|-----------------------------------|

Metalloid Element which has properties between metals and nonmetals

- Ions in an ionic compound are tightly held together in a regular lattice, lattice energy is required to break it up and melt the substance

A metal high in the reactivity series has stable ores and the metal can be obtained only by electrolysis

A metal middle in the reactivity series doesn't form stable ores and can be extracted by reduction reactions (often with carbon)

A metal low in the reactivity series, if present in unstable ores can be extracted by heating

Decomposition Splitting up of a compound (Thermal decomposition - decomposition of a compound by heating)

Combustion is the reaction of a substance with oxygen, total mass of products is greater than the mass of the substance burned, difference being the mass of oxygen combined

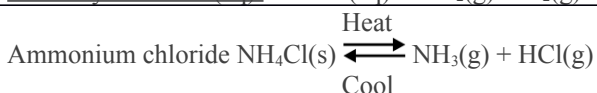
Sublimation of an element/compound is a transition from solid to gas with no intermediate stage

When a change of state takes place the temperature remains constant despite a continuing supply of energy. Latent heat is the energy which is not being used to raise the temperature and supplies particles with the extra energy they require as the state changes (given out when the reverse changes take place)

$\text{Fe(s)} + \text{CuSO}_4\text{(aq)} \rightarrow \text{FeSO}_4\text{(aq)} + \text{Cu(s)}$ Blue solution turns colourless and brown copper is deposited

A displacement reaction where a more reactive metal replaces a less reactive metal in a compound

Electrolysis of HCl(aq): $2\text{HCl(aq)} \rightarrow \text{Cl}_2\text{(g)} + \text{H}_2\text{(g)}$ Cathode: $2\text{H}^+\text{(aq)} + 2\text{e}^- \rightarrow \text{H}_2\text{(g)}$ Anode: $2\text{Cl}^-\text{(aq)} \rightarrow \text{Cl}_2\text{(g)} + 2\text{e}^-$

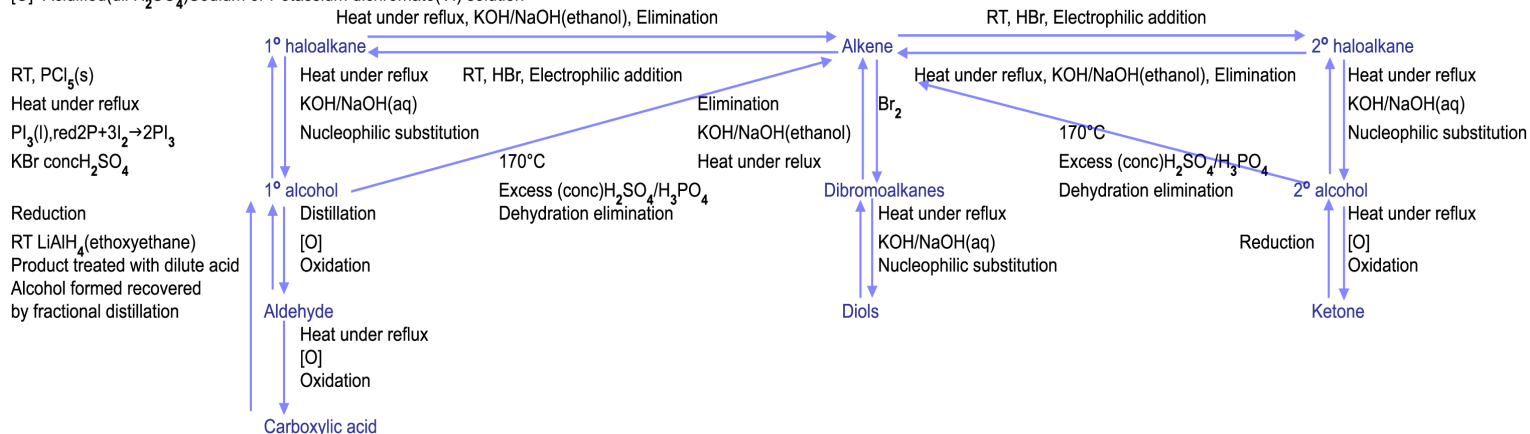


A stopper from a bottle of (conc) $\text{NH}_3\text{(aq)}$ held near a stopper from a bottle of (conc) HCl acid gives a dense white smoke of NH_4Cl

Synthetic Pathways (Series of reactions built up to convert one functional group into another)

- Reactions of functional groups assumed to be the same whether molecules are simple or complicated
- Synthesis of the product molecule possible because in any reaction of a functional group a product is formed capable of conversion into other molecules

[O]=Acidified (dil H_2SO_4) Sodium or Potassium dichromate(VI) solution



$\Delta H = \frac{-5.31 \times 4.18 \times \Delta T}{\text{mass of G used (g)}} \text{ kJ mol}^{-1}$ Would the result for ΔH be more accurate if the temperature of the solution were known to 3dp? Yes, temperature would then be known to a comparable precision to the other factors in the equation

Plan an experiment to investigate concentration on rate of a reaction $\text{Mg(s)} + 2\text{HCl(aq)} \rightarrow \text{MgCl}_2\text{(aq)} + \text{H}_2\text{(g)}$

- Apparatus diagram/description/addition of Mg to acid in appropriate container
- Weigh Mg/cut measured length and measure volume of acid, measure volume of H_2 with time/time how long it takes for Mg to 'dissolve' • repeat with different concentration(s) HCl • repeat with same mass/length Mg and same volume HCl
- one axis labelled concentration/volume H_2 and other axis time/t axis changed to $1/t$

if measured volume H_2 at least two curves showing increasing rate with conc

if measured time to dissolve Mg one line showing time decreasing with conc or $1/t$ increasing with conc

- Acid irritant so wear gloves or hydrogen explosive - no naked flame or Build up of pressure in syringe - attach plunger with string

$(\text{NH}_4)_2\text{CO}_3$ reacts with both 1 mol dm^{-3} (dil) HNO_3 and 1 mol dm^{-3} (dil) KOH in the ratio 1:2

Devise an experiment to determine which of the two reactions is the more exothermic

- Prepare solutions of known concentration of the solid

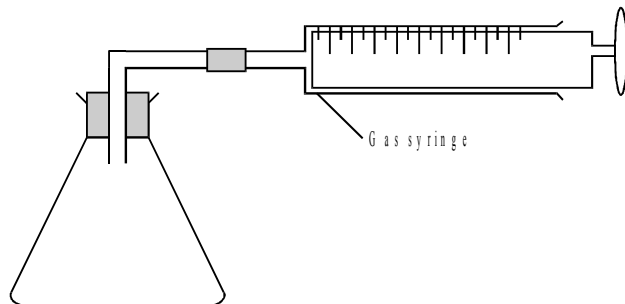
- Suggestion of apparatus used e.g. lagged calorimeter or low mass polystyrene cup
- Use same volume of each solution
- Measure maximum temperature change
- Improve reliability of results, repeat experiment
- Possible sources of error identified
- Reaction with the greater temperature change is the more exothermic
- Since ammonia evolved use a fume cupboard



Plan an experiment, results of which used to calculate the relative molecular mass of the carbonate and identify M

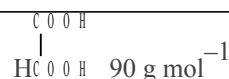
At temp of experiment 1 mole of CO_2 occupies a volume of $24dm^3$

Relative atomic mass: Li = 7, C = 12, O = 16, Na = 23, K = 39, Rb = 85, Cs = 133



- Add M_2CO_3 + acid and stopper flask/use suspended test tube in large flask
- Mass of M_2CO_3 and (final)vol of CO_2 /when effervescence stops record volume of gas in syringe
- Explain conversion volume of CO_2 to moles by correct use of 24
- Use of moles $M_2CO_3 = \frac{\text{mass } M_2CO_3}{M_r M_2CO_3}$ to find M_r • Hence find identity of M
- Escape of gas before bung replaced/solid did not all react/ CO_2 soluble in acid
- Eye protection-acid hazard
- Should not affect identification since even if M_r is slightly wrong it will still correspond to nearest Group 1 metal atomic mass

Plan an experiment to identify an acidic compound, molar mass for an acid estimated to be $88 \pm 2 \text{ g mol}^{-1}$
What simple test would allow butenoic acid to be distinguished from the other two? Devise a plan based upon a quantitative experiment that would allow the other two to be distinguished



3 structures proposed for this acid

Alkene detected using bromine water, only 1 would show decolourisation

Fixed mass of acid, Standard named alkali, Controlled method of addition, To an identified end point of a named suitable indicator, Calculation of mole ratio

(1)A sample of NaCl was thought to contain an impurity of $Ba(NO_3)_2$ A student suggested a flame test

(a)(i)Suggest why a flame test on the mixture would not be a satisfactory way of detecting the presence of barium ions in the sample

(i) • yellow/stronger/persistent Na flame • Obscures/Ba flame *NOT* “makes it difficult to distinguish between the two colours”

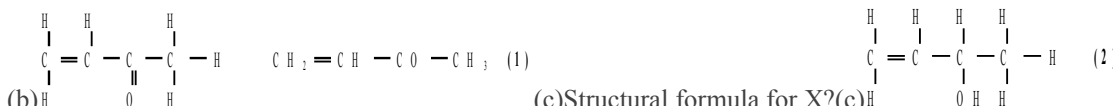
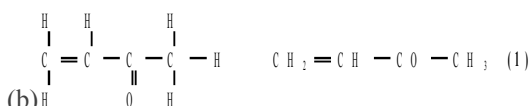
(ii)Suggest a reagent that could be used to produce a ppt of a barium compound from a solution of the sample

(ii) • (conc) H_2SO_4 (solution) of any soluble sulphate ($MgSO_4$, $(NH_4)_2SO_4$, Na_2SO_4)

(2)(a)X(has OH group)decolourised cold potassium manganate(VII) acidified with (dil) H_2SO_4 acid, structure of X suggested by this?

(a)carbon double bond

(b)Complete oxidation of X with potassium dichromate(VI) solution and (dil) H_2SO_4 acid produces Y C_4H_6O structure for Y?



(b) $\begin{array}{c} H & H & & H \\ | & | & & | \\ C & = & C & - & C & - & C & - & H \\ | & & & | \\ H & & & H \end{array}$

(c)Structural formula for X?(c) $\begin{array}{c} H & H & H & H \\ | & | & | & | \\ C & = & C & - & C & - & C & - & H \\ | & & & | \\ H & & & H \end{array}$

X must be a secondary alcohol because ketone formed on oxidation carboxylic acid is not formed

(3)(a)Write an ionic equation for the hydrolysis of 1-bromobutane by water (a) $CH_3CH_2CH_2CH_2Br + H_2O \rightarrow CH_3CH_2CH_2CH_2OH + H^+ + Br^-$

(b)Suggest why ethanol was used in the experiment (b)Solvent/silver nitrate solution in water and bromobutane immiscible

(c)Suggest a reason for the use of a water bath (c)Reaction slow at RT/increases rate/flamable

(4)Describe tests you would use to distinguish between the following pairs of compounds including results

(a) $NaNO_3$ and ammonium nitrate NH_4NO_3 (a) Flame test, sodium salt gives yellow colour, ammonium salt gives no colour

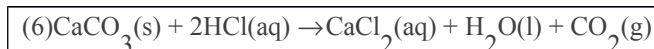
(5)(a)1-bromobutane Bt102 °C may be prepared by the reaction $C_4H_9OH + NaBr + H_2SO_4 \rightarrow C_4H_9Br + NaHSO_4 + H_2O$

(a)Describe how you would use distillation apparatus to give a sample of pure 1-bromobutane

(a)Heat mixture(slowly), collect only distillate produced at around 102 °C at Bt of 1-bromobutane

(b)Suggest 2 reasons why the actual yield was much lower than the max yield

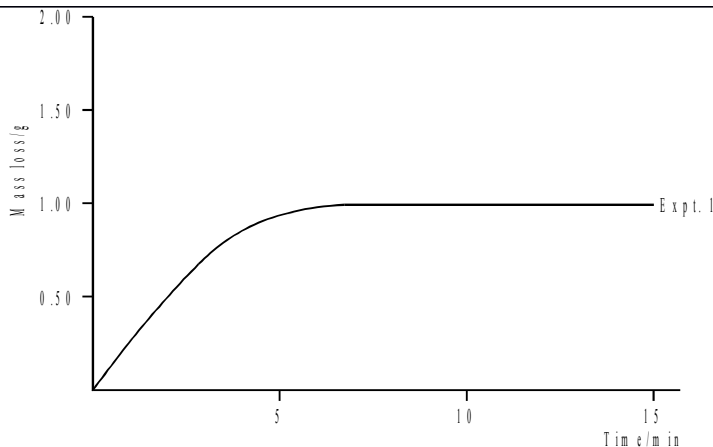
(b)side reactions, reaction incomplete, product lost in purification/transfers



| Experiment | CaCO ₃ | HCl acid |
|------------|-------------------|---|
| 1 RT | Small pieces | 50cm ³ of 1mol dm ⁻³ |
| 2 | Small pieces | 50cm ³ of 1mol dm ⁻³ heated to 80°C |
| 3 RT | One large piece | 50cm ³ of 1mol dm ⁻³ |
| 4 RT | Small pieces | 50cm ³ of 2mol dm ⁻³ |

(a)(i) Explain why there is a loss in mass as the reaction proceeds
(i) CO₂(g) evolved

(ii) Explain the shape of the curve drawn for Experiment 1
(ii) Reaction (fast at first then) slows down/gives off less CO₂ per min when line is horizontal, the reaction has finished/after 6 or 7 minutes/when 1 g of CO₂ lost



Results of Experiment 1

(b) Draw curves on the graph to represent the results you would expect for Experiments 2, 3 and 4. Label the curves 2, 3 and 4
(b) Experiment 2 steeper than 1 and same mass loss Experiment 3 less steep than 1 and same mass loss/reaction incomplete
Experiment 4 steeper than 1 and horizontal at twice mass loss

(7) Suggest one appropriate safety precaution that should be taken as ethanedioic acid is toxic (7) Safety pipette filler

(8) In an experiment to find ΔH, zinc CuSO₄(aq) in a plastic cup Zn(s) + Cu²⁺(aq) → Zn²⁺(aq) + Cu(s)

Suggest reasons why a series of temp readings is taken rather than simply initial and final readings

(8) Reason 1 Any fluctuations in temperature smoothed out / minimises reading error/allows line of best fit to be drawn

Reason 2 Able to allow for cooling effect/able to calculate more accurate temperature change/need to find highest temperature

Another Test List (If you find above one hard to read)

Flame test

1 Clean end of platinum/nichrome wire with (conc)HCl, burning off impurities in a roaring bunsen flame until there's no persistent flame colouration

2 Moisten the end of the clean wire with (conc)HCl and then dip into the sample to be tested

3 Hold the sample at the edge of a roaring bunsen flame

| | | | |
|-----------|-------------|-----------|-------------|
| Lithium | Carmine red | Calcium | Brick red |
| Sodium | Yellow | Strontium | Crimson |
| Potassium | Lilac | Barium | Apple green |

| Gas | Test |
|---------------------------------------|---|
| Ammonia NH ₃ | Pungent smell, Moist litmus paper red → blue, (conc)HCl at mouth of bottle, white smoke forms |
| Carbon dioxide CO ₂ | Pass through lime water, turning lime water milky $\text{CaCO}_3(\text{s}) + \text{H}_2\text{O}(\text{l}) + \text{CO}_2(\text{g}) \rightleftharpoons \text{Ca}(\text{HCO}_3)_2(\text{aq})$ Limewater CO ₂ test $\text{Ca}(\text{OH})_2(\text{aq}) + \text{CO}_2(\text{g}) \rightarrow \text{CaCO}_3(\text{s})$ White ppt + H ₂ O(l) |
| Chlorine Cl ₂ | Swimming pool smell, moist litmus paper blue → red → bleached |
| Hydrogen H ₂ | Lighted splint, burns with squeaky pop |
| Hydrogen chloride HCl | Moist litmus paper blue → red |
| Nitrogen(IV) oxide NO ₂ | Brown gas, acrid smell, moist litmus paper blue → red |
| Oxygen O ₂ | Glowing splint, relights |
| Water vapour H ₂ O | White anhydrous copper(II) sulphate white → blue $\text{CuSO}_4(\text{s}) + 5\text{H}_2\text{O}(\text{l}) \rightleftharpoons \text{CuSO}_4 \cdot 5\text{H}_2\text{O}(\text{s})$ Or dry blue cobalt chloride paper blue → pink |
| Sulphur dioxide SO ₂ | Acrid smell, moist litmus paper blue → red Or potassium dichromate(VI) solution/paper from orange → green |
| Cation | Test |
| Ammonium NH ₄ ⁺ | • Heat, ammonia evolved, moist litmus paper red → blue |
| H ⁺ | • Moist litmus paper blue → red • Add a carbonate, pass gas through lime water, CO ₂ evolved turning lime water milky |
| Copper(II) Cu ²⁺ | • A little, blue ppt forms • In excess, insoluble |
| Iron(II) Fe ²⁺ | • A little, green ppt forms |

• A little, blue ppt forms
• In excess, dissolves and a dark blue solution forms
• A little, green ppt forms

| | | | |
|-------------------------------------|-------------------------------|--|---|
| | | • In excess, insoluble | • In excess, insoluble |
| Iron(III) | Fe ³⁺ | • A little, brown ppt forms • In excess, insoluble | • A little, brown ppt forms • In excess, insoluble |
| Calcium | Ca ²⁺ | • A little, milky suspension forms • In excess, insoluble | • A little, milky suspension forms • In excess, insoluble |
| Magnesium | Mg ²⁺ | Distinguish Mg from Ca through flame test | |
| | | • A little, milky suspension forms • In excess, insoluble | • A little, milky suspension forms • In excess, insoluble |
| Aluminium | Al ³⁺ | • A little, white ppt forms • In excess, dissolves giving colourless solution | • A little, white ppt forms • In excess, insoluble |
| Lead | Pb ²⁺ | No ppt with (dil)H ₂ SO ₄ /cold(dil)HCl/(dil)KI/(dil)Na ₂ S Sodium sulphide | |
| | | • A little, white ppt forms • In excess, dissolves giving colourless solution | • A little, white ppt forms • In excess, insoluble |
| | | White ppt with (dil)H ₂ SO ₄ Yellow ppt with (dil)KI | White ppt with cold(dil)HCl Black ppt with (dil)Na ₂ S Sodium sulphide |
| Zinc | Zn ²⁺ | • A little, white ppt forms • In excess, dissolves giving colourless solution | • A little, white ppt forms • In excess, dissolves giving colourless solution |
| Anion | | Test | |
| Carbonate pH>10 universal indicator | CO ₃ ²⁻ | Add (dil)HCl(aq) Pass gas through lime water, CO ₂ evolved turning lime water milky | |
| | | Or add group II ions, white ppt or heat/add boiling water, no gas evolved | |
| Hydrogen carbonate pH 8-9 | HCO ₃ ⁻ | Add (dil)HCl(aq) Pass gas through lime water, CO ₂ evolved turning lime water milky | |
| | | Or add metal ions no ppt but heating causes white ppt to form or Heat/add boiling water, CO ₂ evolved | |
| Chloride | Cl ⁻ | Acidify with (dil)HNO ₃ (aq) Add AgNO ₃ (aq) Add (dil)NH ₃ to ppt | White ppt AgCl forms Ppt dissolves leaving colourless solution |
| Bromide | Br ⁻ | Acidify with (dil)HNO ₃ (aq) Add AgNO ₃ (aq) Add (conc)NH ₃ to ppt | White ppt AgBr forms Ppt dissolves leaving colourless solution |
| Iodide | I ⁻ | Acidify with (dil)HNO ₃ (aq) Add AgNO ₃ (aq) Add (conc)NH ₃ to ppt | White ppt AgI forms Ppt dissolves leaving colourless solution |
| Nitrate | NO ₃ ⁻ | Acidify with (dil)HNO ₃ (aq) Add Devarda's alloy (powdered Zn, Al) | Heat & hold moist red litmus at mouth of test tube NH ₃ evolved, litmus paper red → blue |
| Sulphate | SO ₄ ²⁻ | Add Barium nitrate Ba(NO ₃) ₂ (aq)/chloride BaCl ₂ (aq) Add HCl(aq) | White ppt Insoluble |
| Sulphite | SO ₃ ²⁻ | Add Barium nitrate Ba(NO ₃) ₂ (aq)/chloride BaCl ₂ (aq) Add HCl(aq) | White ppt Dissolves |
| | | Or add (dil)HCl(aq) Heat | SO ₂ evolved turning potassium dichromate(VI) solution/paper from orange → green |

Practical List

1. Make a salt and calculate the percentage yield (hydrated nickel sulfate)
2. make a salt and calculate the percentage yield (ammonium iron(II) sulfate)
3. carry out and interpret results of simple test tube reactions
4. measuring some enthalpy changes
5. finding the enthalpy of combustion of an alcohol
6. finding an enthalpy change that cannot be measured directly
7. reaction of alkanes
8. reaction of alkenes
9. experiment to find the effect of electrostatic force on jets of liquid
10. solubility of simple molecules in different solvents
11. thermal decomposition of group 2 nitrates and carbonates
12. flame tests on compounds of group 1 and 2
13. simple acid-base titrations
14. oxidation of metal and non-metallic elements and ions by halogens
15. disproportion reactions with cold and hot alkali
16. iodine/thiosulfate titration and the determination of purity of potassium iodate(V)
17. reactions between halogens and halide ions/some reactions of the halides
18. factors that influence the rate of chemical reactions
19. effect of temperature, pressure and concentrations on equilibrium
20. reactions of alcohols
21. preparation of organic liquid (reflux and distillation)
22. preparation of a halogenoalkane from an alcohol
23. reactions of the halogenoalkanes.