Required Practical 1 - Titrations

- This technique can be used to find:

  Concentration **Mr** **Formula** **Water of crystalisation**

- To do this you react a certain volume of a solution with an unknown concentration with a solution of **known concentration**.
- The concentration of the known solution must be accurate and is known as a **standard solution.**

Making a standard solution – Making 250cm^3 of a 0.1 mol dm^{-3} solution of NaOH

- Weigh a known mass (number of moles) out in a weighing boat recording its mass to the number of decimal places on the balance.

n = C x V (dm^3) (250/1000 = 0.25)

n = 0.1 x 0.25

n = 0.025 moles

m = n x Mr

m = 0.025 x 40

m = 1.00g

- Transfer to a beaker and reweigh the weighing boat (as there may be some left in the weighing boat). The difference is the **precise** mass added to a beaker:

Mass of weighing boat + calculated mass NaOH	2.62g
Mass of weighing boat	1.63g
Mass of NaOH dissolved	**0.99g**

- Dissolve in 100cm^3 of distilled water and stir with a glass rod.
- Using a funnel, pour into a volumetric flask.
- Use the wash bottle to wash beaker, funnel and glass rod into the volumetric flask.
- Fill the volumetric flask with distilled water so the meniscus sits on the line.
- Stopper the flask and invert several times to ensure mixing.
- Now calculate the **exact concentration**:

$n = \frac{m}{Mr}$

$n = \frac{0.99}{40}$

n = 0.02475 moles

$C = \frac{n}{V}$

$C = \frac{0.02475}{0.25}$

C = 0.099 mol dm^{-3}

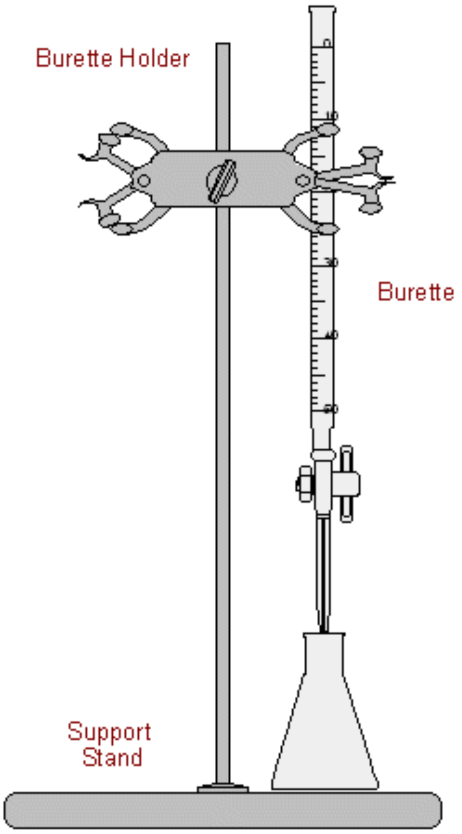
Carrying out a titration:

- Using moles and reacting ratios, you can calculate the concentration of a solution.
- The unknown goes in the conical flask and the known goes in the burette
- The only requirement is that you can tell when one solution has completely reacted with the other.
- Between acids and alkalis, we use indicators to let us know when the resulting solution is neutral.
- An indicator will change colour at the 'end point' (neutral).
- Common indicators are:

Indicator	Acidic colour	Base colour	End point colour
Methyl orange	Red	Yellow	Orange
Phenylphthalein	colourless	Pink	Pale pink

Technique/procedure

Example – finding an unknown concentration of NaOH using 0.10 moldm^{-3} H_2SO_4



1) Rinse the burette with sulphuric acid, H_2SO_4.
2) Fill the burette to the graduation mark ensuring the air is removed from the tap.
3) Rinse a pipette with sodium hydroxide, NaOH fill and transfer 25 cm^3 to a clean, dry conical flask.
4) Add 2-3 drops of indicator.
5) Run the acid into the alkali and stop when the colour changes. This is your **'trial'**.
6) Record the burette readings to 2dp ending 0 / 5
7) Repeat the titration until you get **2 concordant results**
8) Calculate the mean titre to 2dp.

Record results in a table like the one below:

	Trial	1	2	3
Final burette reading /cm^3				
Initial burette reading /cm^3				
Titre /cm^3				
Mean Titre 2dp /cm^3				

A) Aqueous solution / mole calculation – example

In a titration 0.01M sulphuric acid was added to 25cm^3 of sodium hydroxide. Calculate the concentration of the sodium hydroxide given the following results:

	Trial	1	2
Final burette reading /cm^3	**22.3**	**21.8**	**21.7**
Initial burette reading /cm^3	**0.00**	**0.00**	**0.00**
Titre /cm^3	**22.3**	**21.8**	**21.7**
Mean Titre 2dp /cm^3	**21.75**		

1 Write a balanced equation

$H_2SO_{4(aq)} \quad + \quad 2NaOH_{(aq)} \quad \rightarrow \quad Na_2SO_{4(aq)} \quad + \quad H_2O_{(l)}$

2 Calculate the number of moles of H_2SO_4 added from the burette

n of H_2SO_4 = C x V

n of H_2SO_4 = 0.01 x 0.02175

n of H_2SO_4 = 2.175 x 10^{-4}

3 Use the ratio to work out the number of moles of NaOH in the conical flask

H_2SO_4 : NaOH 1 : 2

n of NaOH = 2.175 x 10^{-4} x 2

n of NaOH = 4.35 x 10^{-4}

4 Calculate the concentration of NaOH

$$C = \frac{4.35 \times 10^{-4}}{0.025}$$

C = 0.0174 moldm^{-3}

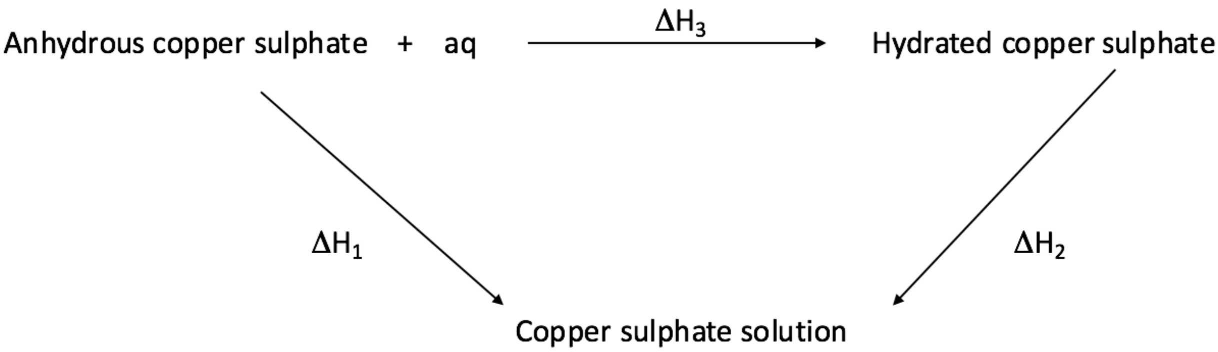
<u>TIP:</u>

Mass, gas and aqueous solution formulas may be used in a combination of ways in these reacting mole calculations

The format remains the same – a starting point – an end point, in the balanced equation

Required Practical 2 – Measurement of an enthalpy change

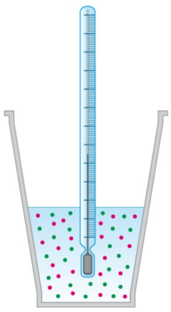
- Hess's Law can be used to find an enthalpy change that cannot be measured directly:

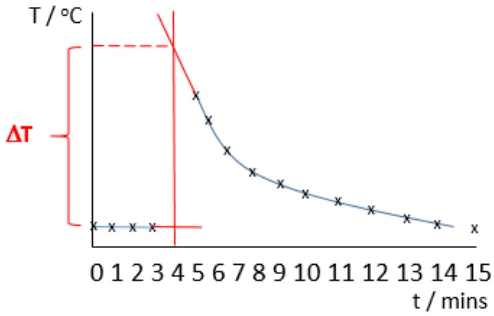


$$\Delta H_3 = \Delta H_1 - \Delta H_2$$

Measuring ΔH_1 and ΔH_2 directly:

1) Weigh 3.90 – 4.10 of anhydrous copper sulphate in a dry stoppered weighing bottle.
2) Place 25cm^3 of water into a polystyrene cup and record its initial temperature, t=0





3) Record the temperature for 3 minutes.
4) On the 4th minute add the anhydrous copper sulphate but do not record the temperature.
5) Stir continuously.
6) 5 – 15 minutes the temperature is recorded.
7) Re weigh the stoppered flask to determine the actually mass of anhydrous copper sulphate added.
8) Plot a graph (as shown above)
9) The points are extrapolated to the 4 minute mark.
10) ΔT is determined from the graph by reading off the Y axis.

Repeat the experiment for hydrated copper sulphate except:

1) Use between 6.20 – 6.30g of hydrated copper sulphate
2) Use 24cm^3 of water. The waters of crystallisation will make the volume up to 25cm^3

Recording results:

	ΔH_1 Mass (g) 2dp	ΔH_2 Mass (g) 2dp
Mass of sample and weighing bottle		
Mass of weighing bottle		
Mass of sample		

Time (mins)	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
ΔH_1 Temp (°C)															
ΔH_2 Temp (°C)															

Calculation

Step 1: The energy change, q:

$$q = m \quad c \quad \Delta T$$

- As **enthalpy** is in **Kj** mol^{-1} and **q** is in j, convert the energy calculated by dividing by 1000).

Step 2: Calculate the number of moles used:

$$\text{Moles} = \frac{\text{Mass}}{\text{Mr}} \quad \text{or} \quad C \times V(dm^3)$$

Step 3: Calculate the enthalpy, ΔH:

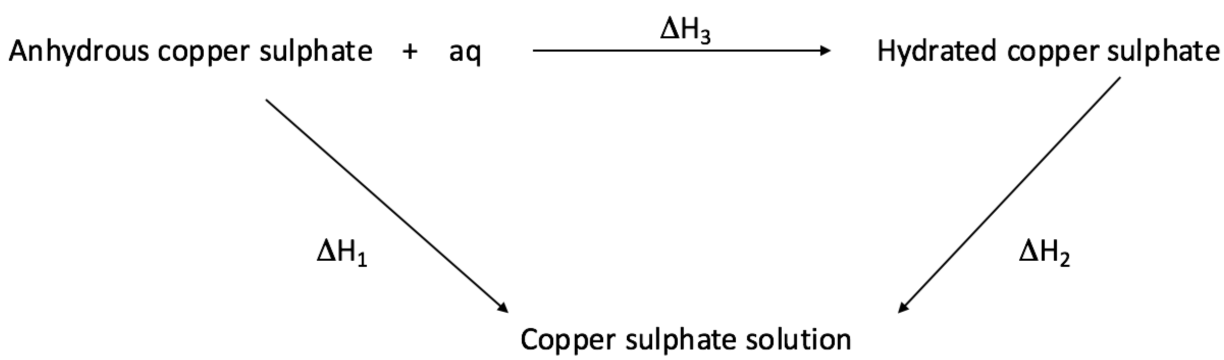
$$\text{Enthalpy, } \Delta H = \frac{\text{Energy, q}}{\text{Moles}}$$

Step 4: Check you have the sign correct:

(-)ve for exothermic reactions

(+)ve for endothermic reactions

Use your answers to ΔH_1 and ΔH_2 to calculate ΔH_3 using Hess's law



$$\Delta H_3 = \Delta H_1 - \Delta H_2$$

Required Practical 3

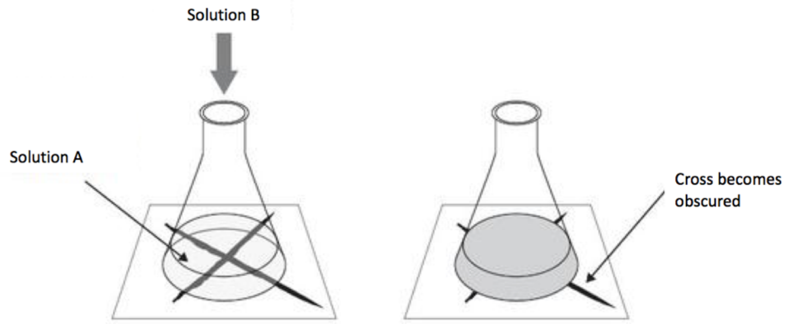
Measuring reaction rates:

1) Precipitates

- Measure the time taken for a precipitate to obscure a cross

$$Na_2S_2O_{3(aq)} + 2HCl_{(aq)} \rightarrow 2NaCl_{(aq)} + S_{(s)} + SO_{2(aq)} + H_2O_{(l)}$$

- Solid sulphur is made which will change the appearance:



Clear and colourless → Yellow solid

- As you cannot see through the yellow solid, a specific amount of sulphur will be made that will obscure a cross underneath on a piece of paper.
- This can be used to investigate the effect of temperature or concentration:

Temperature:		**Concentration:**	
Independent variable:	Temperature	Independent variable:	Concentration
Dependent variable:	Time	Dependent variable:	Time
Control variables:	Concentrations, pressure	Control variables:	Temperature, pressure

Method

- Place 10 cm^3 of 1.0 mol dm^{-3} hydrochloric acid into a boiling tube, place in a water bath until the desired temperature.
- Place 10 cm^3 of 0.1 mol dm^{-3} sodium thiosulphate into a comical flask, place in a water bath until the desired temperature.
- Record the initial temperature then add the hydrochloric acid to the sodium thiosulphate starting the stop clock.
- When the cross is no longer visible, stop the clock and record the final temperature of the reaction mixture.
- Repeat the experiment at 5 different temperatures

Results

Initial temperature / °C	Final temperature / °C	Average temperature / °C	Time for cross to disappear / s	Rate (1/t) / s^{-1}
14	14		115	
21	19		75	
23	23		65	
29	29		48	
41	39		26	
51	49		15	

Analysis

- Plot a graph of average temperature vs rate (1/t)
- Axes **must** be labelled with units
- Mark all points with a x
- You **must** use more than half of the graph paper provided

Analysis questions

- What is the independent variable?

- What is the dependent variable?

- What is the relationship between rate and temperature? Explain your answer using collision theory:

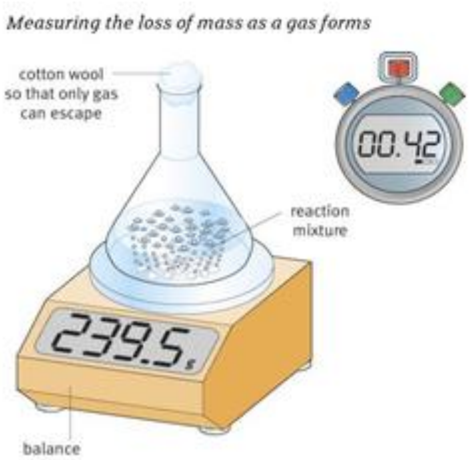
- Sketch a **labelled** Maxwell Boltzmann distribution curve showing how an increase in temperature affect the rate of a reaction:

- Use your Maxwell Boltzmann distribution curves above to explain the effect of temperature on rate:

2) Change in mass

- Record the time as a reaction that loses mass proceeds:

$$Na_2CO_{3(aq)} + 2HCl_{(aq)} \rightarrow 2NaCl_{(aq)} + CO_{2(g)} + H_2O_{(l)}$$



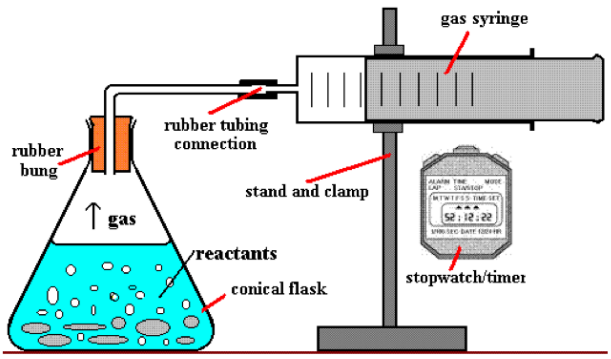
- Carbon dioxide is made which escapes the reaction mixture.
- This will change the mass on the balance.
- As the reaction proceeds, the concentration of the acid decreases.
- The rate at which the mass is lost will be proportional to the concentration of the acid.
- As the reaction proceeds, the rate therefore will slow down.

- This can be used to investigate the effect of temperature or concentration.

3) Gas volumes

- Record the time as a reaction that loses mass proceeds:

$$Mg_{(s)} + 2HCl_{(aq)} \rightarrow MgCl_{2(aq)} + H_{2(g)}$$



- Carbon dioxide is made which escapes the reaction mixture.
- This can be collected in a gas syringe.
- As the reaction proceeds, the concentration of the acid decreases.
- The rate at which the gas is collected will be proportional to the concentration of the acid.
- As the reaction proceeds, the rate therefore will slow down.

- This can be used to investigate the effect of temperature or concentration:

Questions:

1) The Maxwell Boltzmann distribution shows the distribution of energies of molecules in a gas.
   a. In the space below sketch the Maxwell Boltzmann distribution. Add the activation energy and clearly label the axis.

   b. Outline 2 of the key features of the Maxwell Boltzmann distribution curve:

   c. Draw a second Maxwell Boltzmann distribution curve at a higher temperature. **Use the completed diagram** above to explain why an increase in temperature increases the rate of reaction:

2) The production of ammonia is an important industrial process. The reaction is shown below:

$$N_{2(g)} \quad + \quad 3H_{2(g)} \quad \rightarrow \quad 2NH_{3(g)} \qquad \Delta H = -92.0 \text{ kj mol}^{-1}$$

   a. In the space below sketch the enthalpy profile diagram. Add and clearly label the **axis**, the **activation energy** and the **enthalpy change**.

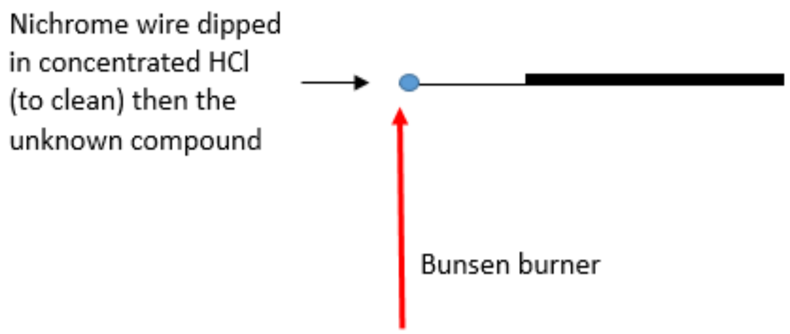
   b. The reaction is catalysed using iron, Fe. Add the profile for this reaction, clearly labelling the activation energy when a catalyst is used

   c. The rate of reaction is also increased using pressure. Explain using collision theory how pressure increases the rate of a reaction

Required Practical 4 – Test for ions

Positive ions:

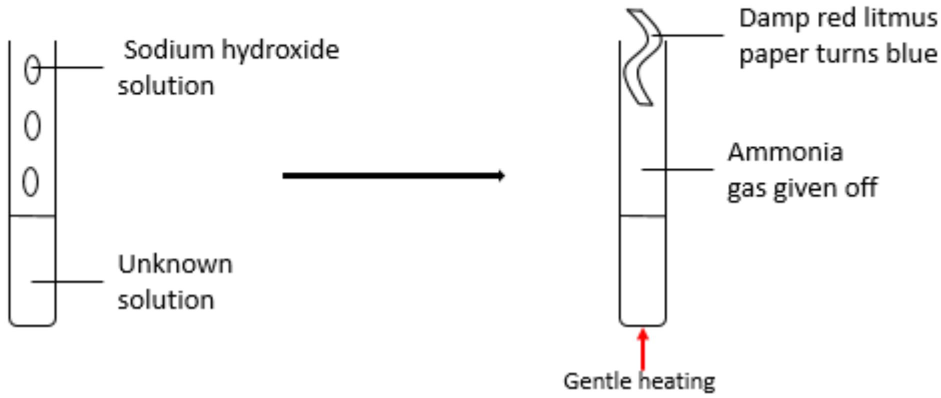
1) Test for Group 2 metal ions, M^{2+} - Flame tests



Metal ion	*Flame colour*
Calcium, Ca^{2+}	**Brick red**
Strontium, Sr^{2+}	**Red**
Barium, Ba^{2+}	**Pale green**

2) Test for ammonium ions, NH_4^+:

$$NH_4^+{}_{(aq)} + OH^-{}_{(aq)} \rightarrow NH_{3(g)} + H_2O_{(l)}$$

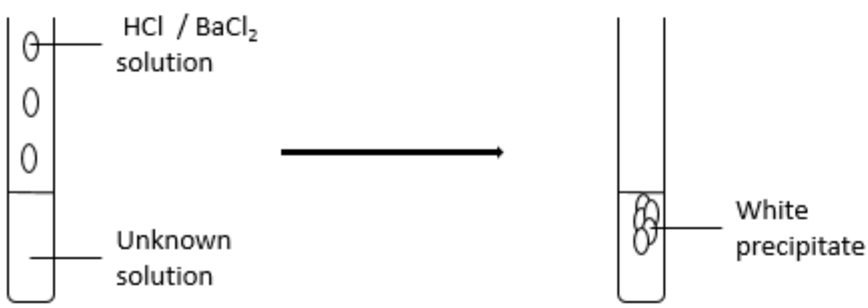


- With sodium hydroxide and heat, ammonia gas is given off.
- Litmus paper must be damp for ammonia gas to dissolve in.
- **Turns Blue**

Negative ions:

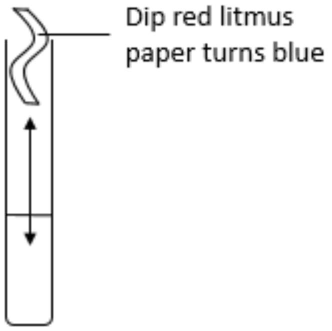
1) Test for sulphate ions, SO_4^{2-}

$$Ba^{2+}{}_{(aq)} + SO_4^{2-}{}_{(aq)} \rightarrow BaSO_{4(s)}$$



- Add **HCl** first – This removes any sulphites or carbonates that may also give a white precipitate.
- Add **$BaCl_2$** solution: White **precipitate of $BaSO_4$** will form.

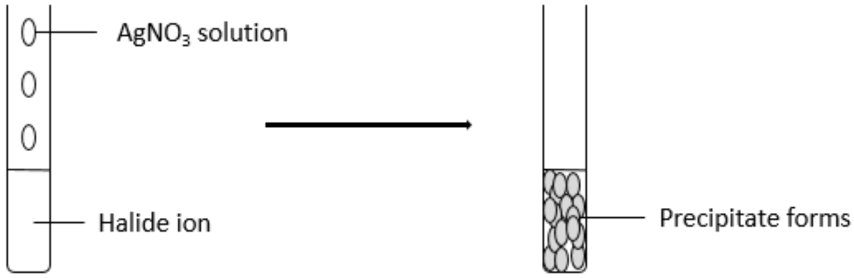
2) Test for hydroxide ions, OH^-



- Dip red litmus paper onto the solution.
- If it turns blue, hydroxide, OH^- are present.

3) Testing for Halide ions, X^-

$$X^-_{(aq)} \quad + \quad Ag^+_{(aq)} \quad \rightarrow \quad AgX_{(s)}$$



- Add **nitric acid** to **remove any other ions** that may interfere with the test.
- Add silver nitrate, **$AgNO_3$.**
- The silver ions, Ag^+ combines with the Halide ions, X^- to **form a silver halide precipitate**

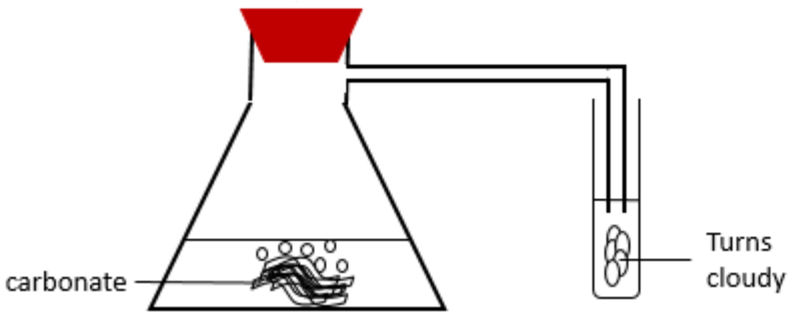
- **Ammonia can be added** as the different silver halides as they have different solubility's in ammonia**.**

Results:

Halide ion	***Observations***	***With Ammonia***	***Solubility of the precipitate***
Cl^-	White precipitate	Dissolves in dilute NH_3 solution	Most soluble
Br^-	Cream precipitate	Dissolves in concentrated NH_3 solution	
I^-	Yellow precipitate	Insoluble	Least soluble

4) Testing for carbonate ions, CO_3^{2-}

$$CO_3^{2-}{}_{(aq)} \quad + \quad 2H^+_{(aq)} \quad \rightarrow \quad CO_{2(g)} \quad + \quad H_2O_{(l)}$$

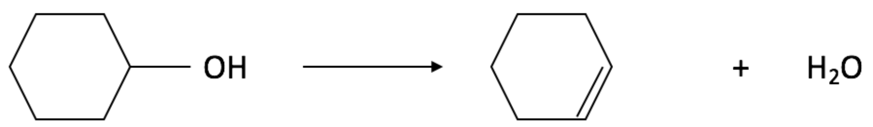


- Add **hydrochloric acid**.
- It will fizz if carbonate present, **CO_3^{2-}**.
- **CO_2** gas is made.
- **CO_2** will turn **limewater cloudy**.

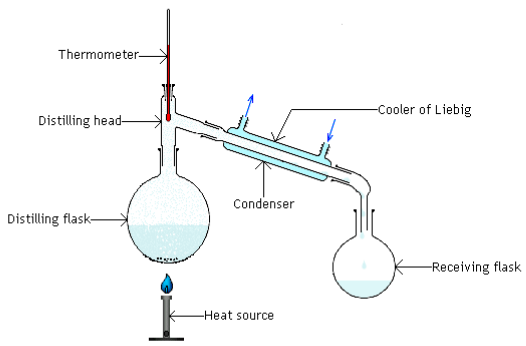
Required Practical 5 – Purification of a product

Dehydration of an alcohol

$C_6H_{11}OH$ → C_6H_{10} + H_2O



1) Heat and 1st distillation



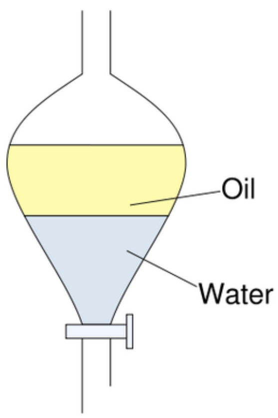
- The heat for the reaction is provided from a distillation process.
- The reason that distillation is used as the source of heat is that the product can be distilled off as it forms.
- The product is usually in a water / acid / reactant mixture.

Intermolecular forces of the reaction mixture

Cyclohexanol	Water	Sulphuric acid	Cyclohexene
H - Bonding	H - Bonding	H - Bonding	Van Der Waals
Strong – High boiling point	Strong – High boiling point	Strong – High boiling point	Weak – Low boiling point

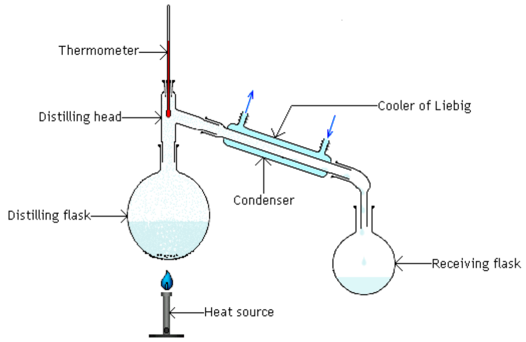
- The alkene only has the weakest VDW forces of attraction it will evaporate 1st
- This allows the crude alkene to be collected as it is formed at its boiling point – 83°C

2) Separation



- The crude cyclohexene will contain water soluble impurities that need separating from the alkene.
- The cyclohexene is transferred to a separating funnel.
- Water is added to the separating funnel and shaken to remove water soluble impurities from the cyclohexene and transfer them to the water.
- Allow to settle. The cyclohexene (hydrocarbon) is less dense than water and does not mix due to their different intermolecular forces.
- Run off the aqueous layer (waste)

3) Purification – 2nd distillation



- Pour the cyclohexene into a round bottom flask.
- Add $CaCl_2$ – a drying agent. This removes any droplets of water trapped in the cyclohexene, allow 20 minutes for this.
- Any other (hydrocarbon based) impurities are removed from a 2nd distillation.
- Only the liquid around cyclohexene's boiling point is collected.
- This is the pure cyclohexene.

Required Practical 6 – Tests for functional groups

The following tests were carried out and the results recorded below:

Organic compound	Na added	Br_2 water added	Tollens' reagent	$K_2Cr_2O_7$ / H_2SO_4	Na_2CO_3 added	NaOH / HNO_3 / $AgNO_3$
A	-	-	*Silver mirror*	*Orange → green*	-	-
B	-	*Orange → colourless*	-	-	-	-
C	*Fizzing*	-	-	-	-	-
D	*Fizzing*	-	-	-	*Fizzing*	-
E	-	-	-	-	-	-
F	*Fizzing*	-	-	*Orange → green*	-	-
G	-	-	-	-	-	*White precipitate*

Compounds A – F are one of the following: Add the letter next to the compound

Carboxylic acid	☐	**Ketone**	☐	**Alkene**	☐	**Halogenoalkane**	☐
Aldehyde	☐	**1° Alcohol**	☐	**3° Alcohol**	☐		

Questions:

1) 2 organic compounds that are functional group isomers of each other have the molecular formula C_3H_6O. When Tollen's reagent was added, only one of them gave a silver precipitate. Identify and draw the structure of the 2 organic compounds. Explain your answer.

2) 3 organic liquids A,B and C are unknown. Upon chemical analysis the following results were obtained:

A: Turned $K_2Cr_2O_7$ / H_2SO_4 orange → green and fizzed with Na
B: Turned $K_2Cr_2O_7$ / H_2SO_4 orange → green and produced a silver precipitate with Tollens' reagent
C: No change with $K_2Cr_2O_7$ / H_2SO_4. Fizzed with Na_2CO_3

Identify with reasons the functional groups present in A,B and C

3) 2 organic liquids X and Y are unknown. Upon chemical analysis the following results were obtained:

A: Did not fizz with Na but turned $K_2Cr_2O_7$ / H_2SO_4 orange → green
B: Turned bromine water orange → colourless **and** fizzed with Na_2CO_3

Identify with reasons the functional groups present in X and Y

4) An organic compound was found to have the following composition by mass.
Its mass spectra had a molecular ion peak of 74.

	C	H	O
% by mass	64.86	13.51	21.62

a. Calculate the molecular formula of the organic compound.

b. The organic compound was found to fizz when Na was added. What functional group must the compound have?

c. Draw the 4 possible isomers that include the functional group identified in (b) and classify them.

d. The organic compound was then refluxed with $K_2Cr_2O_7$ / H_2SO_4 and the oxidising mixture turned orange → green.
What does the colour change tell you about the organic compound?

e. The oxidation product was tested with Na_2CO_3 and there was no fizzing observed. What functional group is in the oxidation product? Draw and name the oxidation product.

f. What does (e) tell you about the structure of the original organic compound? Draw and name the original organic compound.