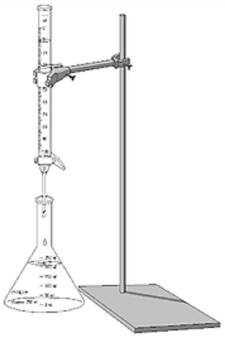
Beatty Secondary School

Secondary 4 Express

Name: ________________________ (   ) Class: 4E1 Date: ________

Practical Revision: Volumetric Analysis (Titration)



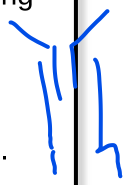
Key Concepts:

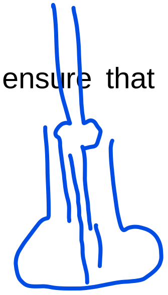
Titrate solution Q with solution P / Titrate solution Q against solution P

solution P in the burette

A. General Tips

1. Rinse the funnel and burette with deionised water before the start of the titration.
2. Check for air bubbles at the tip of the burette / leaking of solution before rinsing with chemicals.
3. **Remove the filter funnel** before the start of titration.
4. Rinse the pipette with deionised water before placing into the reagent bottle.
5. Check if there is liquid in the pipette filler. Deflate it completely to ensure no liquid is trapped inside.
6. Tap the tip of the pipette 3 times into the conical flask to ensure that the maximum volume of solution is pipetted.





B. Presentation of Data and Measurement

Measurement and Accuracy of Data:

apparatus	accuracy
burette	**0.05 cm^3 (2 decimal places)**
pipette	**25.0 cm^3 (1 decimal place)**

Presentation of Data (Titre volumes should be consistent within ± 0.20 cm^3):

~~±0.3~~

titration number	rough	1	2	3
final burette reading / cm^3	25.10	28.60	24.60	49.20
initial burette reading / cm^3	0.00	4.30	0.00	24.60
volume of liquid Q added / cm^3	25.10	24.30	24.60	24.60
best titration results			✓	✓

$\frac{x+y+z}{3} = 24.1333$

Results:

Average volume of Q used = $\frac{24.60 + 24.60}{2}$ = <u>24.60 cm^3</u> (2 decimal places)

$= 24.13\ cm^3$

2 d.p.

Volume of P used = 25.0 cm^3

Key Concepts:

C. Types of Indicators and Colour Changes

indicators	acidic	end-point	alkaline
methyl orange	red	orange	yellow
screened methyl orange	purple	grey	green
thymolphthalein	colourless	colourless	blue

D. Calculations involving titration

1. Percentage composition by mass

$$\%\text{ by mass of an element} = \frac{\text{Number of atoms of element} \times A_r \text{ of element}}{M_r \text{ of compound}} \times 100\%$$

2. Percentage yield determines the percentage of product formed [mole of reactant].

$$\%\text{ yield} = \frac{\text{experimental or actual yield}}{\text{theoretical yield}} \times 100\%$$

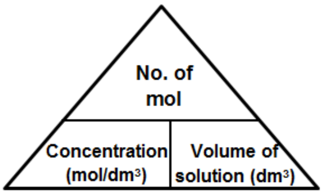
3. Percentage purity determines how much pure substance is found within the sample [mole of product].

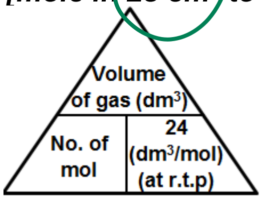
$$\%\text{ purity} = \frac{\text{mass of pure substance}}{\text{mass of sample}} \times 100\%$$

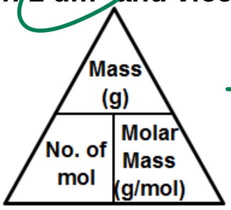
4. Chemical Calculations Formulae

Note calculations on dilution [mole in 25 cm^3 to mole in 1 dm^3 and vice-versa]

$\rightarrow 1000cm^3$







$\frac{mol}{25} \times 1000cm^3$

E. Sources of Error, Effects and Improvement

1. Source of Experimental Error:

There may be presence of other soluble impurities in the acid solution pipetted into the conical flask which may react with the alkali from the burette.

Effect: The volume of solution used in the burette may be larger than expected.

~~affect~~

Or

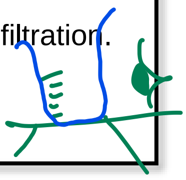
direction

There may be presence of other soluble impurities in the acid solution pipetted into the conical flask which may react with the acid itself.

Effect: The volume of solution used in the burette may be smaller than expected.

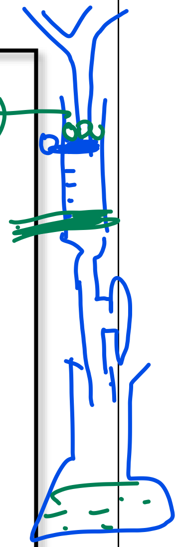
Improvement: Remove the soluble impurities by the precipitation followed by filtration.

Note: Parallax Error is not a source of experimental error.
It is considered as a human error.



F. Rationale behind experimental procedures

1. General Titrations (Acid-Base / Acid-Carbonate)



Q(a) The filter funnel was left on top of the burette during the course of titration. Explain the effect on the titration results.

A(a) Excess solution from the funnel may be added to the burette. The volume of solution added to the conical flask may be larger than expected / The volume of solution recorded in the burette will be smaller than expected volume used.

Improvement: Remove the filter funnel and repeat the titration several times to values ± 0.20 cm^3.

$26 - (-1)$

$= 27$ cm^3

Q(b) A student suggested that the investigation could be improved by making the titrations more accurate. He said that the concentrations of solution in the burette should be reduced. State and explain whether or not this suggestion would make the titrations more accurate.

A(b) The titration results would be more accurate since the titre volume is bigger. Hence there will be a smaller percentage error.

5 , 5.1 cm^3

$\frac{0.1}{5}$

25.1 cm^3 25.0

$\frac{0.1}{25}$

2. Redox Titrations

Q(a) In a titration involving aqueous iron(II) sulfate against potassium manganate(VII), 25 cm^3 of sulfuric acid was added to the conical flask. Explain why sulfuric acid was added and why the volume of acid does not need to be measured accurately.

A(a) The purpose of the acid is to remove soluble carbonate impurities present, hence accurate volumes of the acid added is not necessary.

Q(b) Dilute sulfuric acid is added to acidify aqueous potassium manganate(VII) as it is stable to oxidation and provides hydrogen ions as a catalyst to speed up the reaction. Suggest why dilute hydrochloric acid is **not** used in the reaction.

A(b) Aqueous potassium manganate(VII) is a strong oxidising agent and will oxidise hydrochloric acid to liberate chlorine gas, causing a side reaction.

3. Iodometric Titrations (sodium thiosulfate with potassium iodide/iodate)

*starch

Q(a) Suggest why starch solution is added as an indicator.

A(a) The resulting mixture is a pale yellow solution which is difficult to determine the end-point visually. Starch solution detects any free iodine and reacts with iodine to form a complex to give a dark blue colour. The colour change from blue to colourless is more visible and gives a sharp end-point.

Q(b) Suggest why the starch indicator is **not** added at the start of the titration.

A(b) The starch-iodine complex formed at high iodine concentrations is relatively stable, which is difficult to decompose. The correct titre volume will be higher than expected volume used when starch indicator was added at the start. ↑

Practical Revision: Thermochemistry (Energy Changes)

Key Concepts:

A. Presentation of Data and Measurement

Measurement and Accuracy of Data:

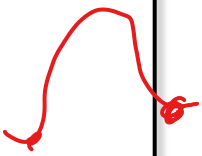
apparatus	accuracy
thermometer	**0.5 °C (1 decimal place)**
measuring cylinder	**0.5 cm^3 (1 decimal place)**
electronic balance	**0.01 g (2 decimal places)**

25.0°C
25.5°C
26.0°C

Presentation of Data:

initial temperature measured / °C	**highest** temperature reached / °C	total temperature change / °C
28.0	28.0	0.0
28.0	29.0	+1.0
28.0	31.0	+3.0
28.0	32.0	+4.0
28.0	33.5	+4.5
28.0	34.5	+6.5
28.0	35.0	+7.0
28.0	34.5	+6.5
28.0	34.0	+6.0

~~final~~ ~~initial~~



maximum

minimum

B. Sources of Error, Effects and Improvement

BI. Thermometric Titration (measuring heat change involving burette and styrofoam cup)

1. Source of Experimental Error: There may be <u>heat loss to the surroundings</u> (for exothermic reaction) / <u>heat gained from the surroundings</u> (for endothermic reaction)

   Effect: The temperature measured will be <u>lower than expected (exothermic) / higher than expected (endothermic)</u>.

   Improvement to <u>minimise heat loss</u>: Use a styrofoam cup covered with lid to reduce heat loss to the surroundings. Nest the styrofoam cup in a beaker to ensure stability.

   <u>Improvements</u> to <u>improve accuracy:</u> Measure the volume of <u>solution using a more precise burette</u> instead of a measuring cylinder / Use a more <u>precise thermometer</u> e.g. more subdivisions, reading to 0.1 °C / Take the initial temperature of both solutions and calculate the <u>mean of temperature</u> / <u>Repeat</u> the experiment and take the <u>average</u> temperature change

0.1°C

2. Source of Experimental Error: There may be <u>slow run of the solution from burette</u>, leading to <u>more heat loss</u> to surrounding before taking temperature readings.

   Effect: The temperature measured will be <u>lower than expected</u>.

   Improvement: Measure the temperature taken <u>without any delay</u> to minimise the heat loss to the surroundings. <u>Repeat</u> the experiment twice and take the average reading to improve accuracy.

BII. Energy Changes involving displacement reactions

1. Source of Experimental Error: The metal displaced may coat onto the more reactive metal, preventing displacement from continuing.

   Effect: The temperature measured will be lower than expected.

   Improvement: Use a glass rod to stir the reaction mixture to prevent coating of the displaced metal.

BIII. Energy Changes involving metal and acid reactions

1. Source of Experimental Error: There may be metal oxide layer coating the metal that reacts with the acid.

   Effect: The temperature measured will be higher than expected. Metal oxides reacts with acids exothermically to release heat energy, leading to an increase in temperature.

   Improvement: Use a sand paper to remove the oxide layer from the metal.

2. Source of Experimental Error: Larger piece of metal may not react fast with acid, leading to more heat loss to the surroundings due to slow reaction

   Effect: The temperature measured will be lower than expected as heat is loss to the surroundings due to slow reaction.

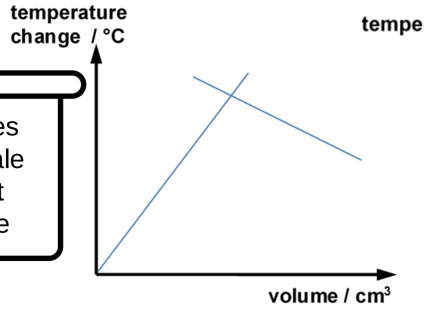
   Improvement: Increase the surface area of the metal by grinding into powdered form.

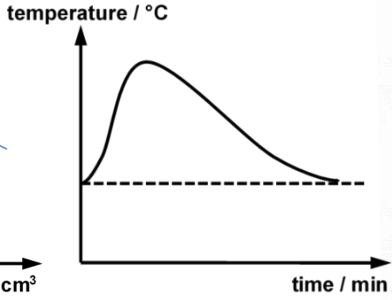
C. Graphical Representation and Explanation

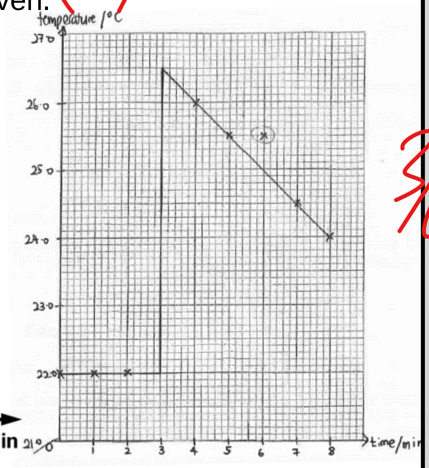
label every 2cm

1. Graph should occupy at least 50% of the graph paper given. **No odd scales!**
2. Draw a line of best fit (can be a curve or straight line).

- **A**xes
- **S**cale
- **P**lot
- **L**ine







3/4

The temperature rises sharply as the concentration of the solutions are the highest at the start of the reaction.

The temperature gradually increases at a decreasing rate as the concentration of the solution decreases. The highest temperature reached indicates the maximum amount of heat given out in the reaction.

The temperature then drops slowly to room temperature as the limiting reactant has been used up and there is no more heat produced.

Practical Revision: Reaction Kinetics (Speed of Reaction)

37·1 s

37 s

Key Concepts:

A. Presentation of Data and Measurement

Measurement and Accuracy of Data:

apparatus	accuracy
stopwatch	**1 s (0 decimal place)**
measuring cylinder	**0.5 cm^3 (1 decimal place)**
electronic balance	0.01 g (2 decimal places)

Presentation of Data:

volume of $Na_2S_2O_3$ added / cm^3	volume of water added / cm^3	volume of HC*l* added / cm^3	time taken, t_1 / s	$\frac{1}{t_1}$ / s^{-1}	total volume of mixture / cm^3
25.0	0.0	5.0	12	0.0730	50.0
20.0	5.0	5.0	16	0.0640	50.0
15.0	10.0	5.0	21	0.0466	50.0
10.0	15.0	5.0	31	0.0294	50.0
5.0	20.0	5.0	62	0.0161	50.0

BI. Speed of Reaction involving precipitation of solid

$$Na_2S_2O_3\,(aq) + 2HCl\,(aq) \rightarrow 2NaCl\,(aq) + SO_2\,(g) + H_2O\,(l) + \mathbf{S\,(s)}$$

1. Source of Experimental Error: The temperature of the reaction is not always constant / fluctuates / temperature change is not the same for each run of mixture.

   Effect: The time taken will be inaccurate as the rate of reaction may not be consistent / rate of reaction is dependent on temperature.

   Improvement: Place the conical flask containing the solution in a water bath to maintain the constant temperature / Use a thermostatically controlled water bath to prevent temperature fluctuations / Take the temperature of the initial mixing and the temperature as soon as the printed insert is obscured and calculate the mean temperature / Take the temperature of both solutions and calculate the mean temperature

2. Source of Experimental Error: The amount of sulfur deposited is not constant / Our perception of when the cross disappears varies from experiment to experiment because the turbidity of the solution decreases with lower concentration / lower temperature.

   Effect: It may lead to some error and inaccuracy to find the time taken for the sulfur to completely obscure / cover the printed insert.

   Improvement: Use a light sensor / colorimeter to avoid subjective judgement of turbidity / Repeat the experiment for a few more times and take the average reading of the time taken to improve the accuracy of the results.

3. Source of Experimental Error: There may be human reaction time due to the delay in the stopping of the stopwatch.

   Effect: The time taken will be longer than expected.

   Improvement: Repeat the experiment for a few more times and take the average reading of the time taken to improve the accuracy of the results.

4. **Human** Error: There is inconsistent swirling of the solution mixture.

   Effect: The time taken will be inaccurate as more swirling will lead to a shorter time taken.

   Improvement: Fix the number of swirling of the solution mixture at the start of the experiment.

BII. Speed of Reaction involving metal and acid reactions

1. Source of Experimental Error: There may be metal oxide layer coating the metal that reacts with the acid.

   Effect: The time taken for the metal to completely react will be longer than expected.

   Improvement: Use a sand paper to remove the oxide layer from the metal.

BIII Speed of Reaction involving decomposition of hydrogen peroxide with the use of catalyst

1 Source of Experimental Error: The delay in connecting the gas syringe leads to loss of some gas.

Effect: The volume of gas collected and recorded will be lower than the actual volume that was produced from the reaction.

Improvement: Use displacement of water with graduated tube.

2 Source of Experimental Error: Friction between the plunger and the syringe leads to erratic spikes in the volume of gas collected caused by the gas compressed in the syringe.

Effect: The volume readings will be inaccurate.

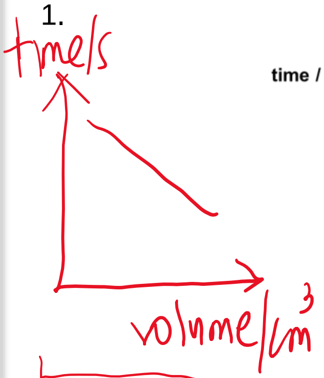
Improvement: Use displacement of water with graduated tube.

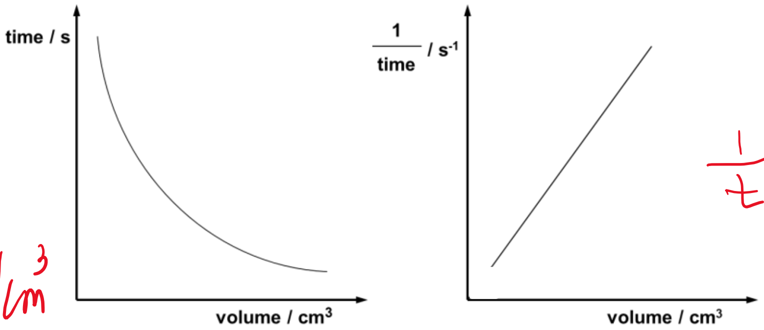
A
S
P
L

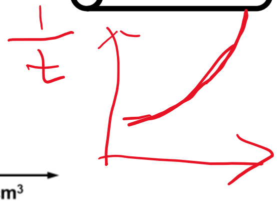
- **A**xes
- **S**cale
- **P**lot
- **L**ine

C. Graphical Representation and Explanation

1.





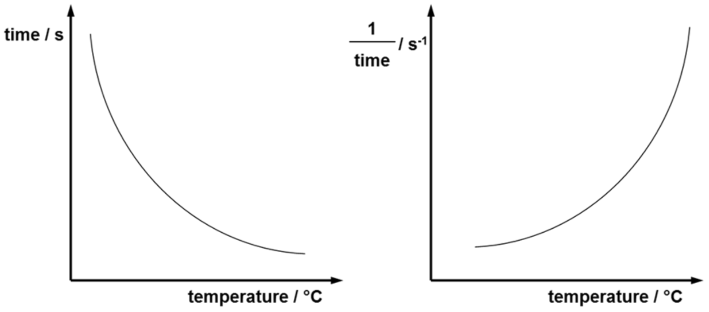


both are possible

As the volume of the solution increases, the concentration of the solution increases. The time taken decreases exponentially with the increase in volume of solution.

$\frac{1}{t_1}$ increases linearly / proportionally with the concentration of the solution.

2.



As the temperature of the solution mixture increases, the time taken for the reaction decreases exponentially.

$\frac{1}{t_1}$ increases exponentially with the increase in the temperature of the solution mixture.

D. Explanation why printed insert is not obscured

Q(a) A student carried out a further experiment to investigate the effect of concentration on the rate of reaction. The student used 10.0 cm^3 of solution P, 15.0 cm^3 of distilled water and 5.0 cm^3 of solution Q. The reaction was carried out at 55.0 °C. The print on the insert never became invisible. Explain why.

A(a) There is not enough / insufficient sulfur precipitate produced to obscure the insert. [concept of limiting reagents]

Practical Revision: Qualitative Analysis (Identification of Ions / Gases)

test	observations	conclusion
(a)		

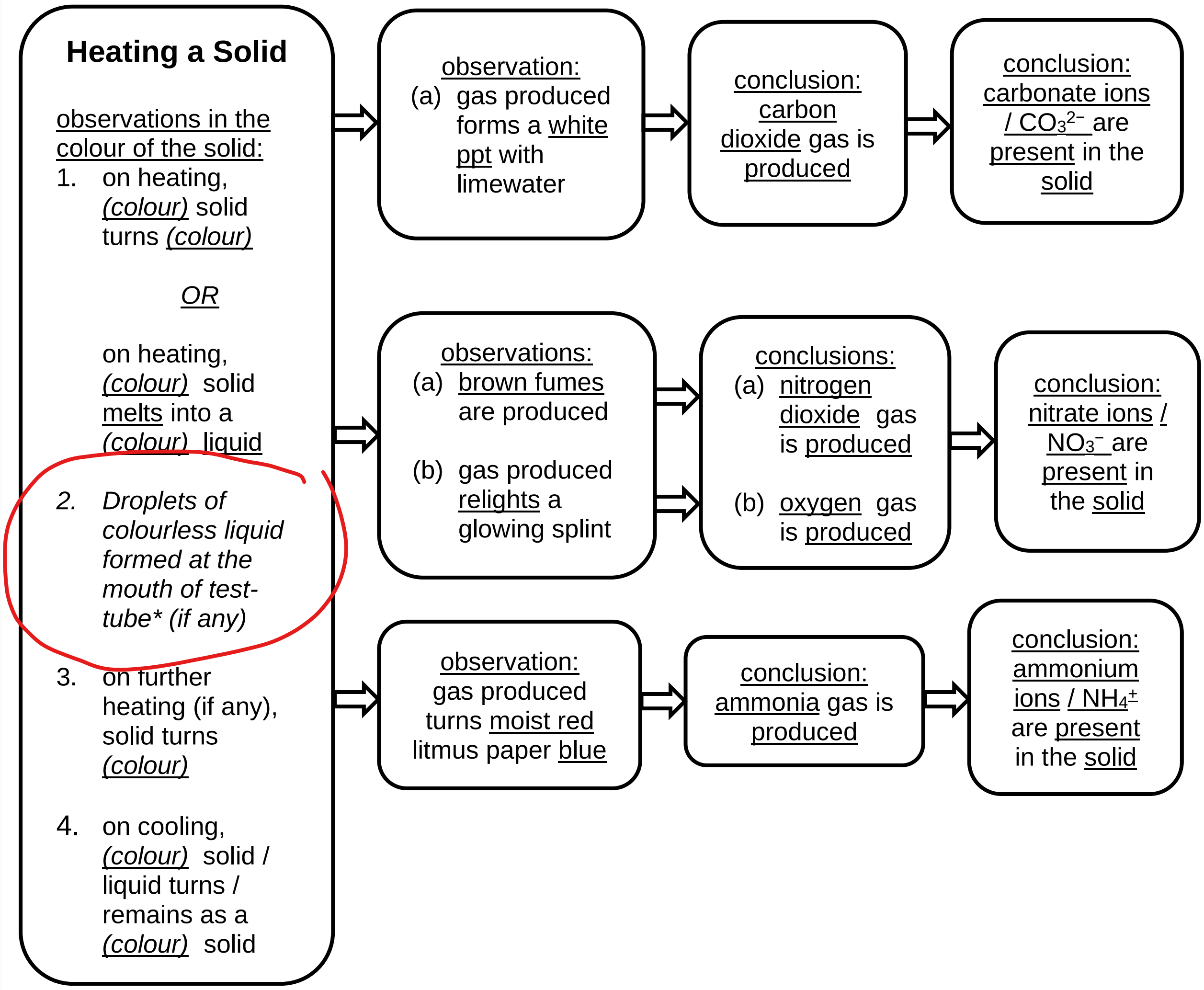
Key Concepts:

A. Flame tests for cations (It burns with a _______ flame / A ______ flame is observed.)

test	observation	identity of cation
Adjust the Bunsen burner to give a blue flame. Moisten the end of a wooden splint with water and dip the moist end of the splint into the sample of solid. Place this end of the splint in the Bunsen burner flame.	It burns with a lilac flame.	potassium ions / K^+ is present
	It burns with an orange flame.	sodium ions / Na^+ is present
	It burns with a brick red flame.	calcium ions / Ca^{2+} is present
	It burns with a pale green flame.	barium ions / Ba^{2+} is present
	It burns with a blue-green flame.	copper(II) ions / Cu^{2+} is present

B. Heating of solids / thermal decomposition of solids (observe for the changes in the colour of the solid and the changes in the physical state upon heating.)

Heat until no further change in colour!



Heating a Solid

observations in the colour of the solid:

1. on heating, *(colour)* solid turns *(colour)*

   OR

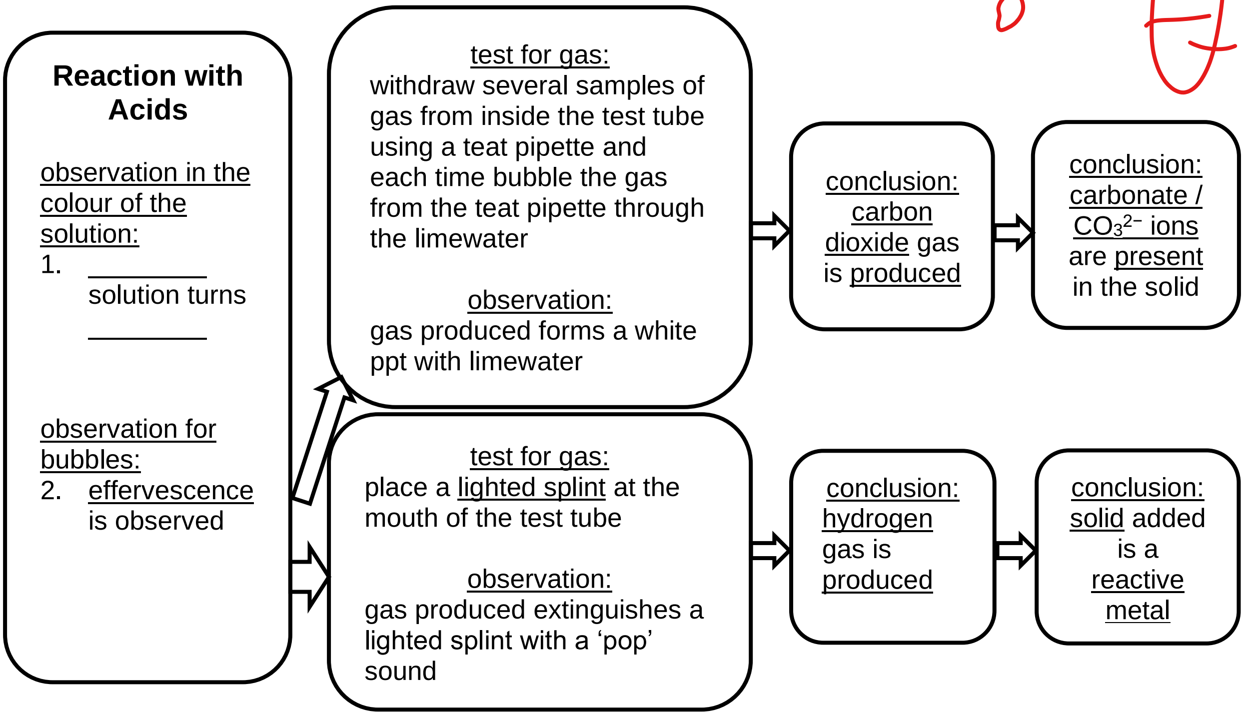
   on heating, *(colour)* solid melts into a *(colour)* liquid
2. *Droplets of colourless liquid formed at the mouth of test-tube* (if any)*
3. on further heating (if any), solid turns *(colour)*
4. on cooling, *(colour)* solid / liquid turns / remains as a *(colour)* solid

observation:
(a) gas produced forms a white ppt with limewater
→ conclusion: carbon dioxide gas is produced
→ conclusion: carbonate ions / CO_3^{2-} are present in the solid

observations:
(a) brown fumes are produced
(b) gas produced relights a glowing splint
→ conclusions:
(a) nitrogen dioxide gas is produced
(b) oxygen gas is produced
→ conclusion: nitrate ions / NO_3^- are present in the solid

observation: gas produced turns moist red litmus paper blue
→ conclusion: ammonia gas is produced
→ conclusion: ammonium ions / NH_4^+ are present in the solid

C. Addition of acids to solids (observe for effervescence)



Reaction with Acids

observation in the colour of the solution:

1. ________ solution turns ________

observation for bubbles:

2. effervescence is observed

test for gas: withdraw several samples of gas from inside the test tube using a teat pipette and each time bubble the gas from the teat pipette through the limewater

observation: gas produced forms a white ppt with limewater

conclusion: carbon dioxide gas is produced

conclusion: carbonate / CO_3^{2-} ions are present in the solid

test for gas: place a lighted splint at the mouth of the test tube

observation: gas produced extinguishes a lighted splint with a 'pop' sound

conclusion: hydrogen gas is produced

conclusion: solid added is a reactive metal

D. Oxidising and reducing agents (observe for the changes in solution colour)

test	observations	conclusion
To 2 cm^3 of acidified potassium manganate(VII), $KMnO_4$, add a few drops of unknown solution.	purple acidified potassium manganate(VII) turns colourless	A reducing agent is present in the unknown solution.
To 2 cm^3 of aqueous acidified iron(II) sulfate, $FeSO_4$, add a few drops of unknown solution.	green solution turns brown / yellow	An oxidising agent is present in the unknown solution.
To 2 cm^3 of aqueous acidified potassium iodide, KI, add a few drops of unknown solution. Add 2 to 4 drops of starch solution / indicator and shake the test tube.	**cream precipitate formed in a brown solution** **blue-black colouration is observed**	An oxidising agent is present in the unknown solution.

Note: hydrogen peroxide can function as an oxidising and/or a reducing agent.

Equation for decomposition: $2H_2O_2 \rightarrow \mathbf{O_2} + 2H_2O$

E. Metal displacement reactions (observe for the changes in solution colour and solid deposit)

test	observations	conclusion
To 2 cm^3 of solution, add 3 pieces of magnesium ribbon to the test tube.	blue solution fades / turns colourless reddish-brown solid / deposit is formed	Magnesium displaces the less reactive metal from the solution.
To 2 cm^3 of solution, add 3 to 4 pieces of granulated zinc to the test tube.	yellow solution fades / turns colourless. grey solid / deposit is formed	Zinc displaces the less reactive metal from the solution.

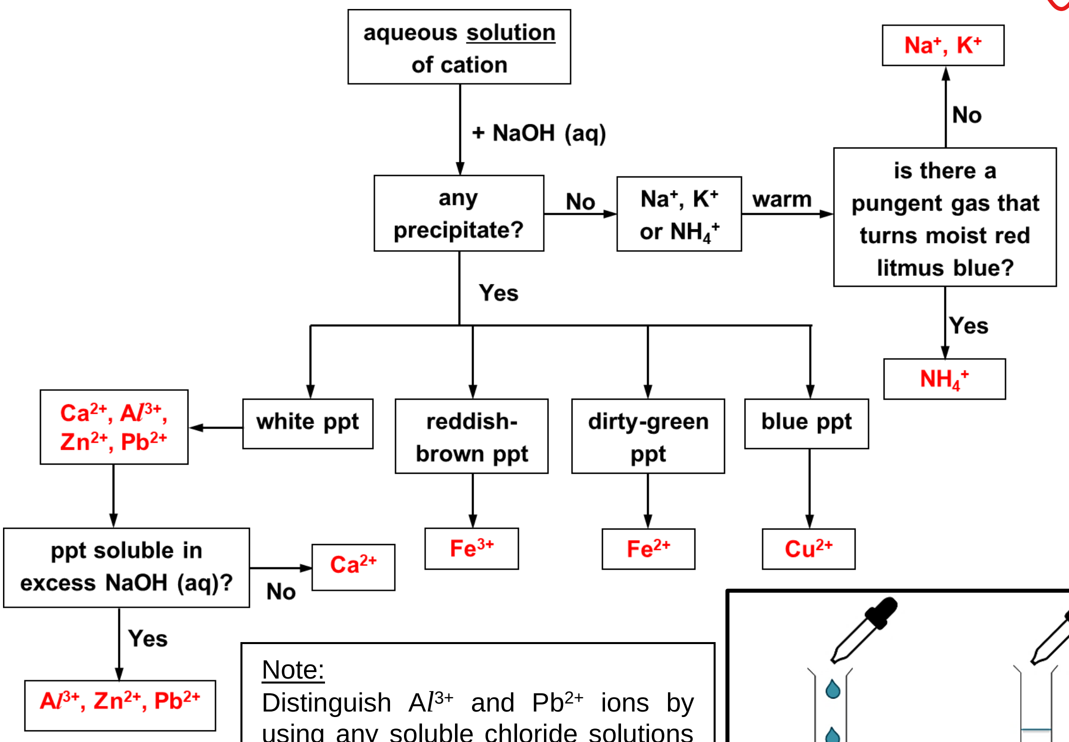
glass rod

Key Concepts:

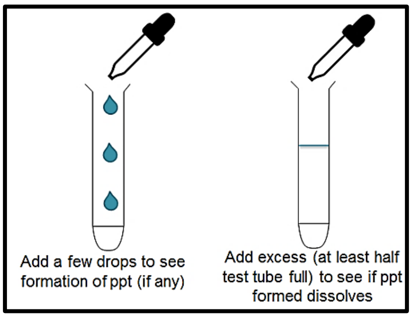
F. Test for cations (Ca^{2+}, Zn^{2+}, Pb^{2+}, Al^{3+}, Cu^{2+}, Fe^{2+}, Fe^{3+}, NH_4^+ ions)

Note: add till no further change = excess

(a) Adding aqueous sodium hydroxide to an unknown solution

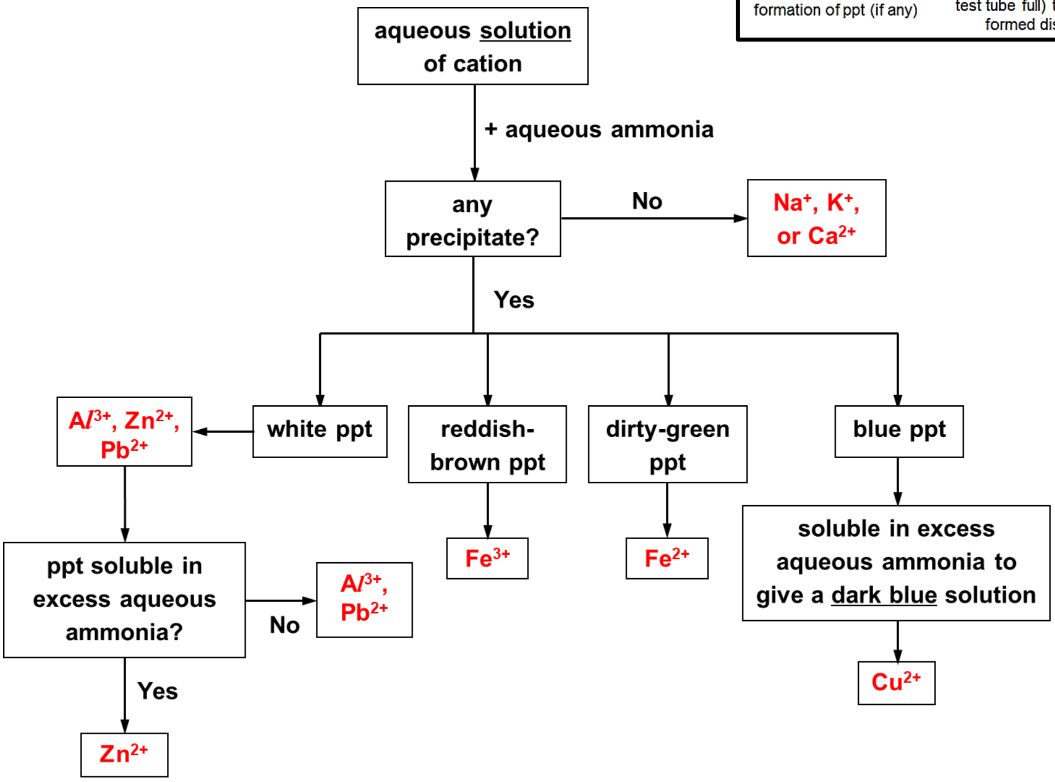


Note:
Distinguish Al^{3+} and Pb^{2+} ions by using any soluble chloride solutions like HCl


Add a few drops to see formation of ppt (if any)

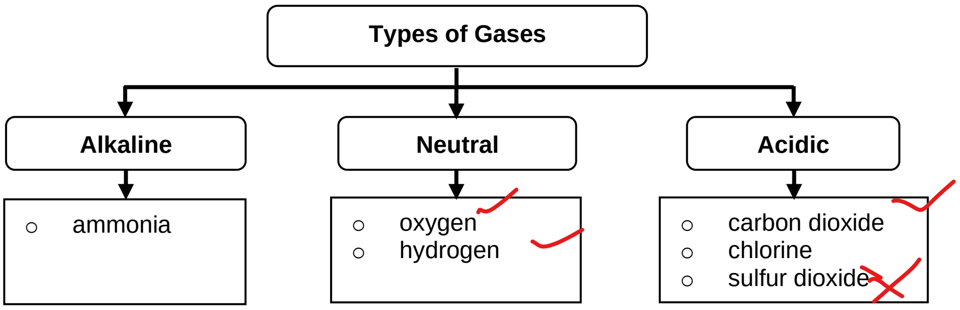
Add excess (at least half test tube full) to see if ppt formed dissolves

(b) Adding aqueous ammonia to an unknown solution



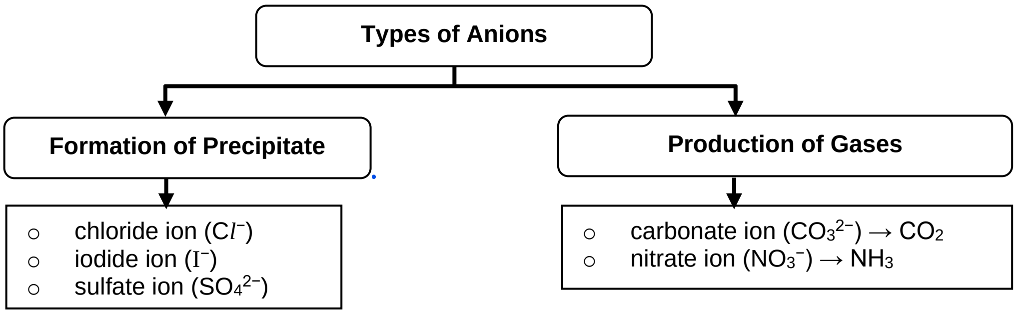
Key Concepts:

G. Test for gases (H_2, O_2, CO_2, SO_2, Cl_2, NH_3)



gas	description of test	observations
ammonia	place a piece of moist red litmus paper at the mouth of the test tube	gas evolved turns moist red litmus paper blue
hydrogen	place a lighted splint at the mouth of the test tube	gas evolved extinguishes a lighted splint with a 'pop' sound
oxygen	insert a glowing splint into the test tube	gas evolved relights / rekindles a glowing splint
carbon dioxide	bubble gas evolved through limewater	gas evolved forms a white precipitate with limewater
chlorine	place a piece of moist blue litmus paper at the mouth of the test tube	gas evolved turns moist blue litmus paper red before bleaches it
sulfur dioxide	place a piece of filter paper soaked with acidified potassium manganate(VII) at the mouth of the test tube	gas evolved turns purple aqueous acidified potassium manganate(VII) colourless

H. Test for anions (Cl^-, I^-, SO_4^{2-}, NO_3^- CO_3^{2-} ions)



anion	description of test	observations / equations
chloride	add dilute nitric acid, then add silver nitrate solution	a white precipitate is formed **Cl^- (aq) + Ag^+ (aq) → $AgCl$ (s)**
iodide	add dilute nitric acid, then add silver nitrate solution	a yellow precipitate is formed **I^- (aq) + Ag^+ (aq) → AgI (s)**
sulfate	add dilute nitric acid, then add barium nitrate solution	a white precipitate is formed **SO_4^{2-} (aq) + Ba^{2+} (aq) → $BaSO_4$ (s)**
carbonate	add dilute acid	effervescence is observed gas evolved forms a white precipitate with limewater **CO_2 (g) + $Ca(OH)_2$ (aq) → $CaCO_3$ (s) + H_2O (l)**
nitrate	add sodium hydroxide solution, then add a piece of aluminium foil before warming the mixture	effervescence is observed gas evolved turns moist red litmus paper blue

Practical Revision: Gravimetric Analysis (Thermal Decomposition of Solids) / Salt Preparation

Key Concepts:

A. Presentation of Data and Measurement

Measurement and Accuracy of Data:

apparatus	accuracy
measuring cylinder	0.5 cm^3 (1 decimal place)
electronic balance	0.01 g (2 decimal places)

Presentation of Data:

Mass of dry sodium chloride with evaporating dish / g	47.15
Mass of empty evaporating dish / g	46.50
Mass of dry sodium chloride / g	0.65

B. Sources of Error, Effects and Improvement

BI. Preparation of Soluble Salts

1. Source of Experimental Error: There is a loss of sodium chloride salt from the evaporating dish due to sputtering.

   Effect: The mass of sodium chloride salt obtained is lower than expected.

   Improvement: Evaporate the solution with a gentler flame.

2. Source of Experimental Error: Sodium chloride salt may absorb moisture from the air / not dried completely.

   Effect: The mass of sodium chloride salt obtained is larger than expected.

BII. Preparation of Insoluble Salts

1. Source of Experimental Error: There may be loss of copper(II) carbonate during filtration or transfer into the container.

   Effect: The mass of copper(II) carbonate salt obtained is lower than expected.

BIII. Gravimetric Analysis / Thermal Decomposition of Solid

1. Water of crystallisation is not completely removed from the hydrated salt.

   Effect: The residue would have a larger mass measured than expected

   Improvement: Heat the solid for a longer period of time until a constant mass is reached.

Colours of Common Substances

General Note:
As a general guide, the colours of most ammonium, Group I and Group II compounds (simple salts like $NaCl$, KOH, $CaSO_4$) are either colourless or white (colourless crystals / white powder).

The aqueous solutions of these compounds are colourless.

Most metals are grey or silver. Copper (reddish-brown / pink) and gold are the two most common exceptions. Most transition metals form coloured compounds.

Substances	Observations / Colours	Remarks
Copper and its compounds		
Copper metal	Reddish-brown / Pink	-
Copper(II) sulfate, $CuSO_4$ solution	Blue	Most copper(II) compounds in the solid or aqueous state are usually blue or green. $CuCl_2$ solution is green when concentrated but blue when dilute
Copper(II) nitrate, $Cu(NO_3)_2$ solution		
Copper(II) chloride, $CuCl_2$ solution		
Copper(II) hydroxide, $Cu(OH)_2$ solid / precipitate		
Copper(II) carbonate, $CuCO_3$ solid	Green	Commonly asked about the thermal decomposition of $CuCO_3$: $CuCO_3 \rightarrow CuO + CO_2$ green black
Copper(II) oxide, CuO solid	Black	
Copper(I) oxide, Cu_2O solid	Reddish-brown / Brick-red	Not commonly asked [Benedict's test for reducing sugars].
Iron and its compounds		
Iron(II) nitrate, $Fe(NO_3)_2$ solution	Green	Most iron(II) compounds in the solid or aqueous state are green.
Iron(II) sulfate, $FeSO_4$ solution		
Iron(II) hydroxide, $Fe(OH)_2$ solid / precipitate		
Iron(II) chloride, $FeCl_2$ solution		
Iron(II) oxide, FeO solid	Black	-
Iron(III) oxide, Fe_2O_3 solid	Reddish-brown	Most iron(III) compounds in the solid state are brown / reddish-brown. Most iron(III) compounds in the aqueous state are yellow / brown.
Iron(III) hydroxide, $Fe(OH)_3$ solid / precipitate	Reddish-brown	
Iron(III) chloride, $FeCl_3$ solution	Yellow / Brown	
Redox Chemistry		
Aqueous Manganate(VII), MnO_4^- ions	Purple	Oxidising Agent Colour change: Purple to Colourless
Aqueous Iodide, I^- ions	Colourless	Reducing Agent Colour change: Colourless to Brown (due to aqueous iodine displaced)
Halogens and halides		
Aqueous chlorine, Cl_2 (aq)	Pale yellow	Commonly asked for observations of colour changes in displacement reactions.
Aqueous bromine, Br_2 (aq)	Reddish-brown / Orange	
Aqueous iodine, I_2 (aq)	Brown	I_2 (s) is black. Some black solid may appear in displacement reactions.
Reaction of metals with steam to form metal oxide and hydrogen		
Magnesium, Mg	Bright white glow	Observed during the reaction. White powder of MgO is formed
Zinc, Zn	Yellow when hot, white when cold	ZnO is yellow when hot, white when cold.
Others		
Carbon powder, C (s)	Black	-
Manganese(IV) oxide, MnO_2 solid [catalyst for decomposition of H_2O_2]	Black	-