H2 Chemistry practical

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§1 Question Types and Answering Techniques

§1.1 Table

Titration results

Mass readings

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§1.2 Calculation

Present full working with units.

§1.3 Graph

Sketching: axes and scale, data points, best-fit line

Interpolation: show construction lines (dotted line, label numerical value), read values up to half of the smallest square

Extrapolation: extend the line (solid line)

Calculate gradient: construct right-angled triangle of size at least half of the page (dotted lines), label coordinates of the two chosen points

§1.4 Sources of error

Sources of error are inherent in an experiment due to the **design** of the experiment. We will need to suggest **improvements** to reduce the sources of error in the experiment.

Remark. Sources of error are not due to the mistakes made by the student conducting the experiment.

Aspects of design which can cause sources of error:

- Apparatus

  There are times when an apparatus is chosen due to the ease of use. Hence, the degree of accuracy may be compromised and this can be a source of error for the experiment.

  Example: A measuring cylinder is used to measure 25 cm³ of a solution instead of a pipette.

  > Source of error: The measuring cylinder used to measure the volume of solution is not as precise as a pipette. It can only measure accurately to 1 cm³.

Improvement: Use a pipette instead of a measuring cylinder to measure the 25.0 cm³ volume.

- Reagents

The stability of the reagent used can be a possible source of error if it decomposes at room temperature, or it reacts with oxygen in the air.

Example: Hydrogen peroxide decomposes slowly into water and oxygen at room temperature even when no catalyst is added. The rate of decomposition increases with light and heat.

Source of error: Since hydrogen peroxide decomposes into water and oxygen at room temperature, there will be a loss of reagent before and during the experiment.

Improvement: Store hydrogen peroxide in a dark glass bottle, away from light and heat.

- Dependent variables

When the dependent variable is temperature change, the source of error can be caused by the inherent inaccuracy in measuring temperature.

Example: Heat is always being lost to the surrounding when the surrounding is cooler compared to the reacting mixture.

Source of error: Heat lost to the surrounding is caused by the poor insulation of the reactants, hence the temperature measured will be lower than expected.

Improvement: Use a polystyrene cup with a lid to minimise heat lost to surroundings.

§1.5 Experiment-specific questions

Refer to below.

§1.6 Safety precaution

Safety hazard	Precaution
H_2 is flammable.	Avoid naked flames.
Alcohols are volatile and toxic.	Containers of alcohols should be covered when not in use.
Short-chain alcohols are flammable and may cause a fire if ignited accidentally.	While the experiment is being conducted, other alcohols should be kept away from the naked flame.
Poisonous gas involved.	Conduct experiment inside fume hood.

§2 Experiments and Set-up (Theoretical Knowledge)

§2.1 Volumetric Analysis

§2.2 Qualitative Analysis

Observations:

1. Colour of precipitate, solubility in excess reagent (**copy from QA notes**)

2. Colour change of solution (**specify initial and final colours**)

3. Test for gas (**copy from QA notes**)

   "**Effervescence**" if gas is insoluble in water, "**evolve**" if gas is soluble in water or no solvent involved.

4. Colour of residue and filtrate (filtration)

For negative results, record observation as the **negative statement of the expected observation**.
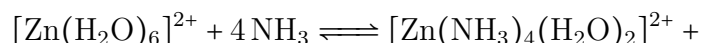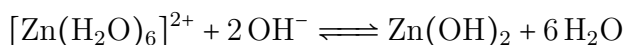
§2.2.1 Test for cations

Refer to QA notes for the complete list.

Al^{3+}, Cr^{3+} and Zn^{2+} ions form **amphoteric** metal hydroxides with OH^-, which dissolve in excess NaOH to form **soluble complex ions**.

$$Al^{3+}(aq) + 3\,OH^- \rightleftharpoons Al(OH)_3\,(s)$$
$$Al(OH)_3\,(s) + OH^- \rightleftharpoons [Al(OH)_4]^-\,(aq)$$
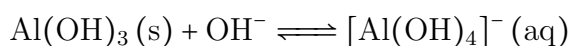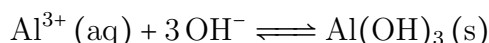
Cu^{2+} and Zn^{2+} form metal hydroxides, which dissolve in excess $NH_3\,(aq)$ to form **soluble complex ions**, where ligand exchange reaction takes place between NH_3 and H_2O.

$$[Zn(H_2O)_6]^{2+} + 2\,OH^- \rightleftharpoons Zn(OH)_2 + 6\,H_2O$$
$$[Zn(H_2O)_6]^{2+} + 4\,NH_3 \rightleftharpoons [Zn(NH_3)_4(H_2O)_2]^{2+} +$$

Reform precipitate from soluble complex ions

- Add 2 to 3 drops of nitric acid. Do not shake test-tube.

$$Zn(OH)_2\,(s) + 2\,OH^-\,(aq) \rightleftharpoons [Zn(OH)_4]^{2-}\,(aq)$$
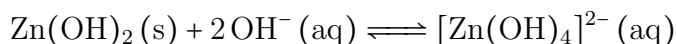
- Add more nitric acid until no further change. Then shake test-tube.

$$Zn(OH)_2 + 2\,HNO_3 \longrightarrow Zn(NO_3)_2 + 2\,H_2O$$
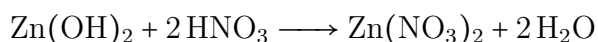
§2.2.2 Test for anions

Refer to QA notes for the complete list.

§2.2.3 Test for gases

Refer to QA notes for the complete list.

§2.3 Gravimetric Analysis

§2.4 Energetics

§2.4.1 Polystyrene cup

§2.4.2 Copper can

> **Why are draught shields arranged around the apparatus?**
> To minimise heat dissipation to the surroundings.

Remark. This method is **highly inaccurate** due to significant heat loss to the surroundings. Hence this method is more commonly used to compare to heat output of different fuels.

§2.4.3 Thermometric titration

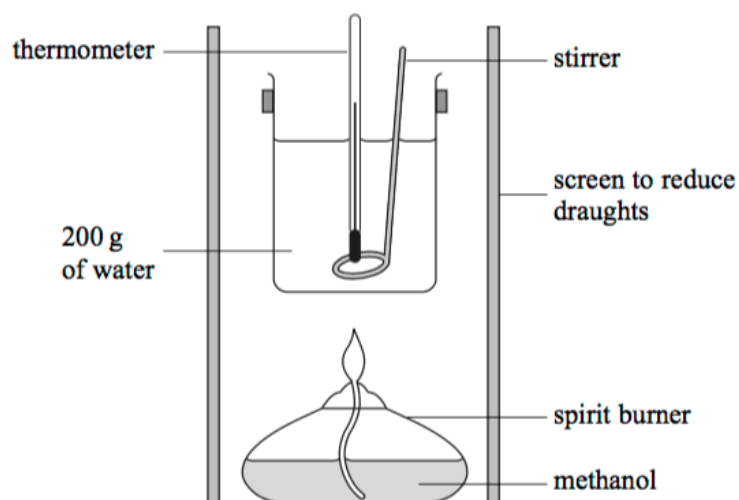To determine the **equivalence point**, monitor changes in temperature after different volumes of (reactant 1) are added to fixed volume of (reactant 2), while keeping total volume of solution constant by adding water.

Plot graph of temperature change against volume of (reactant 1). Draw two intersecting best-fit lines, point of intersection gives the maximum temperature change and equivalence volume.

Remark. At maximum temperature and equivalence volume, maximum $n(H_2O)$ is produced as reactants react in stoichiometric quantities.

§2.5 Kinetics

§2.5.1 Clock experiment (discontinuous method)

Reactions with promiment visual changes, such as precipitation or colour change. Examples include sulfur clock and iodine clock experiment.

For a discontinuous experiment, perform a series of experiments and vary initial concentration of a reactant while keeping all other variables constant.

Rate is given by

$$\text{rate} \propto \frac{1}{\text{time}}$$

For coloured reactants or products, rate is given by

$$\text{rate} \propto \frac{V_{\text{coloured compound}}}{\text{time}}$$

Why is total volume of the reaction mixture kept constant for all experiments?

Total volume is kept constant so that initial concentration is directly proportional to volume, which can be shown using $C_0 V_0 = C_d V_d$.

$$[X]_{\text{given}} V_X = [X]_{\text{initial}} V_T \implies [X]_{\text{initial}} = \frac{[X]_{\text{given}} V_X}{V_T} \implies [X]_{\text{initial}} \propto V_X$$

Using this, we can simply compare the volume of reactant to initial rate to determine order of reaction with respect to each reactant.

Iodine clock experiment

$$H_2O_2 + 2\,I^- + 2\,H^+ \longrightarrow I_2 + 2\,H_2O$$

$$I_2 + 2\,S_2O_3{}^{2-} \longrightarrow 2\,I^- + S_4O_6{}^{2-}$$
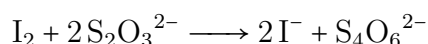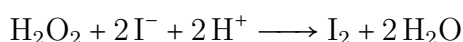
Why does the reaction mixture remain colourless for a period of time?
I_2 is consumed once it is generated. Hence the reaction mixture remains colourless.
Once all $S_2O_3{}^{2-}$ is used up, the second reaction stops. I_2 produced from the first reaction remains in the solution and forms a blue-black complex with starch.

Remark. The time delay of formation of blue-black complex allows us to observe the sudden change in colour of reaction mixture. The time taken for reaction can thus be determined.

Remark. The concentration of KI is varied but the volume of $Na_2S_2O_3$ is fixed, so that the same amount of I_2 is consumed in the second reaction and hence the same amount of I_2 is produced in the first reaction when the colourless mixture turns blue-black.

> **Why is starch solution added?**
> To have a more accurate detection of the end-point colour change.
> (Without the starch indicator, the colour change would be from pale yellow to colourless, hence the end-point colour will not be obvious.)

Sulfur clock experiment

$$\underset{}{S_2O_3{}^{2-}}(aq) + 2\,H^+(aq) \longrightarrow \underset{\text{yellow ppt}}{S\,(s)} + SO_2\,(aq) + H_2O\,(l)$$
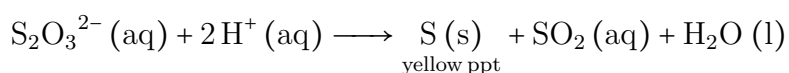
The rate of the reaction can be determined by measuring the time taken to produce a fixed quantity of sulfur.

> **Does it matter whether the measuring cylinders are rinsed with solutions prior to use?**
> Presence of any deionised water will dilute the solution to be measured, giving rise to lower concentration of reactant in the reaction mixture. This error results in longer than expected time measured for the first experiment.

> **Why a watch glass is placed on the beaker?**
> SO_2, a toxic gas, is produced during the reaction. When you observe the "X" from above the beaker, you might inhale too much of this gas. The presence of petri dish helps to reduce this potential hazard.

> **In experiment 2, why 25.0 cm^3 of deionised water is added?**
> This is to ensure that the total volume of the reaction mixture is kept constant. As a result, the concentration of all the reactants in the reaction mixture is directly proportional to the volume of stock solution used.
> When the total volume of the reaction mixture is fixed, the height of the solution in the beaker will also be fixed. This ensures that the amount of sulfur produced to obscure the "X" is approximately the same.

§2.5.2   Change in gas volume

Gas syringe
Reaction between solid and liquid:
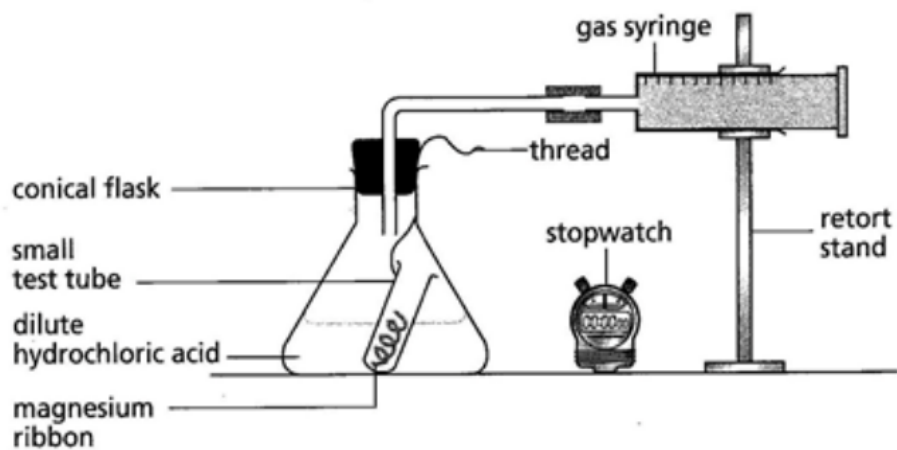
Reaction between two liquids:

Downward displacement of water

§2.5.3   Sampling and titration

> **State the use of cold water.**
> To quench the reaction / slow down the rate of reaction by lowering <u>temperature</u> and <u>concentration</u> of reactant(s).

§3 Planning

Points to include:

1. Instrument and capacity, volume of reagent(s)

2. General procedure

3. Take and record measurements, pre-calculations (if any)

4. Data analysis

Remark. Instruments are **bolded**. Key phrases are <u>underlined</u>. Terms to fill in are in (brackets).

Remark. Adapt the following model answers accordingly, such as using appropriate instruments to measure volumes of certain solutions.

§3.1 Volumetric Analysis

Preparation of standard solution (from solid)

1. Weigh a **weighing bottle** containing (solid) using an **electronic balance** and record the mass.

2. <u>Empty the contents</u> of the weighing bottle into a **100** cm^3 **beaker**.

3. <u>Reweigh</u> the weighing bottle containing any residual (solid). Record the mass and calculate the mass of (solid) used.

4. Add about 50 cm^3 of deionised water to the breaker containing the (solid). Stir with a **glass rod** to <u>ensure that the solid dissolves completely</u>.

5. <u>Transfer</u> this solution into a **250** cm^3 **volumetric flask**.

6. <u>Rinse</u> the beaker thoroughly with deionised water and <u>transfer the washings</u> into the volumetric flask.

7. Add deionised water to the volumetric flask until the liquid level <u>reaches the graduation mark</u>.

8. <u>Stopper</u> the flask and <u>invert a few times to ensure a homogenous solution</u>.

Remark. The first few steps are to ensure that *all* of the given solid dissolves to form a solution.

Preparation of standard solution (dilution)

1. Using a **pipette**, transfer 25.0 cm^3 of (concentration) (solution) into a **100** cm^3 **volumetric flask**.

2. Add deionised water to the volumetric flask until the liquid level reaches the graduation mark.

3. Stopper the flask and invert it a few times to ensure a homogeneous solution.

Titration

1. Fill a 50.00 cm^3 burette with (concentration) (titrant). Record the initial burette reading.

2. Pipette 25.0 cm^3 of (analyte) into a 250 cm^3 conical flask placed on a white tile. Add 2 drops of (indicator).

3. Add (titrant) from the burette into the conical flask with continuous swirling.

4. Add (titrant) dropwise towards the end-point and swirl. Stop the addition when one drop of (titrant) turns the solution from (initial colour) to (final colour).

5. Record final burette reading and calculate titre volume of (titrant).

6. Repeat titration until two titre volumes within ±0.10 cm^3 of each other are obtained.

§3.2 Qualitative Analysis

Test for cation or anion:

1. To 1 cm depth of FA1 in a **test-tube**, add NaOH (aq) slowly, with shaking, until in excess.

Filtration

1. Filter the mixture into a clean test-tube. Wash the residue with deionised water. Residue is ..., filtrate is ...

Heating

1. Use a boiling tube.

§3.3 Gravimetric Analysis

Thermal decomposition

1. Using an electronic balance, weigh and record the mass of a clean, empty and dry crucible.

2. Weigh out accurately about (mass) of powdered mixture into the crucible. Record the total mass of the crucible and its contents.

3. Using a Bunsen burner, heat the crucible and its contents gently for 1 minute, then heat strongly for 5 minutes.

4. Cool the crucible and its contents to room temperature.

5. Reweigh the crucible and its contents using the electronic balance and record the mass.

6. Repeat the heat-cool-reweigh process (steps 3 to 5) until three constant mass readings are obtained. Record this final mass.

§3.4   Energetics

Mix two solutions in polystyrene cup (without temperature correction)

1. Using a 50 cm³ measuring cylinder / burette, add 50 cm³ of (solution 1) into a clean and dry **polystyrene cup**. Support polystyrene cup in a 250 cm³ beaker.

2. Allow (solution 1) to stand for a few minutes. Using a 1°C **thermometer**, measure and record initial temperature of (solution 1).

3. Using another 50 cm³ measuring cylinder / burette, measure 50 cm³ of (solution 2).

4. Allow (solution 2) to stand for a few minutes. Measure and record initial temperature of (solution 2) using a clean thermometer.

5. Rapidly pour (solution 2) into the polystyrene cup containing (solution 1). Cover the cup with a **lid** that is snuggly fitted with a thermometer. Stir the mixture gently using the thermometer.

6. Record the highest (or lowest) temperature reached. Calculate the temperature change.

Copper can calorimetry

1. Using a 50 cm³ measuring cylinder (or burette), add 50.0 cm³ of (fuel) into a **spirit burner**.

2. Weigh the spirit burner containing the fuel using an **electronic balance** and record its mass.

3. Ensure that the **wick** is soaked in the fuel. Adjust the wick such that it is about 2 cm above the cap of the burner.

4. Using another 100 cm³ measuring cylinder, measure 100 cm³ of water into the **copper can**.

5. Allow the water to stand for a few minutes. Measure and record initial water of water in the copper can using a 0.2°C (or 1°C) **thermometer**.

6. Place the copper can on a **tripod stand** with **wire gauze**. Place the spirit burner right below the copper can.

7. Arrange **draught shields** around the apparatus.

8. Light the wick of the burner and ensure that the flame is centred under the copper can.

9. Stir the water gently using the thermometer throughout the time it is being heated.

10. Extinguish the flame when the water temperature has risen by 10°C. Continue stirring the water and record the highest temperature reached. Calculate the temperature change of water.

11. Reweigh the spirit burner containing the unburnt (fuel) after it has cooled to room temperature. Record the mass and calculate the mass of (fuel) combusted.

12. Empty (fuel) from spirit burner and water from the copper can. Repeat steps 1 to 11 using (other fuels).

Thermometric titration (add portions)

1. Pipette 25.0 cm³ of (solution 1) into a <u>clean and dry</u> **polystyrene cup** that is <u>supported</u> in a 250 cm³ **beaker**.

2. Fill 50.00 cm³ **burette** with (solution 2).

3. Allow (solution 1) to <u>stand for a few minutes</u>. Using a 1°C **thermometer**, measure and record initial temperature of (solution 1).

4. Add 5.00 cm³ of (solution 2) from burette into polystyrene cup. <u>Stir</u> mixture gently with the thermometer. Read and record maximum temperature and actual total volume of (solution 2).

5. Repeat step 4 until a total of 50.00 cm³ of (solution 2) has been added.

§3.5 Kinetics

Quenching and sampling

1. Fill a 50.00 cm³ **burette** with (titrant).

2. Transfer 100 cm³ of (reactant 1) into a 250 cm³ **conical flask**.

3. Add 50 cm³ of (reactant 2) into the same **conical flask** and start the **stopwatch** immediately. Stir the mixture using a **glass rod** to ensure even mixing.

4. At time = 5 min, pipette 10.0 cm³ of the reaction mixture into a 250 cm³ **conical flask**. Immediately add 50 cm³ of cold deionised water into the conical flask using a 50 cm³ **measuring cylinder**. Record the exact time at which cold deionised water was added into the conical flask.

5. Titrate the solution in the conical flask against (titrant) in burette. Swirl the mixture in the conical flask and titrate continually until one drop of (titrant) added changes colour of the solution in conical flask from (initial colour) to (final colour).

6. Repeat steps 4 to 5 at time = 10, 15, 20 and 25 min respectively.

7. Record all titration results in a table.

Remark. A large volume of cold deionised water (at least double that of sample withdrawn) should be added to significantly slow down the rate of reaction.

Change in gas volume (no dropping funnel)

1. Using a 50.0 cm³ **measuring cylinder**, add 25.0 cm³ of (reactant 1) into a 250 cm³ **conical flask**.

2. Using a 10.0 cm³ **measuring cylinder**, measure out 2.5 cm³ of (reactant 2).

3. <u>Transfer</u> (reactant 2) into the conical flask and immediately <u>insert a rubber bung</u> into the conical flask. The rubber bung has a delivery tube connected to rubber tubing, with the other end of the tubing inserted into an **inverted 50.00** cm³ **burette filled with water**.

4. Start the **stopwatch** immediately and gently swirl the conical flask continuously to ensure that the reagents are mixed well.

5. Record the water level in the burette at fixed time intervals of 0.5 min. (continuous) **OR** Monitor the water level in the burette and stop the stopwatch when 40.00 cm³ of gas is produced. Record the time taken. Empty and wash the conical flask, repeat steps. (discontinuous)

Change in gas volume (dropping funnel)

1. Using a 50.00 cm³ **burette**, transfer 25.00 cm³ of (reactant 1) into a **conical flask**.

2. Add 2 cm³ of (reactant 2) into a 10 cm³ **dropping funnel**.

3. <u>Open the tap</u> of the dropping funnel to introduce (reactant 2) into the conical flask.

4. <u>Close the tap</u> of the dropping funnel once all (reactant 2) has been added into the conical flask. <u>Start the stopwatch immediately</u>.

5. <u>Swirl</u> the conical flask to ensure reagents are mixed well.

6. Record the volume of gas collected in the gas syringe at <u>every 0.5 min interval</u> until 3 <u>consecutive</u> <u>constant</u> readings are obtained.

Clock experiment

1. Using a 20.0 cm³ **pipette**, transfer 20.0 cm³ of (reactant 1) into a 250 cm³ **conical flask**.

2. Using 50.00 cm³ **burette**, transfer 10.00 cm³ of (reactant 2) into a **beaker**.

3. Pour the solution from the beaker into the conical flask. Start the **stopwatch** immediately.

4. Swirl the flask once to ensure good mixing throughout the experiment.

5. Stop the stopwatch when (visual observation).

6. Record the time taken to the nearest second.

7. Repeat steps 1 to 6 for Experiments 2 to 5.

Remark. Separate the reagents into *two groups*, where reagents in one group do not react with one another. This is for easy addition of reagents simultaneously.

§4 Notes

§4.1 Significant figures

Leave all calculated answers to **3 significant figures**.

Leave all intermediate workings to **5 significant figures**.

§4.2 Precision of measuring instruments

Instrument	Smallest division	Decimal places
electronic balance	0.001 g	3 d.p.
10 cm^3 pipette	exact	1 d.p.
25 cm^3 pipette	exact	1 d.p.
50.00 cm^3 burette	0.1 cm^3	2 d.p., 0.05 cm^3
10 cm^3 measuring cylinder	0.2 cm^3	1 d.p.
25 cm^3 measuring cylinder	0.5 cm^3	2 d.p.
50 cm^3 measuring cylinder	1 cm^3	1 d.p., 0.5 cm^3
0.2°C thermometer	0.2°C	1 d.p.
1°C thermometer	1°C	1 d.p., 0.5°C
stopwatch	0.01 s	1 s

§A   QA Notes

§B   References

- A Level H2 Chemistry Practical

- A-Level H2 Chemistry Practical Planning 5 Years Analysis

- Tips for Planning Questions