

H2 Chemistry (Practical)

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Format

Paper 4 has a 20% weightage of the A-Level assessment.

Total duration: 2 h 30 mins

Total marks: 55 marks

Component	Time	Weightage
Planning	30 mins	5%
Manipulation, Measurement and Observation (MMO) Presentation of Data and Observation (PDO) Analysis, Conclusions and Evaluation (ACE)	2 h	15%

§1 Question Types

§1.1 Decimal places & significant figures

Leave all calculated answers to **3 s.f.**

Leave all intermediate workings to **5 s.f.**

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

§1.2 Table

- Headings
- Units
- Consistent d.p.

§1.3 Calculation

Present full working with units.

§1.4 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

§1.5 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:** degree of accuracy
- **Reagents:** stability of reagent used can be a possible source of error if it decomposes at room temperature or reacts with oxygen in air
- **Dependent variables:** e.g. if dependent variable is temperature change, source of error can be caused by the inherent inaccuracy in measuring temperature i.e. heat lost to surroundings

§1.6 Planning

General outline

1. Apparatus
2. Procedure
3. Measurements
4. Diagram
5. Safety precautions

Refer to section [3](#).

§2 Experiments

Candidates should be able to use appropriate apparatus/equipment to record a range of measurements such as mass, time, volume and temperature.

§2.1 Titration

Record burette readings to 2 d.p.

Titration no.	1	2	3
Final burette reading / cm ³			
Initial burette reading / cm ³			
Volume of XXX added / cm ³			

Place ticks below to indicate the values to be used in average titre volume calculation.

$$\text{Average volume of XXX added} = \frac{15.40 + 15.30}{2} = 15.35 \text{ cm}^3 \text{ (2 d.p.)}$$

Remark. When doing calculations involving dilution, take note of the volumes involved.

§2.1.1 Acid-base titration

Colour changes of pH indicators

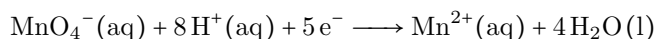
Indicator	pH range	in acid	end point	in alkali
methyl orange	3.2 – 4.4	red	pale orange	yellow
screened methyl orange	3 – 5	magenta	grey	green
methyl red	4 – 6	red	pale orange	yellow
thymolphthalein	9.4 – 10.6	colourless	pale blue	blue
thymol blue	1.2 – 2.8 & 8.0 – 9.6	red	yellow	blue

Remark. Thymol blue is commonly used for double indicator titration.

Choice of pH indicator is appropriate if its pH transition range lies within range of rapid pH change over equivalence point.

§2.1.2 Redox titration

Potassium manganate(VII) titration:

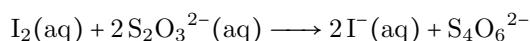


Titration is carried out in acidic medium using $\text{H}_2\text{SO}_4(\text{aq})$.

Remark. $\text{HCl}(\text{aq})$ is not suitable, as it can be oxidised to give Cl_2 .

$\text{HNO}_3(\text{aq})$ is not suitable, as it is a strong oxidising agent and may interfere with the oxidising action of MnO_4^- .

Iodometric titration:



Starch indicator is added for more distinct colour change so that end-point can be determined more accurately. (Without which, colour change would be from pale yellow to colourless, so end-point colour change would be less distinct.)

Remark. Reactive metals e.g. Zn act as reducing agents.

§2.1.3 Indirect titration

Back titration: concentration of analyte is determined by reacting it with a known amount of excess reagent. The remaining excess reagent is then titrated with another, second reagent. The second titration's result shows how much of the excess reagent was used in the first titration, thus allowing the original analyte's concentration to be calculated. You may be asked to prepare standard solutions.

§2.2 Gravimetric analysis

Mass readings, record to 3 d.p.

mass of FA1 + weighing bottle / g	
mass of empty weighing bottle / g	
mass of FA1 used / g	

Thermal decomposition: use heat to decompose a compound to give solid residue and gaseous product (resulting in change in mass), and the difference in mass account for mass lost

Mass of crucible / g	
Mass of crucible + sample / g	
Mass of sample used / g	
Mass of crucible + contents after 1st heating / g	
Mass of crucible + contents after 2nd heating / g	
Mass of crucible + contents after 3rd heating / g	
Mass of residue / g	

How to heat solids

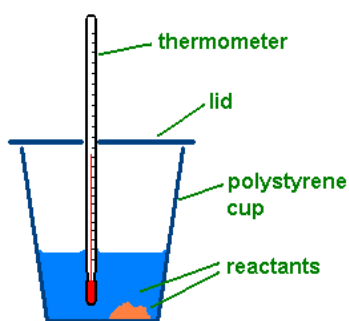
- Heat gently initially to drive off water vapour, then strongly to ensure complete decomposition
Gentle heating: airhole of Bunsen burner half-open
Strong heating: airhole of Bunsen burner fully open
- If solid gives off water when heated, hold test tube slightly sloping downwards to prevent condensed water from running back down

Crucible vs boiling tube

- Open crucible has larger surface area which allows water vapour to be lost more easily when heated.
Using boiling tube, water vapour formed will condense on cooler part of boiling tube, and escapes less easily.
- If lid not used for crucible, some of the heated solid may be lost via wind or through "splitting" during strong heating.

§2.3 Thermochemistry (energetics)

§2.3.1 Calorimetry using polystyrene cup



Polystyrene cup with lid provides sufficient insulation to minimise heat transfer between contents & surroundings

Beaker is used to support polystyrene cup, prevent it from toppling over.

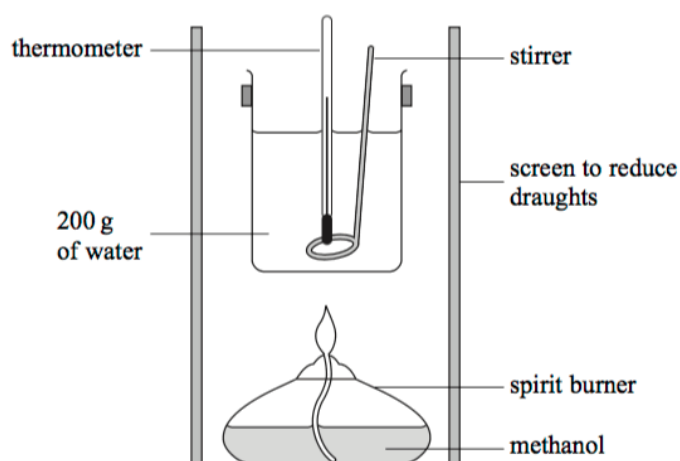
Mixing two solutions (without temperature correction):

Initial temperature of solution 1, $T_1 / ^\circ\text{C}$	
Initial temperature of solution 2, $T_2 / ^\circ\text{C}$	
Average initial temperature, $T_i / ^\circ\text{C}$	
Final temperature, $T_f / ^\circ\text{C}$	
Change in temperature, $\Delta T / ^\circ\text{C}$	

Mixing two solutions (with temperature correction):

t / min	$T / ^\circ\text{C}$
\vdots	\vdots

§2.3.2 Calorimetry using copper can



Mass of spirit burner + fuel before combustion / g	
Mass of spirit burner / g	
Mass of spirit burner + fuel after combustion / g	
Mass of fuel combusted / g	

Initial temperature, T_i / °C	
Final temperature, T_f / °C	
Change in temperature, ΔT / °C	

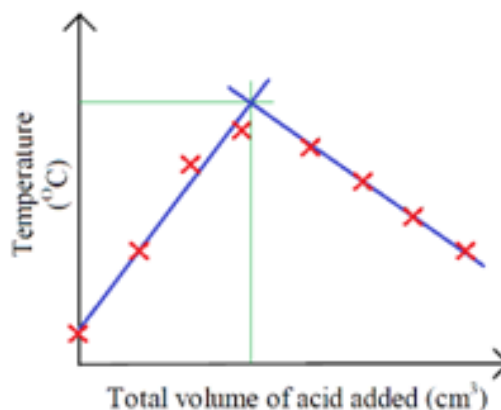
Why are draught shields arranged around the apparatus?

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 heat output of different fuels.

§2.3.3 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.

- Before eq volume, acid is limiting reagent i.e alkali is in excess. As volume of alkali increases, more heat is released as more acid is neutralised.
- At eq volume, stoichiometric quantities of acid and alkali are reacted, max $n(\text{H}_2\text{O})$ produced, so max heat evolved.
- After eq volume, total volume of solution increases while no more heat produced.

§2.4 Chemical kinetics

Types of experiments

1. Continuous method: determine order of reaction using half-life
 - Sampling and titration
 - Change in gas volume
 - Change in colour intensity
2. Initial rate method: determine order of reaction using table
 - Clock experiment
 - Change in gas volume

§2.4.1 Sampling and titration

1. Prepare reaction mixture
2. Withdraw a sample from reaction mixture
3. Quench reaction in sample
4. Analyse sample using titration

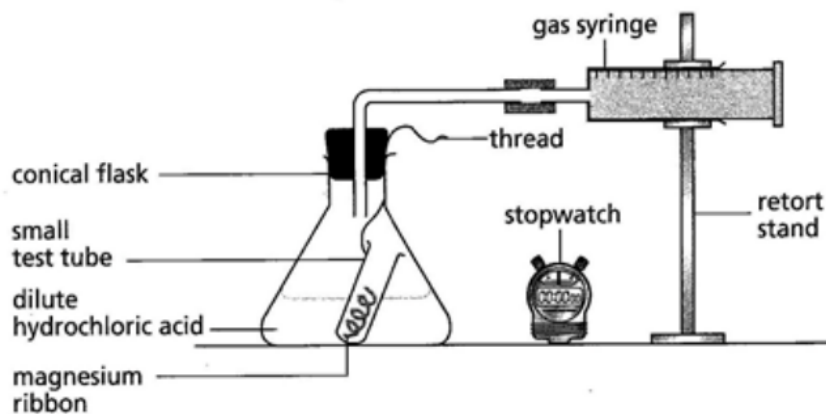
What is the purpose of adding large volume of cold water?

To quench the reaction – slow down rate of reaction by lowering temperature and concentration of reactants.

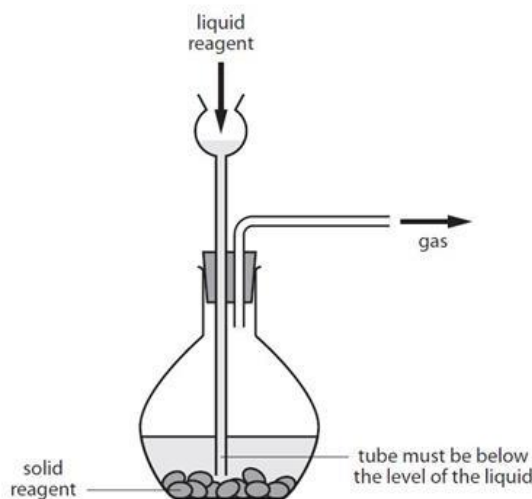
§2.4.2 Methods of gas collection

Gas syringe:

For reaction between solid and liquid

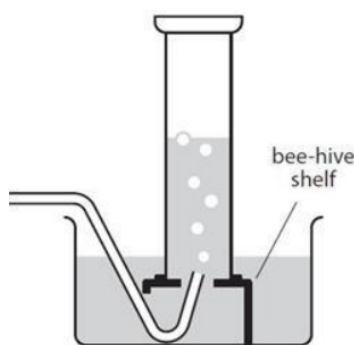


For reaction between two liquids



- Rubber bung used to seal conical flask, prevent escape of gas to surroundings. It is fitted with delivery tube connected to gas syringe.
- Gas syringe is well-greased.
- Solid-liquid: test-tube with thread attached – to initiate reaction, pull thread to mix solid and solution.
- Liquid-liquid: dropper funnel used to introduce a solution into reaction vessel.

Downward displacement of water: (for gases insoluble in water)



- Fill burette with water, invert with its mouth submerged into water tub. Water level in burette is adjusted by using the tap until it reaches 50.00 cm³.
- Measuring cylinder can be used in place of burette.

§2.4.3 Clock experiment

Discontinuous experiment: perform a series of experiments and vary initial concentration of a reactant while keeping all other variables constant

Total volume of reaction mixture is kept constant for all experiments, so that initial concentration is directly proportional to volume:

$$[\text{FA1}]_{\text{mixture}} = \frac{n_{\text{FA1}}}{V_T} = \frac{[\text{FA1}]_{\text{initial}} \times V_{\text{FA1}}}{V_T} \implies V_{\text{FA1}} \propto [\text{FA1}]_{\text{mixture}}$$

since $[\text{FA1}]_{\text{mixture}}$ and V_T are constants.

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

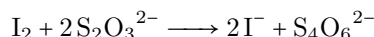
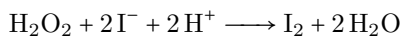
For clock experiments where time is measured based on formation/removal of fixed amount of substance,

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

For clock experiments where time is measured based on disappearance of coloured reactant,

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

Iodine clock experiment:



Product I_2 forms blue-black complex with starch. Time taken for colourless mixture to turn blue-black is measured.

Why does the reaction mixture remain colourless for a period of time?

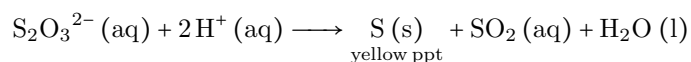
I_2 is consumed once it is generated. Hence the reaction mixture remains colourless.

Once all $\text{S}_2\text{O}_3^{2-}$ is used up, the second reaction stops. I_2 produced from the first reaction remains in the solution and forms 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 $\text{Na}_2\text{S}_2\text{O}_3$ 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.

Sulfur clock experiment:



Rate of reaction can be determined by measuring 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?

When total volume of reaction mixture is fixed, height of solution in beaker is fixed. This ensures that amount of sulfur produced to obscure the “X” is approximately the same.

§2.5 Qualitative inorganic analysis

Identify the type of reaction for each chemical test before you carry out the test – know what to expect

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, in the case of filtration

For negative results, record observation as the **negative statement of the expected observation**.

Test for cations	<p>1. Precipitation</p> $\text{Zn}^{2+}(\text{aq}) + 2\text{OH}^{-}(\text{aq}) \longrightarrow \text{Zn}(\text{OH})_2$ <p>2. Formation of complex</p> <p>Al^{3+}, Cr^{3+}, Zn^{2+} form amphoteric metal hydroxides with OH^{-}, which dissolve in excess NaOH to form soluble complex ions.</p> $\text{Zn}(\text{OH})_2(\text{s}) + 2\text{OH}^{-}(\text{aq}) \rightleftharpoons [\text{Zn}(\text{OH})_4]^{2-}(\text{aq})$ <p>Cu^{2+} and Zn^{2+} form metal hydroxides, which dissolve in excess $\text{NH}_3(\text{aq})$ to form soluble complex ions, where ligand exchange reaction takes place between NH_3 and H_2O.</p> $[\text{Zn}(\text{H}_2\text{O})_6]^{2+}(\text{aq}) + 2\text{OH}^{-}(\text{aq}) \rightleftharpoons \text{Zn}(\text{OH})_2(\text{s}) + 6\text{H}_2\text{O}(\text{l}) \quad (1)$ $[\text{Zn}(\text{H}_2\text{O})_6]^{2+}(\text{aq}) + 4\text{NH}_3(\text{aq}) \rightleftharpoons [\text{Zn}(\text{NH}_3)_4(\text{H}_2\text{O})_2]^{2+}(\text{aq}) + 4\text{H}_2\text{O}(\text{l}) \quad (2)$ <p>3. Reformation of precipitate</p> $\text{Zn}(\text{OH})_2(\text{s}) + 2\text{OH}^{-}(\text{aq}) \rightleftharpoons [\text{Zn}(\text{OH})_4]^{2-}(\text{aq})$ <p>H^{+} added neutralises OH^{-}, so $[\text{OH}^{-}]$ decreases. By LCP, POE shift left to increase $[\text{OH}^{-}]$ partially, hence reforming $\text{Zn}(\text{OH})_2$ ppt.</p> <p>4. Acid-base reaction</p> $\text{Zn}(\text{OH})_2(\text{s}) + 2\text{H}^{+}(\text{aq}) \longrightarrow \text{Zn}^{2+}(\text{aq}) + 2\text{H}_2\text{O}(\text{l})$ <p>5. Redox reaction</p> $\text{Zn}(\text{s}) + 2\text{Fe}^{3+}(\text{aq}) \longrightarrow \text{Zn}^{2+}(\text{aq}) + 2\text{Fe}^{2+}(\text{aq})$ $5\text{Fe}^{2+}(\text{aq}) + \text{MnO}_4^{-}(\text{aq}) + 8\text{H}^{+}(\text{aq}) \longrightarrow 5\text{Fe}^{3+} + \text{Mn}^{2+}(\text{aq}) + 4\text{H}_2\text{O}(\text{l})$ <p>6. Ligand exchange reaction</p> <p>In excess NH_3, ligand exchange reaction between NH_3 and H_2O forms soluble complex ion $[\text{Zn}(\text{NH}_3)_4(\text{H}_2\text{O})_2]^{2+}$, lowers $[\text{Zn}(\text{H}_2\text{O})_6^{2+}]$. By LCP, POE of (1) shifts left to increase $[\text{Zn}(\text{H}_2\text{O})_6^{2+}]$ partially, IP falls below K_{sp} of $\text{Zn}(\text{OH})_2$, hence $\text{Zn}(\text{OH})_2$ dissolves.</p> <p>Remark. If two cations involved, no need to consider mixture of colours of ppt. Just copy and paste from QA Notes.</p> <p>Oxidising agents: H_2O_2 (effervescence of O_2), MnO_4^{-}, $\text{Cr}_2\text{O}_7^{2-}$ Reducing agents: H_2O_2 (effervescence of O_2), KI, reactive metals</p> <p>Remark. Mn^{2+} can only be oxidised to MnO_2 (black ppt), not all the way to MnO_4^{-}.</p>
Test for anions	When testing for halides, must use both AgNO_3 and NH_3 .
Test for gases	Test for gas whenever appropriate.

§2.6 Qualitative organic analysis

Chemical tests

- **Confirmatory test** for a functional group which is unique to this functional group
- **Non-confirmatory test** which gives positive observation for more than one functional group

Be familiar with *all organic chemistry reactions*.

§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 **highlighted**. Key phrases are underlined. 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 Titration

Safety precautions

- Acids or bases are corrosive in nature. Always wear gloves, lab coat and safety goggles to minimise direct contact with chemicals.

Preparation of standard solution: (from solid)

1. Weigh **weighing bottle** containing (solid) using **electronic balance**, record mass.
2. Empty the contents of weighing bottle into 100 cm³ **beaker**.
3. Reweigh weighing bottle containing any residual (solid). Record mass, calculate mass of (solid) used.
4. Add about 50 cm³ of deionised water to the breaker containing (solid). Stir with **glass rod** to ensure that (solid) dissolves completely.
5. Transfer solution into 250 cm³ **volumetric flask**.
6. Rinse beaker thoroughly with deionised water, transfer washings into volumetric flask.
7. Add deionised water to volumetric flask until liquid level reaches graduation mark.
8. Stopper flask, invert a few times to ensure homogeneous solution.

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

Preparation of standard solution: (from solution)

1. Using **pipette**, transfer 25.0 cm³ of (concentration) (solution) into 100 cm³ **volumetric flask**.
2. Add deionised water to volumetric flask until liquid level reaches graduation mark.
3. Stopper flask, invert it a few times to ensure homogeneous solution.

Titration:

1. Fill 50.00 cm³ **burette** with (concentration) (titrant). Record initial burette reading.
2. Pipette 25.0 cm³ of (analyte) into 250 cm³ **conical flask** placed on **white tile**. Add 2 drops of (indicator).
3. Add (titrant) from burette into conical flask with continuous swirling.
4. Add (titrant) dropwise towards the end-point and swirl. Stop the addition when one drop of (titrant) turns 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 cm³ of each other are obtained.

§3.2 Qualitative analysis

Test for cation or anion:

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

Filtration

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

Heating

1. Use boiling tube.

§3.3 Gravimetric analysis

Safety precautions

- If toxic gas gives off, perform experiment in fumehood.
- Irritants involved. Wear gloves, lab coat and safety goggles to minimise direct contact with chemicals.
- While heating boiling tube, point it away from yourself and others to ensure that no one is injured when contents spurt during heating.

Thermal decomposition:

1. Using **electronic balance**, weigh and record mass of clean, empty and dry **crucible** (or boiling tube).
2. Weigh out accurately about (mass) of powdered mixture into crucible. Record total mass of crucible and its contents.
3. Using **Bunsen burner**, heat crucible and its contents gently for 1 minute, then strongly for 5 minutes.
4. Cool crucible and its contents to room temperature.
5. Reweigh crucible and its contents using electronic balance, record mass.
6. Repeat heat-cool-reweigh process (steps 3 to 5) until three constant mass readings are obtained. Record this final mass.

§3.4 Thermochemistry (energetics)

Mix two solutions in polystyrene cup (without temperature correction)

1. Using 50 cm³ measuring cylinder / burette, add 50 cm³ of (solution 1) into clean and dry polystyrene cup. Support polystyrene cup in 250 cm³ beaker.
2. Allow (solution 1) to stand for a few minutes. Using 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 clean thermometer.
5. Rapidly pour (solution 2) into the polystyrene cup containing (solution 1). Cover cup with lid that is snuggly fitted with a the Stir mixture gently using thermometer.
6. Record highest (or lowest) temperature reached. Calculate temperature change.

Copper can calorimetry:

1. Using 50 cm³ measuring cylinder (or burette), add 50.0 cm³ of (fuel) into spirit burner.
2. Weigh spirit burner containing (fuel) using electronic balance, 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 copper can.
5. Allow the water to stand for a few minutes. Measure and record initial water of water in copper can using 0.2°C (or 1°C) thermometer.
6. Place copper can on tripod stand with wire gauze. Place spirit burner right below copper can.
7. Arrange draught shields around the apparatus.
8. Light the wick of burner, ensure that the flame is centred under copper can.
9. Stir the water gently using thermometer throughout the time it is being heated.
10. Extinguish the flame when water temperature has risen by 10°C. Continue stirring water and record highest temperature reached. Calculate temperature change of water.
11. Reweigh spirit burner containing unburnt (fuel) after it has cooled to room temperature. Record mass, calculate mass of (fuel) combusted.
12. Empty (fuel) from spirit burner and water from copper can. Repeat steps 1 to 11 using (other fuels).

Thermometric titration: (add portions)

1. Pipette 25.0 cm³ of (solution 1) into clean and dry polystyrene cup, that is supported in 250 cm³ beaker.
2. Fill 50.00 cm³ burette with (solution 2).
3. Allow (solution 1) to stand for a few minutes. Using 1°C thermometer, measure and record initial temperature of (solution 1).
4. Add 5.00 cm³ of (solution 2) from burette into polystyrene cup. Stir mixture gently with 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

Sampling and titration:

1. Fill 50.00 cm^3 **burette** with (titrant).
2. Transfer 100 cm^3 of (reactant 1) into 250 cm^3 **conical flask**.
3. Add 50 cm^3 of (reactant 2) into the same conical flask, start the **stopwatch** immediately. Stir the mixture using **glass rod** to ensure even mixing.
4. At time = 5 min, pipette 10.0 cm^3 of reaction mixture into 250 cm^3 **conical flask**. Immediately add 50 cm^3 of cold deionised water into conical flask using 50 cm^3 **measuring cylinder**. Record the exact time at which cold deionised water was added into conical flask.
5. Titrate the solution in conical flask against (titrant) in burette. Swirl mixture in 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 50.0 cm^3 **measuring cylinder**, add 25.0 cm^3 of (reactant 1) into 250 cm^3 **conical flask**.
2. Using 10.0 cm^3 **measuring cylinder**, measure out 2.5 cm^3 of (reactant 2).
3. Transfer (reactant 2) into the conical flask and immediately insert a rubber bung into 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 cm^3 burette filled with water**.
4. Start the **stopwatch** immediately, gently swirl the conical flask continuously to ensure reagents are mixed well.
5. Record water level in burette at fixed time intervals of 0.5 min. (continuous method) **OR**
Monitor water level in burette and stop the stopwatch when 40.00 cm^3 of gas is produced. Record the time taken. Empty and wash the conical flask, repeat steps. (discontinuous method)

Change in gas volume (dropping funnel):

1. Using 50.00 cm^3 **burette**, transfer 25.00 cm^3 of (reactant 1) into **conical flask**.
2. Add 2 cm^3 of (reactant 2) into 10 cm^3 **dropping funnel**.
3. Open the tap of the dropping funnel to introduce (reactant 2) into conical flask.
4. Close the tap of the dropping funnel once all (reactant 2) has been added into conical flask. Start the **stopwatch** immediately.
5. Swirl conical flask to ensure reagents are mixed well.
6. Record volume of gas collected in gas syringe at every 0.5 min interval until 3 consecutive constant readings are obtained.

Clock experiment:

1. Using 20.0 cm^3 **pipette**, transfer 20.0 cm^3 of (reactant 1) into 250 cm^3 **conical flask**.
2. Using 50.00 cm^3 **burette**, transfer 10.00 cm^3 of (reactant 2) into **beaker**.
3. Pour the solution from beaker into 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.

§3.6 Qualitative organic analysis

Safety precautions

- Position organic samples away from heat sources such as Bunsen burner.
- Do not heat test-tube containing organic sample using Bunsen burner or naked flame as organic compounds are highly flammable; use hot water bath instead.
- Do not smell or inhale any vapours released by the organic samples.
- Do not pour organic samples down the sink; dispose all organic samples properly into a waste container.

Use a table to present answer:

test	expected observations for positive test	compound identified
1. To 1 cm depth of each unknown sample in separate test-tubes, add a few drops of 2,4-DNPH.	Orange ppt formed.	butanal & butanone
2. To 1 cm depth of each unknown sample in separate test-tubes, add a spatula of $\text{PCl}_5(\text{s})$.	White fumes formed. Gas evolved turned moist blue litmus paper red.	butan-1-ol & butanoic acid
3. To 1 cm depth of $\text{H}_2\text{SO}_4(\text{aq})$ in separate test-tubes, add 1 cm depth of KMnO_4 . Then add 1 cm depth of each unknown sample separately. Heat the test-tubes in a water bath.	Purple KMnO_4 decolourised.	butanal & butan-1-ol

Each sample can be identified based on the combination of positive tests observed.

Positive test	Sample
Tests (1) and (3)	butanal
Test (1) only	butanone
Tests (2) and (3)	butan-1-ol
Test (2) only	butanoic acid

Remark. Do NOT use negative observation to identify compounds!

Remark. If $\text{HCl}(\text{g})$ released, write:

White fumes formed. Gas evolved turns moist blue litmus paper red.

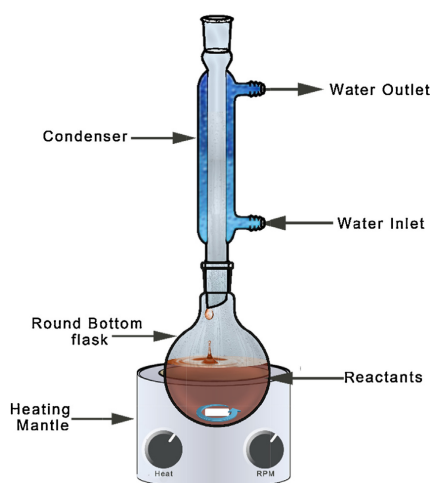
§3.7 Organic synthesis

Organic synthesis experiment usually involves

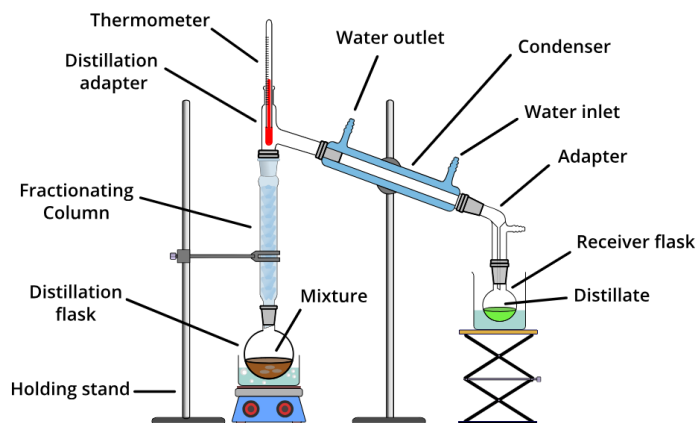
1. Reaction
2. Separation
3. Purification
4. Identification

Experimental setups

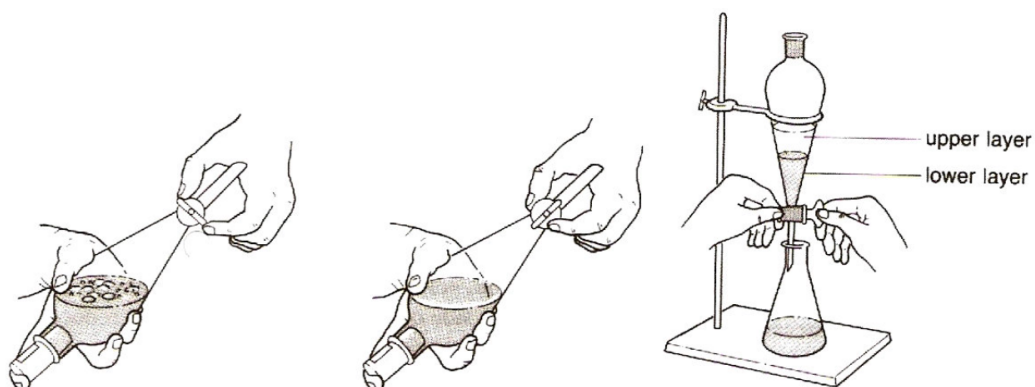
- **Heat under reflux:**



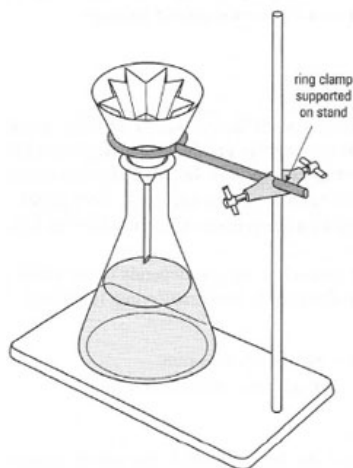
- **Heat with distillation:** separate two miscible liquids



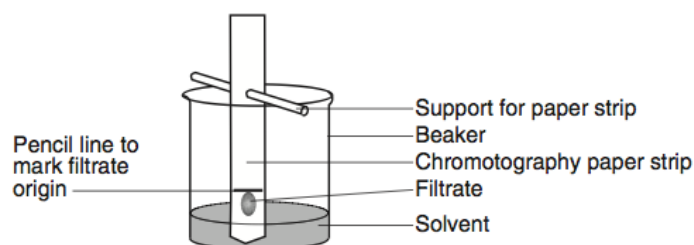
- **Separating funnel:** separate two immiscible liquids



- **Filtration:** separate solid + liquid mixture



- **Chromatography:** separate organic compounds



- **Sublimation and recrystallisation:** purify organic compounds obtained
- **Melting point test:** determine purity of organic compounds

Remark. Conducting a large-scale organic synthesis involving reflux, distillation and purification would not be expected during the practical examination.

Safety precautions

- Flammable chemical: ensure no naked flame around the chemical
- Irritant: wear goggles, carry out experiment in fume cupboard
- Toxic chemical: wear gloves, carry out experiment in fume cupboard
- Corrosive chemical: wear gloves to avoid contact, avoid spillage
- Harmful gas / vapours: carry out experiment in fume hood to minimise inhalation

§3.7.1 Reaction

Heat under reflux:

1. Transfer reaction mixture to a round-bottom flask.
2. Immerse round-bottom flask in water bath placed on heat plate / ice-water bath.
3. Slowly introduce reactant dropwise into flask via dropping funnel + keep contents well-shaken (optional)
4. Step up the apparatus for reflux as shown above.

Heat with distillation:

§3.7.2 Separation

Filtration:

1. Filter the reaction mixture. The filtrate / residue is ...

Separating funnel:

1. Transfer reaction mixture to separating funnel.
2. Stopper and shake separating funnel well, leave mixture to stand.
3. Turn the tap of separating funnel to drain the lower layer into a clean dry 100 cm³ beaker. Discard the top layer.

§3.7.3 Purification

Recrystallisation: (for solids)

1. Dissolve (solid) in a minimum volume of hot water in a small beaker. Do this by adding hot water in small amounts until (solid) just dissolves. Stir the mixture with glass rod to help (solid) dissolve. When dissolved completely, set the solution aside to cool slowly.
2. When crystallisation is completed, filter resulting mixture to obtain the crystals. Rinse the crystals with some cold water during the filtration process.
3. Dry the crystals in a dessicator containing some drying agent to obtain pure (solid).

Drying agents: (for liquids)

1. Add anhydrous calcium chloride, let it stand for 20 minutes. Filter the mixture.

§3.7.4 Identification

Melting/boiling point determination:

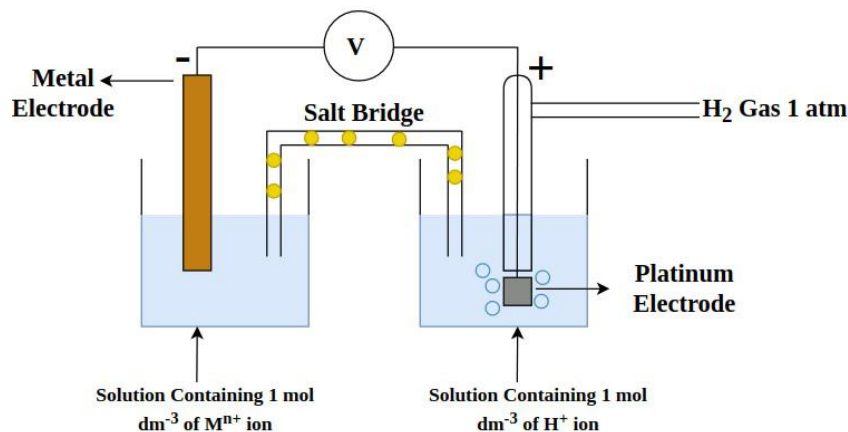
1. Place a small amount of (compound) into melting point capillary tube, and determine its melting point using melting point apparatus.
2. If pure, its melting point would be around $x^{\circ}\text{C}$, and melting would be completed within small temperature range.

§3.8 Electrochemistry

Safety precautions

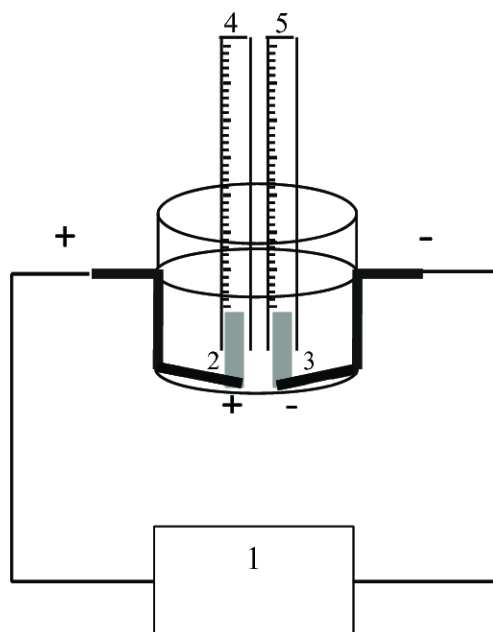
- When handling corrosive electrolytes e.g. acids, wear goggles and gloves OR handle chemicals in fume hood and avoid spillage.
- To avoid electrical shock from high voltages and currents, wear insulated gloves.

Electrochemical cell:



1. (Prepare reactants)
2. Transfer (reactant 1) into a 100 cm³ beaker using (instrument).
3. Transfer (reactant 2) into another 100 cm³ beaker using (instrument).
4. Set up apparatus as shown in the diagram above.
 - a complete circuit that includes voltmeter, salt bridge, electrodes + electrolyte in a thermostatically controlled water bath
 - correct half cells (correct electrodes and solutions of correct concentration)
5. Ensure that **thermostatic water bath** is maintained at 25°C.
6. Record cell potential E using voltmeter.

Electrolytic cell: (collect gas evolved)



1. Assemble the apparatus as shown in the diagram above.
 - complete circuit that includes DC power source + ammeter + electrodes + electrolyte
 - inverted measuring cylinder / burette over the electrode
 - correct labelling of anode and cathode
 - correct polarities of electrodes
2. Record initial reading of the measuring cylinder / burette.
3. Switch on the power supply. Adjust current setting to 0.50 A. Start the **stopwatch** when current reading is 0.50 A.
4. At the end of 10 minutes, stop the stopwatch.
5. Switch off the power supply. Record the final reading of the measuring cylinder / burette.

Remark. Volume of gas collected (using downward displacement of water) is higher than theoretical volume, as it also include volume of water vapour.

§3.9 Equilibria

§3.9.1 Acid-base equilibria

Titration (refer to section [3.1](#))

§3.9.2 Solubility equilibria

Preparation of saturated solution of sparingly soluble salt:

1. Using **measuring cylinder**, transfer 150 cm^3 of deionised water into a dry 250 cm^3 **conical flask / beaker**.
2. Using a **spatula**, add solid (salt) into the flask. Stir contents of flask using **glass rod** until no more solid can dissolve.
3. Immerse the flask in a thermostatically-controlled water bath maintained at 25°C .
4. Allow solution to stand for a few minutes with occasional stirring or swirling.
5. Filter solution through dry filter paper into dry 250 cm^3 conical flask.

9 Qualitative Analysis Notes*[ppt. = precipitate]***9(a) Reactions of aqueous cations**

<i>cation</i>	<i>reaction with</i>	
	NaOH(aq)	NH ₃ (aq)
aluminium, Al ³⁺ (aq)	white ppt. soluble in excess	white ppt. insoluble in excess
ammonium, NH ₄ ⁺ (aq)	ammonia produced on heating	–
barium, Ba ²⁺ (aq)	no ppt. (if reagents are pure)	no ppt.
calcium, Ca ²⁺ (aq)	white ppt. with high [Ca ²⁺ (aq)]	no ppt.
chromium(III), Cr ³⁺ (aq)	grey-green ppt. soluble in excess giving dark green solution	grey-green ppt. insoluble in excess
copper(II), Cu ²⁺ (aq),	pale blue ppt. insoluble in excess	blue ppt. soluble in excess giving dark blue solution
iron(II), Fe ²⁺ (aq)	green ppt., turning brown on contact with air insoluble in excess	green ppt., turning brown on contact with air insoluble in excess
iron(III), Fe ³⁺ (aq)	red-brown ppt. insoluble in excess	red-brown ppt. insoluble in excess
magnesium, Mg ²⁺ (aq)	white ppt. insoluble in excess	white ppt. insoluble in excess
manganese(II), Mn ²⁺ (aq)	off-white ppt., rapidly turning brown on contact with air insoluble in excess	off-white ppt., rapidly turning brown on contact with air insoluble in excess
zinc, Zn ²⁺ (aq)	white ppt. soluble in excess	white ppt. soluble in excess

9(b) Reactions of anions

<i>anion</i>	<i>reaction</i>
carbonate, CO_3^{2-}	CO_2 liberated by dilute acids
chloride, $\text{Cl}^-(\text{aq})$	gives white ppt. with $\text{Ag}^+(\text{aq})$ (soluble in $\text{NH}_3(\text{aq})$)
bromide, $\text{Br}^-(\text{aq})$	gives pale cream ppt. with $\text{Ag}^+(\text{aq})$ (partially soluble in $\text{NH}_3(\text{aq})$)
iodide, $\text{I}^-(\text{aq})$	gives yellow ppt. with $\text{Ag}^+(\text{aq})$ (insoluble in $\text{NH}_3(\text{aq})$)
nitrate, $\text{NO}_3^-(\text{aq})$	NH_3 liberated on heating with $\text{OH}^-(\text{aq})$ and Al foil
nitrite, $\text{NO}_2^-(\text{aq})$	NH_3 liberated on heating with $\text{OH}^-(\text{aq})$ and Al foil; NO liberated by dilute acids (colourless $\text{NO} \rightarrow$ (pale) brown NO_2 in air)
sulfate, $\text{SO}_4^{2-}(\text{aq})$	gives white ppt. with $\text{Ba}^{2+}(\text{aq})$ (insoluble in excess dilute strong acids)
sulfite, $\text{SO}_3^{2-}(\text{aq})$	SO_2 liberated with dilute acids; gives white ppt. with $\text{Ba}^{2+}(\text{aq})$ (soluble in dilute strong acids)

9(c) Tests for gases

<i>gas</i>	<i>test and test result</i>
ammonia, NH_3	turns damp red litmus paper blue
carbon dioxide, CO_2	gives a white ppt. with limewater (ppt. dissolves with excess CO_2)
chlorine, Cl_2	bleaches damp litmus paper
hydrogen, H_2	“pops” with a lighted splint
oxygen, O_2	relights a glowing splint
sulfur dioxide, SO_2	turns aqueous acidified potassium manganate(VII) from purple to colourless

9(d) Colour of halogens

<i>halogen</i>	<i>colour of element</i>	<i>colour in aqueous solution</i>	<i>colour in hexane</i>
chlorine, Cl_2	greenish yellow gas	pale yellow	pale yellow
bromine, Br_2	reddish brown gas / liquid	orange	orange-red
iodine, I_2	black solid / purple gas	brown	purple