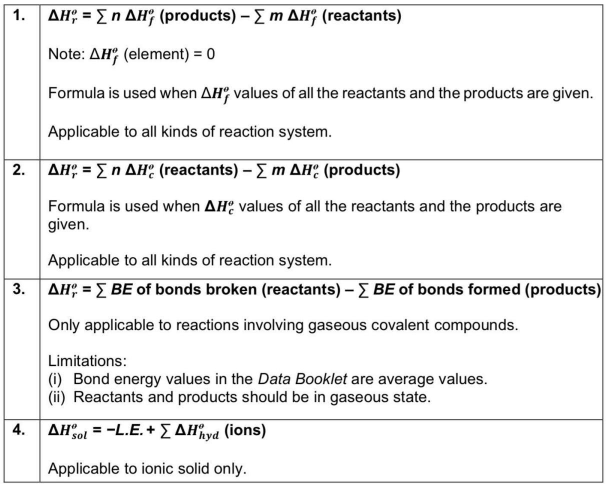
EXPERIMENTAL PROCEDURES

Titration

1. **SF, DP**
    - **Burette – 2 d.p.**
        - All numbers should either end with .00 or .05 – note when u are faking your data
    - **Pipette – 1 d.p.**
    - **Temperature – 1 dp (half of 0.2 deg cel)**
    - **Mr – 1 dp, no units**
2. **Notes**
    - Remember to write units for calculations – write at the start of the paper + signs (they seem to stress negative/positive sign a lot for qns, including change in temp/gradient qns, so make sure to incl signs as well)
    - Don't stress if stuck at mole calculations, read question, read provided info, understand aim of experiment
    - If u use solid to make a standard solution, remember that in ur calculations, use ur measured mass of solid and not the mass u were supposed to add.
    - Calculating % uncertainty:
        - Absolute uncertainty = (½ smallest division) x (number of readings)
        - Burette volume requires 2 readings, so ±0.05 x 2. Mass change/loss also requires 2 readings. Pipette only requires 1 reading so 0.1x1.
        - In your working and final answer, remember to add ±.
        - Eg qn: which piece of apparatus causes the greatest percentage error in your experiment? → asking for u to calculate and compare % uncertainties.
            - Enthalpy = mc (change in T) / amount of LR: when calculating % uc of **change** in T, need to x2 to the ½ of smallest division.
    - % error (difference):

    - no need ±
    - 3 sf
- If qn gives u mass of a compound that has water of crystallisation, the Mr that u divide by must include mass of water of crystallisation!
- If asked to calculate number of moles of electrons, leave as whole number, cuz oxidation states only exist as whole numbers.
- Memorise indicators and their colours (for universal indicator, check the colour of the paper against the chart of pH values given and state the pH value) – methyl orange for acidic || thymol blue and phenolphthalein for alkaline
- If ur not sure what gas to test for, look at the eqn at the start of the practical → gives you the products to test for
- When qn says 'calculate ___ **change**', make sure your final answer has a +/- sign to indicate whether the value increased or decreased.
- If questions asks about why this solution is acidic, explain in terms of charge/size ratio, high charge/size, lets it polarise and weakens O-H bond in H2O, causing it to undergo hydrolysis to give [complex ion], making it an acidic solution
- For planning qns, rmb to draw your table
- If asked the role of xyz in a particular reaction, it's mostly either a catalyst or an acid/base or a R.A/O.A..
- Two readings from an instrument means the overall uncertainty is 2 x the uncertainty of the instrument
- If you pour out a solid from a bottle and the qn asks you to weigh the bottle after pouring, in your table, you must plot as "mass of empty bottle + RESIDUE" – this is because there is bound to be residue after pouring a solid out.
- You quench a mixture by either diluting the solution in a large amount of solvent (like water) or removing the catalyst so that reaction stops happening.
- Tabulation of data and graphing is a huge chunk of marks for prac, so make sure you don't lose any silly marks there.

  - For initial rates method, you have to use rate against conc graph to determine the order (if the line is a straight line passing through the origin, this is first order). For continuous method, you would use a conc against time graph (if curve has a constant half life of t1/2, then it is first order).
  - Be SUPER CLEAR about your apparatus in your planning qn - eg. 250cm^3 conical flask, 100cm^3 measuring cylinder, etc. Don't lose silly marks over this :(



1.	**$\Delta H_r^o = \sum n\, \Delta H_f^o$ (products) – $\sum m\, \Delta H_f^o$ (reactants)** Note: ΔH_f^o (element) = 0 Formula is used when ΔH_f^o values of all the reactants and the products are given. Applicable to all kinds of reaction system.
2.	**$\Delta H_r^o = \sum n\, \Delta H_c^o$ (reactants) – $\sum m\, \Delta H_c^o$ (products)** Formula is used when ΔH_c^o values of all the reactants and the products are given. Applicable to all kinds of reaction system.
3.	**$\Delta H_r^o = \sum$ *BE* of bonds broken (reactants) – $\sum$ *BE* of bonds formed (products)** Only applicable to reactions involving gaseous covalent compounds. Limitations: (i) Bond energy values in the *Data Booklet* are average values. (ii) Reactants and products should be in gaseous state.
4.	**$\Delta H_{sol}^o = -L.E. + \sum \Delta H_{hyd}^o$ (ions)** Applicable to ionic solid only.

They asked the last eqn before, so maybe it's gud to know these (mostly the last one hehe)

Indicator	**When the equivalence point is…**	**Acidic medium**	**Turning point**	**Alkaline medium**
Phenolphthalein	Alkaline	Colourless	–	Pink
Thymolphthalein		Colourless	–	Blue
Bromothymol blue - Minion! - Blue in base	Neutral	Yellow	Green	Blue
Methyl Orange	Acidic	Red	Orange	Yellow
Screened methyl orange		Purple	Grey	Green

Remember acidic → alkaline: ROYGBIV

If asked for color change, it’s always color change from acidic/alkaline medium to turning point color, never acidic to alkaline or alkaline to acidic.

- KMnO4 (self-indicating). “Titrate until the first permanent pale pink is observed.”
- For planning, just stick to methyl orange and phenolphthalein. Others are just in case qns ask.

○

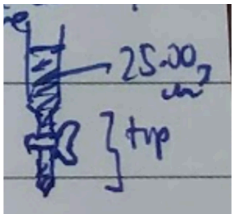
Initial burette reading/cm^3		
Final burette reading/cm^3		
Titre volume/cm^3		
Add your ticks for consistent readings (+- $0.10cm^3$) →		

3. How to wash equipment

- **Burette** – tap water, DI water, titrant
- **Pipette** – tap water, DI water, analyte
- **Conical flask, volumetric/graduated flask** – tap water and DI water only
- **Impt but I keep not doing this: Rinse inner side of conical flask with DI water near endpoint so that total volume measured by burette reacts with analyte in conical flask**
  - This is because some of the titrant that flows into the conical flask may stick onto the walls of the conical flask instead of participating in reaction
  - DI water added will not affect titre value

4. Titre Level Questions

- **Higher than expected when**
    - **Adding titrant to burette immediately after washing with tap and DI water**
        - Water in burette dilutes titrant, lower [titrant] needs larger volume to be reacted with analyte, thus increasing titre value
    - **Air gap/column in burette tip**
        - Solution will fill burette tip first before it is transferred, hence volume measured will be larger than actual volume transferred, thus increasing titre value
    - **Blowing last drop of solution in pipette tip into conical flask**
        - No need to do this because last drop has already been calibrated for, so addition of this last drop into the conical flask will only increase the amount of analyte; greater amount of titrant required to react with it, thus increasing titre value
    - **Rinsing conical flask with analyte**
        - Greater amount of analyte involved then there should be, thus increasing titre value
    - **Not rinsing down sides of conical flask with DI water near endpoint**
        - I personally haven't been doing this gg, idk if you guys do it but it's good practice so pls do it for prelims and A's!!
        - Some of the titrant on the sides of the conical flask may not react with analyte but still measured by burette, thus increasing titre value
    - **Filling burette tip to e.g. 25.00cm3, then transferring all of it into graduated flask**
        - [standard solution] higher than actual since larger volume of solution added to graduated flask than intended (as it includes the volume inside the tip – see below)

      - 

      - Thus increased amount of analyte, increased amount of titrant required to react with it, increasing the titre value
  - **Lower than expected when**
    - **Washing pipette with only tap and DI water, wiping tip with tissue and then immediately pipetting analyte**
      - Water inside the pipette results in smaller volume of analyte being drawn up; smaller amount of analyte require smaller amount of titrant, thus decreasing titre value
    - **Air bubble in pipette**
      - Analyte fill up pipette to 25.0cm3 earlier than it should, smaller actual amount of analyte require smaller amount of titrant to react, thus decreasing titre value
    - **Rinsing conical flask with titrant**
      - Some of the titrant already in the conical flask may have already reacted with the analyte, so less volume of titrant required to be released from the burette for reaction, thus decreasing titre value
  - **Inconsistent when**
    - **Using standard solution without inverting or shaking**
      - [standard solution] inconsistent as [analyte] not uniform, could be higher or lower in different parts of the graduated flask, hence titre value will be inconsistent too

5. **Types of titration**
  - **Acid-base** – no change in oxidation number, use indicator
  - **Redox** – change in oxidation number, some are self-indicating (e.g. when MnO_4^- ⇒ Mn^{2+}, purple ⇒ permanent pale pink), some still require indicator (e.g. iodometric titration requires starch indicator)

- **Iodometric**
  - **Procedure + reactions**
    - **Add oxidising agent X with iodide containing solution into conical flask**
    - **Add H_2SO_4 into the conical flask as well** – provides acidic medium for reaction between OA and iodide to produce H_2O, whereby the H comes from H_2SO_4
    - $X + I^- + H^+ \rightarrow I_2 + \text{another form of } X + H_2O$
    - **Now titrate I_2 with $S_2O_3^{2-}$ until solution turns pale yellow, then add 10 drops of starch, titrate till blue-black turns colourless**
    - $I_2 + 2 S_2O_3^{2-} \rightarrow S_4O_6^{2-} + 2I^-$
  - **Colour changes to note**
    - **Rxn 1: Colourless (iodide) ⇒ brown (iodine) ⇒ pale yellow** (now this is when you add **starch**, otherwise it's hard to tell when pale yellow turns colourless when reaction finishes, so gotta make it blue-black for colour change to be obvious)
    - **Rxn 2: Pale yellow + starch = blue-black** (I_2 turns starch blue-black) ⇒ **colourless (reaction complete!)** (cuz I_2 has been completely reacted away by $S_2O_3^{2-}$)
  - **Why is starch only added when the end-point is near**
    - If starch was added in large amount at the start of the expt, I_2 forms insoluble starch-iodine complex
    - I_2 takes longer to completely react with thiosulfate
    - Discharge of blue-black coloration is delayed
    - To observe end point color change (blue-black to colourless), more than expected volume of thiosulfate may be added
    - Titre volume is larger than expected

- **Back titration & gas collection over water**
  - Back titration involves solids that are insoluble in water / when reaction between analyte and titrant is very slow

Qualitative Analysis

1. Organic

Precautions

- Don't pour organic waste into the sink! Pour into organic waste bucket
- Warm compounds by putting them inside hot water baths, do not heat them over naked flames
- Corrosive → use rubber gloves, perform expt in fume hood

Identification tests

– If you know the thing you're testing contains any of these functional groups, but the test doesn't come back positive, then it's likely that the concentration is too low to give a positive result (but is still present)

- **Alkene**
  - E+ add: Add 1-2 drops of $Br_2(aq)$ to 1cm depth of unknown liquid ⇒ orange $Br_2(aq)$ decolourised
  - Mild ox: Add 1cm3 NaOH, followed by 1-2 drops of $KMnO_4(aq)$ to 1cm depth of unknown liquid ⇒ Purple $KMnO_4$ decolourised, brown MnO_2 ppt (due to alkaline medium) formed upon leaving to stand
  - Vig ox: Add 1cm3 H_2SO_4, followed by 1-2 drops of $KMnO_4(aq)$ to 1cm depth of unknown liquid, warm in water bath ⇒ Purple $KMnO_4$ decolourised
- **Halogenoalkane**
  - Nu: sub, followed by precipitation:
    - i) NaOH(aq), heat (via warming in water bath for 5 mins) – nu: sub where R-OH replaces R-X, X- ion becomes a free ion
    - ii) Cool, acidify with $HNO_3(aq)$ – neutralisation to remove excess OH-
    - iii) Add $AgNO_3(aq)$ – precipitation of AgX
- **Alcohol**
  - Redox (primary/secondary/tertiary): Add a small piece of Na metal to 1cm depth of unknown liquid ⇒ effervescence observed and **Na metal dissolved**, colourless odourless gas evolved extinguishes lighted splint with a pop sound
  - Oxidation (primary/secondary): Add 1cm3 H_2SO_4, followed by 1-2 drops of $KMnO_4(aq)$ / $K_2Cr_2O_7(aq)$ to 1cm depth of unknown liquid, warm in water bath ⇒ Purple $KMnO_4$ decolourised / orange $Cr_2O_7^{2-}$ reduces to green Cr^{3+}
  - Oxidation (for methyl carbinol): To 1cm3 of NaOH(aq), add I_2 (aq) dropwise until no further change. Add 2-3 drops of unknown liquid. Warm if

necessary ⇒ yellow ppt of CHI3 formed, brown I2 decolourises – step-down reaction!

- **If excess I2 is used, write: “yellow ppt formed in brown solution”.**

- Phenol
    - Add a drop of neutral FeCl3 to a small quantity of phenol in water ⇒ yellow Fe3+ solution gives violet colouration with phenol (we don’t need to know the formula of the resultant compound)
    - E+ sub: To 2-3 drops of unknown liquid, add Br2(aq) dropwise till in excess ⇒ orange Br2(aq) decolourised, white ppt formed
- Aldehyde
    - Oxidation: Tollen’s [Ag(NH3)2]+ ⇒ silver mirror / grey ppt formed
        - Prepare Tollen’s reagent first by adding 1cm depth of NaOH(aq) to 1cm depth of AgNO3(aq), followed by NH3(aq) dropwise till ppt formed dissolves
        - Then add 2-3 drops of unknown liquid, warming if necessary
    - Oxidation: Fehling’s (CuSO4) ⇒ brick-red Cu2O ppt formed
        - Prepare Fehling’s solution first by mixing 1cm3 of CuSO4(aq) with 1cm3 of sodium tartrate dissolved in NaOH(aq)
        - Then add 2-3 drops of unknown liquid, warming if necessary (long duration of heating is required here)
    - Oxidation: KMnO4 (aq) / K2Cr2O7 (aq – in theory yes but will not use this during practical), H2SO4 (aq), heat ⇒ Purple KMnO4 decolourised / orange Cr2O7 2- reduces to green Cr3+
- Carbonyl compounds in general (aldehydes, ketones)
    - Condensation: Add 2-3 drops of 2.4-DNPH (Brady’s reagent) to 2-3 drops of unknown liquid ⇒ orange ppt formed
- Methyl carbonyl
    - Oxidation: To 1cm3 of NaOH(aq), add I2 (aq) dropwise until no further change. Add 2-3 drops of unknown liquid. Warm if necessary ⇒ yellow ppt of CHI3 formed, brown I2 decolourises
- Carboxylic acid
    - Acid-base: Add aq Na2CO3 to 1cm depth of unknown liquid ⇒ effervescence of colourless, odourless CO2 gas, forming white ppt with Ca(OH)2 (aq)
- Acyl chloride
    - Precipitation: Add aq AgNO3 to 1cm depth of unknown liquid ⇒ white AgCl ppt formed
- Amine
    - Moist red litmus paper
- Phenylamine
    - E+ sub: To 2-3 drops of unknown liquid, add Br2(aq) dropwise till in excess ⇒ orange Br2(aq) decolourised, white ppt formed
- Amine salts

  - <u>Neutralisation:</u> NaOH(aq), warm; use moist red litmus paper to test for NH_3 evolved
- Amide
  - <u>Alkaline hydrolysis:</u> NaOH(aq), prolonged heating in hot water bath; use moist red litmus paper to test for NH_3 evolved

Use evidence from observations to support conclusions about organic molecule's structure:

1) Observation
2) Type of reaction
3) Deduced functional group present

Template:

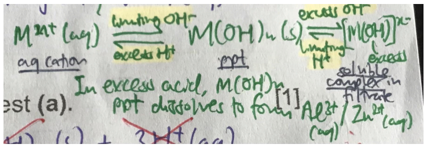
Eg long ass preparation of Tollen's reagent (Ag_2O is brown solid)

"Brown ppt formed in colourless solution"

"Brown ppt dissolves in NH_3 to give a colourless solution"

2. Inorganic

Instruction	What ur supposed to think of	Recording template of observation
Heat strongly in boiling tube	Water of crystallisation Gas	On gentle heating, colourless liquid condenses on cooler part of boiling tube Effervescence observed upon heating. [gas test] - Effervescence only possible in solutions, not in solids. For solids, just insert gas observation.
Add solid to boiling tube	Nothing LOL	Record color of solid
Warm	Gas	Effervescence observed upon

		warming. [gas test]
Add aluminium foil and heat gently/warm	Gas (usually NH3) - Presence of NO3 - - Presence of NO2 - (if NO is liberated by dilute acids, becoming pale brown NO2 in air)	Effervescence observed upon addition of Al (s). Pungent, colourless gas evolved, turning moist red litmus paper blue.
Add distilled water (or acid/alkali) to solid	- Dissolution - Transition metal ions	(Color of solid) solid dissolves, forming (colourless/coloured) solution - If acid causes effervescence, still rmb to write about the dissolution “white solid dissolves in acid to form a colourless soln”
AgNO$_3$	- Test for halides - Redox rxn ○ Eg Fe^{2+} → Fe^{3+}	Color of ppt + solubility in excess NH3
NaOH / NH3	Test for cations	Color of ppt + solubility in excess - Special: for Mn^{2+}, order is: color of ppt → insoluble in excess → turning brown on contact with air
Filter		Colour of filtrate and residue
Filter and add acid to filtrate	Complex ion shit (Zn^{2+}/Al^{3+}) - Theory: LCP and stuff  - - Zn^{2+} ⇒ Zn(OH)$_2$ ⇒ Zn(OH)$_4^{2-}$ OR Zn(NH$_3$)$_4^{2+}$ - Al^{3+} ⇒ Al(OH)$_3$ ⇒ Al(OH)$_4^-$ - TM Cation <-> ppt <->	(Color of ppt) formed which dissolves on shaking in excess acid to give (colourless) solution

	complex	
Dil acid	- CO_3^{2-}/SO_3^{2-}/NO_2^- - Test for metal (MASH) - H_2SO_4: provide SO_4^{2-} to distinguish between Ba^{2+} & Ca^{2+} - HCl: provide Cl^- to test for Ag+	
Ba^{2+}	- Test for $CO3^{2-}$/sulfite/sulfate	White ppt formed with $CO3^{2-}$/sulfite/sulfate
KI	- Redox (it is a reducing agent, so it tests for oxidising agents) ○ Turns colourless ⇒ brown with (Fe3+ for example) - Test for Ag^+ (AgI is yellow)	Solution turns brown.

CO_3^{2-}	- Cations with high charge:size ratio (Fe3+, Al3+, Cr3+)
	  ○ Hydrolysis
	  ○ Produces H+
	- CASCW
	  ○ CO_2 formed
		For acidic cations with high charge:size ratio, M(OH)3 will be produced as ppt. CO2 observed as H+ produced from hydrolysis reacts with CO3 2- to give CO2
		
		For NH4+, no ppt produced as products formed are soluble. CO2 observed however, as NH4+ reacts with CO3 2- to give CO2
		
		CO2: Effervescence observed, gas gives white ppt with Ca(OH)2 (aq)
		
		For non-acidic metal cations, MCO3 will be produced as ppt. No CO2 observed.

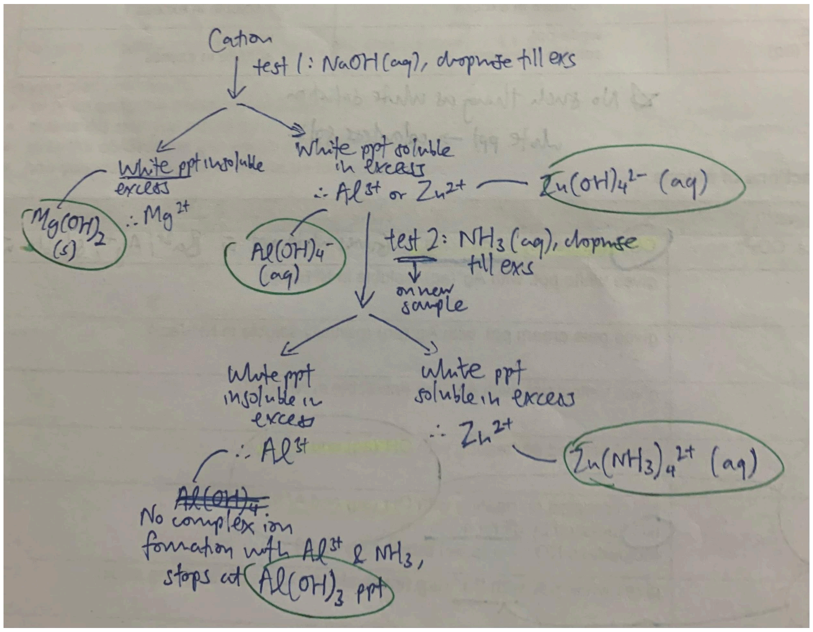
KMnO4	Redox (it is an oxidising agent, so it tests for reducing agents)	

	● Turns purple ⇒ colourless with SO_3^{2-}/NO_2^-/ etc	
Universal indicator		Universal paper turned **from yellow** to (new colour) + **suggested pH**

Standard observations

- CO2
    - Effervescence observed. Gas evolved formed white ppt in $Ca(OH)_2$.
- NH3
    - Gas evolved turns moist red litmus paper blue.
- Soluble ion
    - (colour) ppt formed, soluble in excess NaOH, forming a (colourless) solution

Cation tests (for the white ppt mfs)

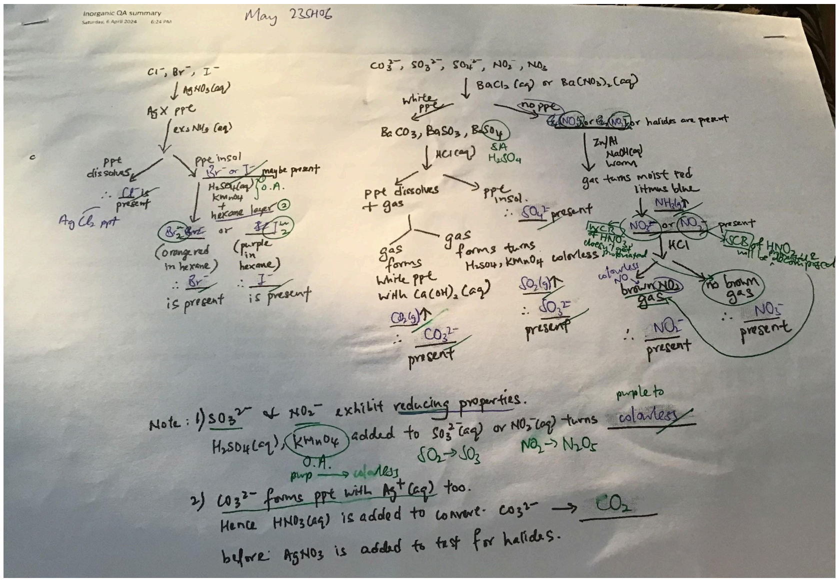


Procedure to test for cations:

- Ba2+/Ca2+: add h2so4 → ppt
- Al3+/Zn2+: to solution of [al(oh)4]+ (ppt soluble in excess naoh), add H+ → hydroxide ppt reappears

- If the sample has 2 cations where 1 is soluble in excess NaOH, 1 is insoluble, add NaOH in excess and filter. Conduct tests on the filtrate containing 2nd cation to confirm its identity.

Anion tests



Inorganic QA summary
Saturday, 6 April 2024 6:24 PM

May 23SH06

Note: 1) SO_3^{2-} & NO_2^- exhibit reducing properties.
H_2SO_4(aq), $KMnO_4$ added to SO_3^{2-}(aq) or NO_2^-(aq) turns colorless purple to colorless
O.A.
purp → colorless
$SO_2 \rightarrow SO_3$ $NO_2 \rightarrow N_2O_5$

2) CO_3^{2-} forms ppt with Ag^+(aq) too.
Hence HNO_3(aq) is added to convert CO_3^{2-} → CO_2
before $AgNO_3$ is added to test for halides.

Procedures to test for anions:

- CO_3^{2-}: To 1 cm depth of FAX, add HNO3 (aq) → eff, gas forms white ppt in ca(oh)2
- X^-: To 1 cm depth of FAX, add Ag(NO3) dropwise, followed by NH3 (aq) in excess.
    - Note difference between this inorganic X- test and organic RX test! In inorganic chem, u alr have X-, so there is no need to add NaOH to form X-.
- SO_4^{2-}: To 1 cm depth of FAX, add Ba(NO3)2 (aq) dropwise, followed by HNO3 in excess
    - U CANNOT ADD BA(SO4) CUZ UR TESTING FOR SULFATE! U CAN'T ADD SULFATE!

Notes for effervescence

- "Bubbles" forming at **top** of mixture is NOT effervescence, is boiling
- Bubbling throughout solution is effervescence
- No such thing as effervescence observed when solid → gas

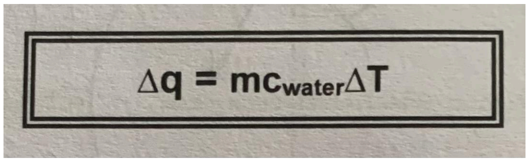
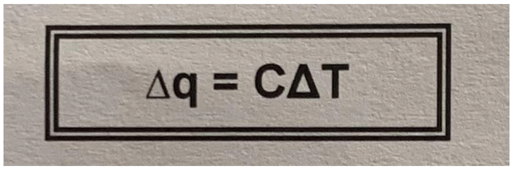
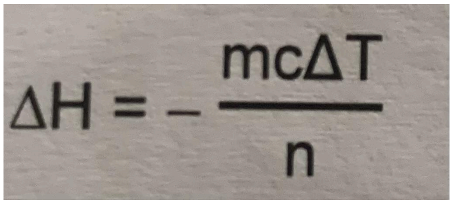
Colours:

- Cu: red brown solid
- Cr: grey solid

All possible complexes – each complex has their respective ppts too, but the ppts dissolve in excess NaOH/NH3, reforming when you add H+ due to POE shifts

- Al(OH)4 -
- Zn(OH)4 2-
- Zn(NH3)4 2+
- Cr(OH)6 3- (dark green solution)
- Cu(NH3)4 2+ (dark blue solution)

Thermochemistry

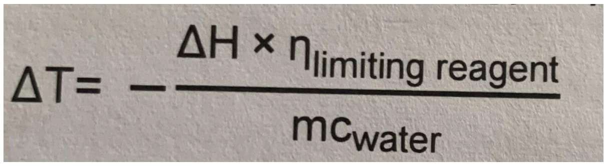
1. **Notes**
    - Temp change → need +/- (make clear the unclear)
        - In ur table temp change need sign!!!
        - Temp change = $T_{final} - T_{initial}$
    - If instructions say record maximum temperature, T_m, in ur table u must use that symbol.
    - Heat change, q → **no** need +/-
    - Enthalpy change: check if there is a trick. Does qn want standard enthalpy? Or a multiple of standard enthalpy based on eqn?
        - Enthalpy change of decomp is supposed to be 1 mol of chemical decomposed
    - 1 dp for temp
    - 3dp for mass
2. **Equations (heat change VS enthalpy change)**
    - 
      $\Delta q = mc_{water}\Delta T$
    - **^ heat change (unit in <u>Joules</u> per mole)**
    - c = specific heat capacity of water, 4.18 J g-1 K-1
    - Note that m is the mass of **solution**, does NOT include mass of solid reacted
    - 
      $\Delta q = C\Delta T$
    - ^ If calibration of calorimeter has already been done
    - C = energy needed to raise temp of a fixed mass of water and container by 1 deg celsius
    - 
      $\Delta H = -\frac{mc\Delta T}{n}$
    - **^ enthalpy change (unit in <u>kilojoules</u> per mole)**
    - n is amt of water formed (based on amt of limiting reagent)

When asked to find enthalpy change of reaction 1, eg

Rxn 1: $2NaHCO_3 + H_2SO_4 \rightarrow \ldots$

$\Delta H = \frac{-mc\,\Delta T}{amount\ of\ NaHCO3}$ ← this is for 1 mole of $NaHCO_3$

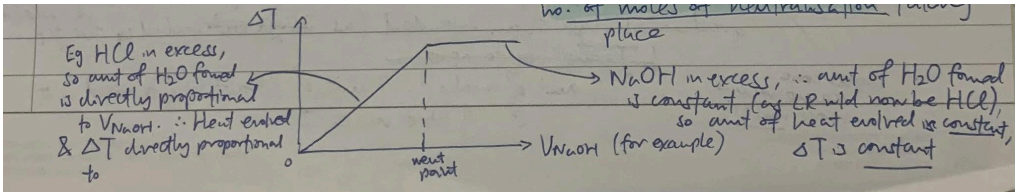
ΔH_1 = 2 ΔH ← in rxn 1, there are 2 moles of $NaHCO_3$

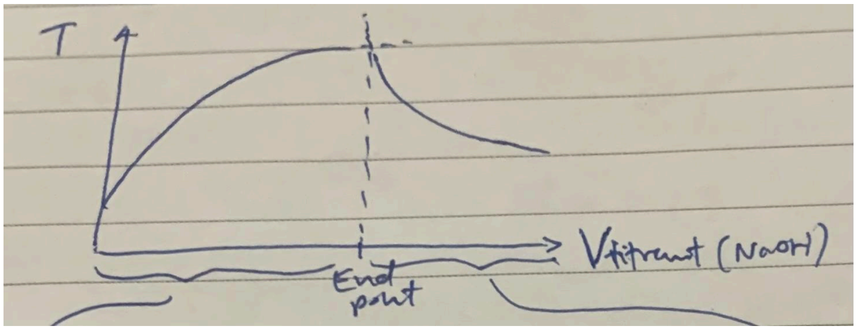
- 

  $$\Delta T = -\frac{\Delta H \times n_{\text{limiting reagent}}}{mc_{\text{water}}}$$
- **Constant**: c of water VS **Variables**: ΔH (indep variable to be found out after conducting expt), n of limiting reagent, mass of water
-

Point of comparison	SASB	WASB / SAWB
Magnitude of ΔH	Larger	Smaller, part of the energy released from neutralisation is used to completely dissociate the weak species, hence smaller magnitude of ΔH
Magnitude of ΔT	Larger	Smaller, (same reasoning as above I think) part of the energy released from neutralisation is used to completely dissociate the weak species, hence smaller magnitude of ΔT which also affects ΔH
Volume of reactants required for neutralisation	Same, as long as concentration of reactants remain the same	

- Also take into account whether the acids; bases are monoprotic/monobasic (same thing); monoacidic or diprotic/dibasic (same thing); diacidic, etc

3. **Direct Mixing vs Cooling Curve**
    - Direct mixing is when you calculate temp change by taking max temp minus min temp, without taking into account heat exchange with the surroundings
    - Cooling curve is when you calculate temp change by drawing 2 best-fit lines over the temperatures before/after mixing/adding, and then extrapolating both lines to the time of mixture/addition to determine Tf and Ti for Tmax. (** more accurate of the two)
        - Doing so helps us take into account heat exchange with the surroundings, which is especially significant for non-instantaneous reactions
4. **Different graph shapes**
    - **When total volume is kept constant**
        - **Why add DI water to keep total V constant?** So that temp change is directly proportional to number of moles of neutralisation (H2O formed) taking place
        - 

        - Shape of graph (aka temp change) dependent on moles of H2O formed, depending on the LR
        - **Before equivalence point**, NaOH was the LR
        - **After equivalence point**, HCl becomes the LR. Amt of HCl is pretty much fixed already, so temp change stays constant
    - **When total volume is NOT constant – Thermometric Titration**
    - 

    - **Before equivalence (not end oops oops) point**
        - Reaction is exothermic
        - Same amt of heat energy released with each fixed additional volume of e.g. NaOH

        - Same heat energy causes smaller temp rise due to increasing total volume/mass of reaction mixture
        - Hence, smaller temp rise when approaching equivalence point
    - **After equivalence point**
        - Reaction is complete; no more heat is produced
        - Since temp of NaOH added is lower than temp of reaction mixture, there is a cooling effect as NaOH is continuously added
        - Cooling effect is reduced as temp difference between NaOH and reaction mixture starts to decrease

[hai i can’t contribute rn bc im doing ki but maybe i wrote smth here u guys can refer to :3]

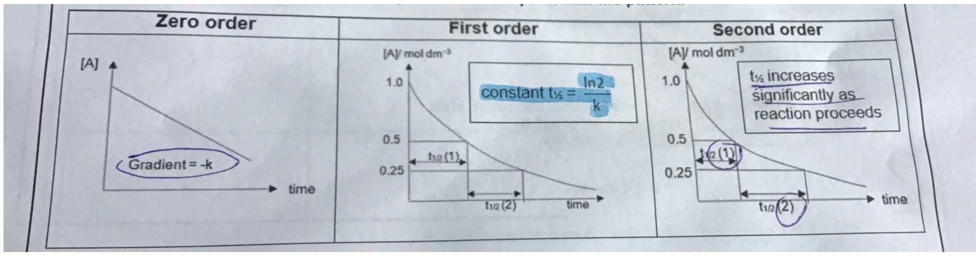
Reaction Kinetics

Gonna update more here after zoom tuition hehehe :3 zoomzoooozozomsdmdOozozmmzz

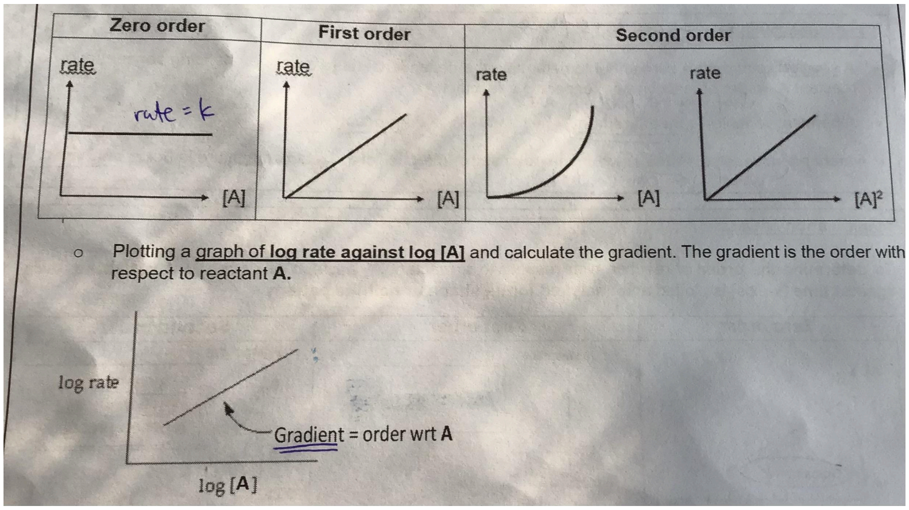
Rate directly proportional to 1/t (when specified by practical qn)

1. **Graphs**

- Explain unusual features of graph
    - Try to guess concept tested: autocatalyst
    - Initial slow rate ← high activation energy ← repulsive force between like charges on ions
    - Rxn sped up ← product acts as catalyst (eg Mn2+)
    - Final rate is slow again ← reactants are used up
- **Continuous**



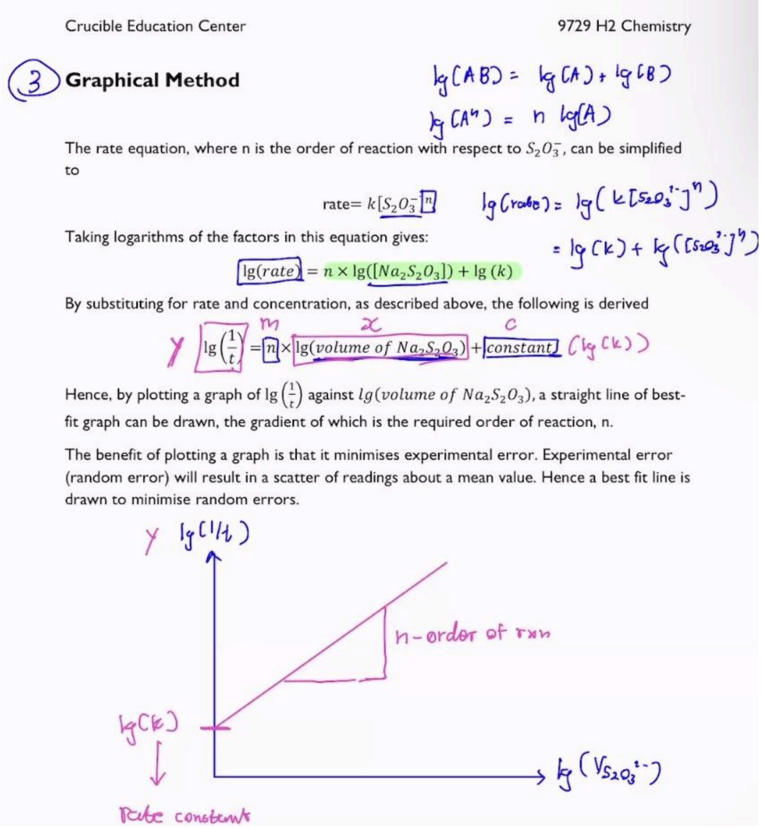
- **Initial Rate (Discontinuous)**



○ Plotting a graph of log rate against log [A] and calculate the gradient. The gradient is the order with respect to reactant **A**.

2. **Methods**

- Continuous Method

    - Quenching agents
        - Na_2CO_3, $NaHCO_3$ – bases, neutralising acid catalyst, causing rxn to slow down
        - Cold water – lower temp, lower average KE, thus lower rate of effective collisions
        - Why is it necessary to add FAX: it quenches reaction by removing the catalyst. WIthout it, reaction will proceed, …
    - E.g. V of S_2O_3 2- proportional to [aliquot] in iodine clock
- Initial Rate (Discontinuous) Method
    - Total volume is constant across experiments (by varying volume of DI water added), hence can treat volume as if it is [reactant] in mixture
- How to deduce order of reaction
    - Inspection or math method
    - V x t or V² x t method
        - If V (of A) x t constant, 1st order wrt reactant A
        - If V² (of A) x t constant, 2nd order wrt reactant A
    - Graphical method
        - E.g. rate = $k[S_2O_3^{2-}]^n$
        - $\lg(\text{rate}) = n \times \lg[S_2O_3^{2-}] + \lg(k)$
        - $\lg(1/t) = n \times \lg(\text{volume of } Na_2S_2O_3) + \text{constant}$
        - 

Crucible Education Center 9729 H2 Chemistry

③ **Graphical Method**

$\lg(AB) = \lg(A) + \lg(B)$

$\lg(A^n) = n \lg(A)$

The rate equation, where n is the order of reaction with respect to $S_2O_3^-$, can be simplified to

$$\text{rate} = k\underline{[S_2O_3^-]}^{\boxed{n}}$$

$\lg(\text{rate}) = \lg(k[S_2O_3^{2-}]^n)$

$= \lg(k) + \lg([S_2O_3^{2-}]^n)$

Taking logarithms of the factors in this equation gives:

$$\boxed{\lg(rate)} = n \times \lg(\underline{[Na_2S_2O_3]}) + \lg(k)$$

By substituting for rate and concentration, as described above, the following is derived

$$y \quad \boxed{\lg\left(\frac{1}{t}\right)} = \overset{m}{\boxed{n}} \times \overset{x}{\boxed{\lg(\underline{volume\ of\ Na_2S_2O_3})}} + \overset{c}{\boxed{constant}} \ (\lg(k))$$

Hence, by plotting a graph of $\lg\left(\frac{1}{t}\right)$ against $lg(volume\ of\ Na_2S_2O_3)$, a straight line of best-fit graph can be drawn, the gradient of which is the required order of reaction, n.

The benefit of plotting a graph is that it minimises experimental error. Experimental error (random error) will result in a scatter of readings about a mean value. Hence a best fit line is drawn to minimise random errors.

PLANNING

Methodology

Repeat experiment (minimum 4 experiments) with varying variable _____, but keep other variables constant such as _________.

Calculations

(eg. min mass of ___ required)

Procedure

1.

2.

Capacities of apparatus:

1. 250 cm3 round bottom flask
2. 10/50/100 cm3 measuring cylinder
3. 50.00 cm3 burette
4. 25.0 cm3 pipette
5. 100 cm3 beaker
6. 250 cm3 conical flask

- Preparation of standard solution (from solid)

1. Weigh __ grams of solid in a **clean and dry** weighing bottle
2. Transfer solid to a small clean beaker
3. Reweigh residual solid in weighing bottle
4. Record masses in table

Mass of weighing bottle and solid /g	
Mass of weighing bottle **and residual solid** /g	
Mass of solid transferred to beaker /g	

5. Dissolve solid in beaker using deionised water. Transfer solid and all washings into a 250cm^3 volumetric flask.
6. Top up volumetric flask to the mark with deionised water, stopper, invert and shake to ensure homogeneous solution. Label as FA_.

  - Table not needed if qn is not focused on this.
    - Scenarios:
      - Not using volumetric flask to dissolve solid
  - Stir to speed up dissolution.

- Preparation of dilute solution (from concentrated solution)

1. Using a 50.00cm3 burette, measure 25.00cm3 of 1.50 mol/dm3 of CuSO4 solution into a 250cm3 volumetric flask (dependent on dilution factor)
2. Fill up to mark with deionised water
3. Stopper, invert and shake well to get a homogeneous diluted CuSO4 solution. Record burette readings in table 1.

- Titration
- Determine dilution factor
  - Qn will tell u if there is a need for dilution (preparation of standard solution)
- Determine who goes into burette, who goes into pipette
  - Let solution with unknown conc be the analyte in pipette. So that u know its volume (25.0cm3)
- Suitable indicator
  - 2-3 drops
  - Choose based on end-point pH
  - If I_2 is used up, end point colour change is blue-black colouration **disappears**. Cuz I_2 reacted away, cannot turn starch blue-black anymore. **10 drops**!! Not ur usual 2-3 drops.
    - I- is colourless/pale yellow
    - I2 is brown
    - I2 turns starch blue-black

1. [burette] Top up a 50.00cm3 burette with FA1.
2. [pipette] Use a 25.0cm3 pipette to transfer 25.0cm3 FA2 into conical flask.
3. [indicator] Add 2-3 drops of (indicator)
4. [how u know expt ended] Titrate FA1 against FA2, until indicator changes colour from __ to __.
5. [table] Record details in table (see table above, under titration).
6. [reliability] Repeat titration until results are consistent, within **±0.10cm3**

- Calculate % by mass of KI → find mass of KI using Mr of KI, not Mr of I^-.

- Procedure example for thermometric titration

1. Using a pipette, transfer 25.0cm3 of NaOH solution into a styrofoam cup, placed in another styrofoam cup supported by a beaker with a lid
2. Using a 0.2 deg celsius division thermometer, record the initial temperature of the NaOH solution
3. Fill burette with 50.00cm3 of HNO3 solution
4. Using the burette, add 2.00cm3 of HNO3 into the styrofoam cup
5. Using the thermometer, record the highest temperature of mixture reached. Record results in table 1.
6. Using the burette, continue adding 2.00cm3 portions of HNO3 and record the highest temperature of the mixture after each addition, as well as the total volume of HNO3 added up to that point.
7. Repeat step 6 until the temperature of the reaction mixture decreases with a constant gradient after max temperature

Table 1

Total volume of HNO3 added/cm3	**Highest temperature of mixture, T/ deg C**
0.00	T1
2.00	T2
…	…
E.g. 30.00	E.g. T15

- **No need "initial burette reading" and allat (cuz u are adding equal volume in every interval)**

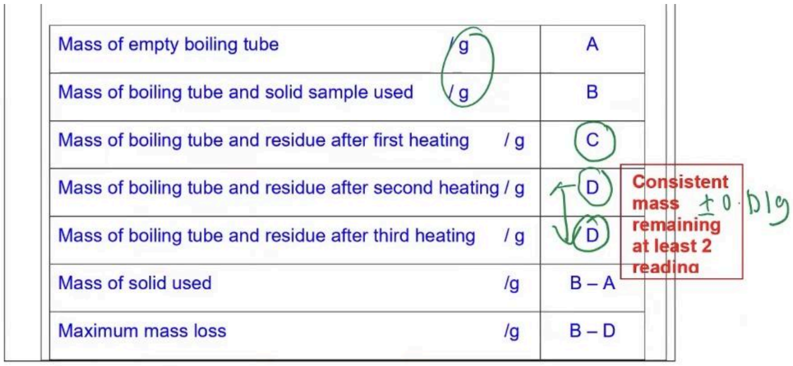
- Procedure example for gravimetric analysis

1. Weigh a **dry**, empty boiling tube using an electronic balance and record its mass
    a. (make sure it's dry!) [also the word record here means to add this value into your table]
2. Weigh about ___g of the mixture into the boiling tube. Record the total mass of the boiling tube and its contents.

3. Heat the boiling tube and its contents gently first, then strongly for about 5 to 10 minutes.
    a. (gently so that the contents don't explode out bc trapped air inside the solid sample may suddenly expand, then you can blast it)
    b. Additional details:
        i. Tap boiling tube intermittently against heat proof mat so that all solid particles are evenly heated
        ii. Heat along length of boiling tube to drive off any water vapour that has condensed on the cooler part of the boiling tube
4. After heating, place the hot boiling tube on a heat proof mat to cool.
5. Once the boiling tube has cooled down, weigh the boiling tube containing the residue and record its mass.
6. Repeat **heat-cool-weigh** process until consistent mass of residue is obtained (**±0.010g**) (rmb the "1" is in the 2nd dp). Record all masses.

- If using filter paper to collect residue, rmb to weigh ur filter paper first
- If transferring solid to somewhere else, rmb to rinse, transfer all washings
- Weighing ppt → rmb to wash ppt with limited water (so no ions on ppt)

table



Mass of empty boiling tube /g	A
Mass of boiling tube and solid sample used /g	B
Mass of boiling tube and residue after first heating / g	C
Mass of boiling tube and residue after second heating / g	D
Mass of boiling tube and residue after third heating / g	D
Mass of solid used /g	B – A
Maximum mass loss /g	B – D

Consistent mass remaining at least 2 reading

±0.01g

- Write "solid" in table instead of writing out its formula
- equate mass loss (B-D) to any gas that might have escaped during vapourisation (like CO2 and H2O) – so you can find amount of gas escaped by during mass loss/Mr of CO2 + H2O
- To find x in formula (solid.xH2O), use mass of h2o and mass of anhydrous solid (not solid.xH2O! Cuz u dont know its Mr) → find molar ratio using amounts

- Boiling tube vs crucible

Crucible better. H2O product might condense and flow back into hot boiling tube → crack

- Energetics

Calculating dilution factor:

Constraints: ΔT must be 5-10 deg cel, volume must be within 80% of styrofoam cup (meaning 80cm3)

Use $\Delta H =- \frac{mc\Delta T}{amount}$, use volume as 80cm3, find ΔT

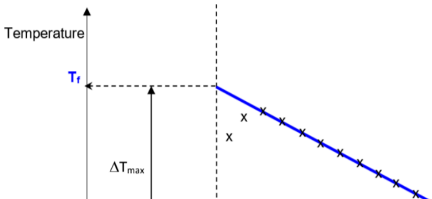
Find dilution factor to get ΔT within 5-10.

Volume of reactant to add in volumetric flask: no need to use the 80cm3. Just use the 250cm3 capacity and whack. Eg dil factor is 10, so add 25cm3. (250/25=10)

1. Insert standard solution and finding mass of solid standard procedures
    a. If burette is used to measure volume of reactant, plonk the titration table.
2. [before] Use a 0.2°C division thermometer to measure the temperature of solution every 0.5 min for 2.5 min.
3. [during + how to tell when to stop] At t=3.0 min, add reactant. Measure and record temperature every 0.5min, until temp has reached maximum value.
4. [after] Continue measuring temp every 0.5 min for 3 more min.
5. Cover styrofoam cup with lid to minimise heat exchange with environment
6. Plot Temp against time
7. By **extrapolation,** determine min and max temp at time of mixing.
8. Table

Time/min	Temp/°C	Time/min	Temp/°C
0.00 (stopwatch is to 2dp)		3.50	
0.50		4.00	
1.00		4.50	
1.50		5.00	
2.00		5.50	
2.50		6.00	
3.00		6.50	

Temp against time (total volume is increasing):



- 2 **straight line** graphs
- 1st graph is **horizontal**

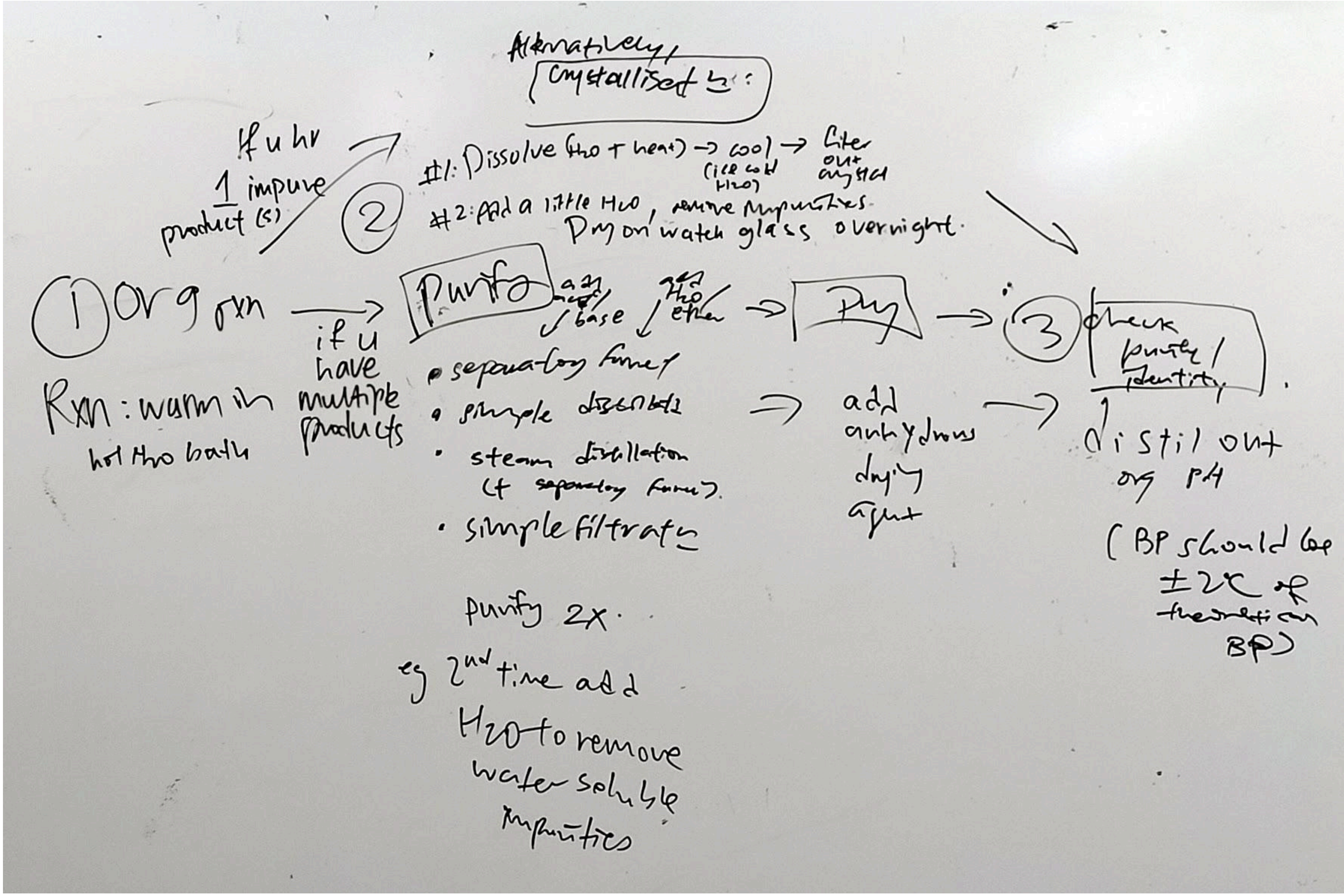
- **Rxn kinetics**

1. Using a 10 cm3 measuring cylinder, measure out 5.0 cm' of the glucose solution.
2. Using another burette, add 24.0 cm^3 of aqueous KMnO4 into a dry 100 cm^3 conical flask / beaker.
3. Using a 25 cm' measuring cylinder, add 20.0 cm^3 H2SO4 into the conical flask containing KMnO4 and swirl the solution.
4. Place the conical flask in a thermostatic water bath set at 15 °C (or any temperature below 29.5°C) and use a thermometer to measure the temperature of the solution in the conical flask.
5. Once the temperature of the solution reaches 15ºC, add the glucose solution from the measuring cylinder into the conical flask and start the stopwatch immediately.
6. Use the thermometer to measure the temperature of the reaction mixture once the stopwatch has been started. This is the initial temperature of the reaction mixture.
7. Stop the stopwatch once the reaction mixture turns colourless.
8. Record the time taken.
9. Use the thermometer to measure the temperature of the reaction mixture once it turns colourless. This is the final temperature of the reaction mixture.

- Organic reaction

- Cannot heat organic reactants with direct flame. Use hot water bath with electronic heater that is temperature controlled (thermostatically-controlled hot water bath).
- Heat under reflux: use condenser and round bottom flask.

    - Top of the condenser must be open. No thermometer in condenser. Prevent pressure build-up.
    - Cold water enters near rxn mixture. (imagine cold water as a stopper, prevent vapour from leaving)
- Use fume hood. Cuz organic chemicals harmful when inhaled.
- Safety: lab coat, goggles, fume hood, gloves, avoid skin contact
- If u are measuring time for a rxn to occur, and measuring temperature also, pour rxts in different conical flasks in hot water bath, ensure solutions have reached desired temperature before adding together and starting rxn.



Alternatively,
Crystallised 's:

If u hv 1 impure product (s)

② #1: Dissolve (H₂O + heat) → cool (ice cold H₂O) → filter out crystal

#2: Add a little H₂O, remove impurities. Dry on watch glass overnight.

① Org rxn → if u have multiple products → Purify (add acid/base ↓, add H₂O/ether ↓) → Dry → ③ check purity/identity

Rxn: warm in hot H₂O bath

- separatory funnel
- simple distillation
- steam distillation (+ separatory funnel).
- simple filtration

purify 2x.

eg 2nd time add H₂O to remove water soluble impurities

→ add anhydrous drying agent →

distil out org pdt

(BP should be ±2°C of theoretical BP)