Planning

- Volumetric analysis
- Gravimetric analysis
- Gas collection
- Energetics
- Equilibrium
- Kinetics
- Electrochemical
- Organic qualitative
- Inorganic qualitative
- Organic synthesis

Main components

- Aim
- Theory (dependent, independent variable)
- Pre-experiment calculations, assumptions made, balanced equations
- Procedure, sequence of steps, apparatus, mass or volume
- Results (tables, graphs)
- Safety considerations: e.g. gloves and lab coat should be worn as concentrated sulfuric acid is corrosive and can cause burns if in contact with skin

Volumetric analysis

- Acid-base titrations
 - Strong acid-strong base, weak acid-strong base, strong acid-weak base
 - o Double indicator (mixture of NaOH and Na2CO3 against st aq HCl)
- Redox
 - o Acidified KMnO4 or acidified K2Cr2O7 or iodine-thiosulfate titration
- Back titrations
 - Excess of one reagent react with compound, titration to determine unreacted reagent
- Precipitation titration
 - Mohr's titration (determine [Cl-] by titration against AgNO3 using K2CrO4 as indicator)

Take note

- Preparation of standard solution using a 250cm3 volumetric flask → from a given solid sample/to dilute a given standard solution
- To titrate A (in conical flask) against B (in burette)
- Volume to be pipetted (10.0cm3 or 25.0cm3 \rightarrow depending on pipette)
- Assume titration result between 20.00-25.00cm3
 - Lower than 10.00: % error associated with titre volume would be rather high, hence inaccurate
 - Higher than 40.00: titration process time-consuming and tedious, need to refill burette after each titration

- Suitable indicators for acid-base: e.g. titration involves the rxn between a SA and WB. MO is a suitable indicator since its **pH range coincides with the region of rapid pH change** in this titration
 - SA-SB: phenolphthalein, thymol blue, methyl orange, screened MO, Bromothymol blue (pH change: 4-10)
 - SA-WB: MO, Screened MO (pH change: 3.5-6.5)
 - WA-SB: phenolphthalein, thymol blue (pH change: 7.5-10.5)
- Colour change at end point
 - MO: 3.2-4.4: red to orange to yellow
 - Screened MO: 3.2-4.4: violet to grey to green
 - Bromothymol blue: 6.0-7.6: yellow to green to blue
 - Thymol blue: 8.0-9.6: yellow to green to blue
 - Phenolphthalein: 8.3-10.0: colourless to light pink to pink
- Redox titration (no indicator required as colour change at end point is distinct)
 - Acidified KMnO4 in
 - Conical flask: purple → colourless
 - Burette: colourless \rightarrow pale pink
 - Iodometric: starch added to conical flask containing iodine near end-point
 - Presence of starch forms soluble bluish-black complex with iodine, turns colourless at end point
 - Cannot use HCl as it would be oxidised, use H2SO4
- Safety considerations
 - Fill the solution in the burette with the aid of a filter funnel and at eye-level → to prevent any accidental spillage of the solution into the eyes
 - When pipetting a solution, pipette filler should be used and there should not be any sucking of solution by mouth → to avoid accidental contact or swallowing of corrosive and toxic chemicals which can cause serious harm

Examples

Calculation of mass of sample to be used

- Mass of solid to be dissolved in water and made up to 250cm3 in a 250cm3 volumetric flask
 - 25.0cm3 of prepared solution is to be pipetted into conical flask and titrated against standard dilute HCl in burette
 - Let volume of HCl used in titration be 25.00cm3 (assume 20.00-25.00) → back calculate mass of Na2CO3

- 1. Using an electronic balance, weigh out accurately about 1.66 g of the impure X sample in a clean and dry weighing bottle
- 2. Dissolve the sample in some deionised water in a 100cm3 beaker. Ensure quantitative transfer of the sample from the weighing bottle to the beaker
- 3. Transfer the solution in the beaker quantitatively into a 250 cm3 graduated flask with the aid of a funnel and a glass rod. Rinse the beaker a few times with deionised water and transfer all the washings into the graduated flask.
- 4. Fill the graduated flask to the 250cm3 mark with more deionised water. Use a dropper to add the deionised water drop by drop when nearing the mark

- 5. Stopper the graduated flask and shake the solution thoroughly to ensure that it is homogeneous. Label the solution FA3
- 6. Pipette 25.0cm3 of FA3 into a 250cm3 conical flask. Add 2 drops of MO indicator
- 7. Fill the burette with the 0.100moldm-3 HCl provided. Titrate the solution in the conical flask with the standard dilute HCl placed in the burette
- 8. Stop the titration when one drop of the HCl added changes the colour of the solution in the conical flask from yellow to orange
- 9. Repeat the titration until at least 2 consistent results are obtained

Calculations

- Let average volume of dilute HCl used by V cm3
- Let actual mass of Na2CO3 used be m grams

Assumptions

- Assume that the impurity does not react with dilute HCl

Back titration

- Determine excess reagent to be used, estimate volume to be used (find exact/min to react with limiting reagent, then increase from there)

Procedure

- 1) Using an analytical balance, weigh accurately about 3.00 g of the impure Na2CO3 into a clean and dry weighing bottle. Record the total mass of weighing bottle and solid
- 2) Using a burette, measure 48.00cm3 of 1.00moldm-3 HCl into a 250cm3 conical flask
- 3) Add Na2CO3 from the weighing bottle into the conical flask containing the acid carefully and immediately cover the conical flask with a filter funnel to prevent acid spray from escaping
- 4) Weigh the emptied weighing bottle and record its mass. Calculate the mass of Na2CO3 that is added into the conical flask using the two mass readings recorded
- 5) When effervescence has ceased, rinse the filter funnel with some deionised water so that any acid present in the filter funnel may be washed into the conical flask
- 6) Add 2 drops of MO into the solution in the conical flask, and titrate this solution against 0.100moldm-3 NaOH. Stop the titration when end point is reached (colour changes from pink to orange)

Calculations

Assume volume of HCl and NaOH used is x and y cm3, mass of sample is m grams
 x/1000-y/1000 (total minus excess)

Deduce relative molecular mass

- 1) using an analytical balance, weigh accurately about 4.80g of FA 1 in a dry and clean weighing bottle
- 2) Dissolve the FA1 in some deionised water in a 100cm3 beaker. Ensure quantitative transfer of FA1 from the weighing bottle to the beaker by rinsing the weighing bottle a few times with small volumes of deionised water and transfer all the washings into the beaker
- 3) Transfer the solution from the beaker quantitatively into a 250cm3 volumetric flask with the aid of a funnel and glass rod. Make up to the 250cm3 mark using more deionised water,

using a dropper when nearing the mark. Stopper the volumetric flask, shake the solution thoroughly and label it FA3

- 4) Pipette 25.0cm3 of FA3 into a conical flask and add 2 drops of phenolphathalein
- 5) Ttrate the FA3 solution against FA2 (in burette). The end-point is reached when the solution in the conical flask changes form colourless to a pale pink
- 6) Repeat the titration to get at least 2 consistent results (getting at least 2 titre volumes which do not differ by more than 0.10cm3)

Preparing more concentrated solution

- 1) Calculate mass of solid required to make a more concentrated solution in 250cm3 volumetric flask
- 2) Using an analytical balance, weigh about 10.80g of X into a clean and dry weighing bottle
- 3) Transfer X to a 250cm3 beaker quantitatively by rinsing the weighing bottle...(same as earlier)

Calculations: derive mass/mol, let volume of FA2 required for titration be Vcm3, let mass of acid be m grams

Gravimetric analysis

Using accurate measurement of mass to quantify a sample

Thermal decomposition

- Use heat to decompose a compound to give solid residue and gaseous product (resulting in change in mass) → difference in mass account for mass lost
- Procedure
 - Record mass of empty boiling tube/crucible
 - Weigh total mass of weighed compound + boiling tube
 - Heat the sample gently initially, followed by strong heating for >10min
 - Cool and weigh boiling tube and contents
 - Repeat process until constant mass is obtained (completely decomposed)
- Safety considerations
 - Allow crucible/boiling tube to cool before handling it with test-tube holder/tongs
 - Do not heat at only one spot, move it up and down to ensure homogenous heating
 - Point boiling tube away from any living beings
- General decomposition reactions
 - Carbonates: MO + CO2
 - Sulphites: MO + SO2
 - Hydroxides: MO + H2)
 - Nitrates: Gp1 \rightarrow NO2- + O2, generally: MO + NO2 + O2
 - Ammonium compounds: ammonia gas
 - Salts containing water of crystallisation: anhydrous salt + H2O

Examples

Procedure

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

- 2) Weigh out accurately about 5.00g of solid X into the crucible. Record the total mass of the crucible and the solid X
- 3) Using a Bunsen burner, heat the crucible and is contents gently at first, and then heat strongly for 10 min
- 4) Cool and weigh the crucible and its contents
- 5) Repeat the heating-cooling-weighing process until constant results are obtained (i.e. when 2 consecutive mass readings have a difference less than 0.01g)

Mass of empty crucible/g	А
Mass of crucible and X/g	В
Mass of crucible and its contents	
After first heating/g	С
After second heating/g	D
After 3 rd heating/g	D

Calculations

- Mass of X left: D-A (anhydrous salt)
- Mass of H2O driven off: B-D

Determining % by mass of a particular compound in a mixture (only 1 decomposes)

- 1) Weigh an empty, dry and clean boiling tube and record its mass Ag
- 2) Using a spatula, transfer about 2.00g of the mixture of X and Y provided into the boiling tube. Weigh the boiling tube and its contents and record the total mass (Bg)
- 3) Using a Bunsen burner, heat the boiling tube with its contents gently at first, and tehn strongly for 10 min
- 4) Allow the boiling tube and its contents to cool. Then weigh the cooled boiling tube and its contents and record the total mass (Cg)
- 5) Reheat the boiling tube and its contents for another 10 min, then cool and weigh. Repeat this heating-cooling-weighing process until constant mass is achieved. The latter indicates that complete decomposition of X has occurred

Why crucible without lid might be more appropriate

- Heating the sample suing an open crucible will allow water vapour to be lost more easily. With a boiling tube, the water vapour evolved will condense on the upper inner walls of the boiling tube to form tiny water droplets. Further heating will be required to remove these water droplets

Mass of empty crucible/g	А
Mass of crucible and X/g	В
Mass of crucible and its contents	
After first heating/g	С
After second heating/g	D
After 3 rd heating/g	D

Mass/molar mass to find out no. of moles ...

Safety hazards

- Point the boiling tube away from yourself and others. This is to ensure that no one gets hurt in case of spurting
- Handle the boiling tube with a test tube holder at all times. This s to ensure that your hands do not get burnt

Precipitation

- Determine amount of ion which forms an insoluble compound when you add a reagent
- Both ions and ppt reagent must be in aqueous state

Procedure (general)

- 1) Weigh accurately a sample of the soluble analyte salt
- 2) Dissolve the salt in deionised water in a 50cm3 beaker
- 3) Add a solution of the precipitating reagent and stir the mixture
- 4) Allow the ppt to settle
- 5) Filter the mixture through a pre-weighed sintered glass crucible
- 6) Wash the ppt with several portions of cold deionised water (prevent dissolution). Dry the ppt and glass crucible in the oven
- 7) Cool the ppt and glass crucible in a dessicator for about 10min. weigh accurately the ppt and glass crucible
- 8) The heating-cooling-weighing are repeated until a constant mass is obtained

Insoluble salts

- AgX, PbX, BaSO4, CaSO4, PbSO4, all carbonates, all OH-, most chromates (CrO42-)
- If sulfate is also present in solution of halides (vice versa) should not use Pb(NO3)2 as PbSO4 is insoluble and will ppt out tgt with PbX
- NaOH cannot ppt out Pb2+, Ag+, Ba2+, Zn2+
 - Pb(OH)2/Zn(OH)2 is amphoteric, dissolves in excess NaOH
 - Ba(OH)2 has appreciable solubility (inaccurate)
 - Ag(OH) is unstable and decomposes to form Ag2O

Examplesn

Procedure

- 1) Weigh accurately 0.5g of the sample into a small beaker
- 2) Add 20cm3 of dilute HNO3 to the sample. Stir and mix well (dissolve both salts)
- Add 20cm3 of hot, dilute hydrochloric acid to the resulting solution. Stir the mixture vigorously (assume sample contains 100% of particular salt, calculate concentration of HCl needed)
- 4) To check if all Pb2+ has ppt out, pour small amount of solution (without ppt) and test with a few drops of concentrated HCl. If no ppt is formed, it confirms that all Pb2+ have ppt out and none are in the solution
- 5) Filter the mixture to obtain the ppt
- 6) Dry the ppt and crucible in the oven for about 10min
- 7) Cool the ppt an crucible in a dessicator
- 8) Weigh the ppt and crucible

Calculations

- Assume mass of ppt obtained is m grams, determine no. of moles of Pb2+

Gas Collection

- For reactions that produce a gas
 - Acid-carbonate
 - o Acid-metal
 - o Decomposition
- Common apparatus
 - Frictionless graduated gas syringe (100cm3)
 - o Inverted burette filled with water
 - Inverted measuring cylinder filled with water (only for gases w poor solubility in water)
- Errors
 - \circ Swirling of flask is variable \rightarrow varying volume of gas collected
 - Gas collected can be compressible
 - o Volume collected dependent on temp and pressure
 - o Gas collected deviates from ideal gas
 - Some gas may dissolve in water (if water displacement method used)
 - Some gas may escape from set-up
 - If using gas syringe, gas collected must be at least half, but not exceeding capacity of syringe
- Examples
 - \circ Adding a solid into a solution
 - Gas syringe held by retort sand
 - Conical flask has a side arm which connects to gas syringe via rubber tubing
 - Small test tube containing solid is tied to a string (Prevent gas loss)
 - \circ Adding a solution into solid
 - A pump is required to force the solution from the burette into the flask
 - o Acid-metal rxn
 - Should not be used when gas is soluble n water
 - Burette can be used in place of measuring cylinder water displacement method

Examples

Assumptions

- Assume gas syringe is 100cm3
- Assume 72.0cm3 of gas is collected (more than half gas syringe)
- Assume ideal gas law (rtp 1 mol of gas occupies 24dm3)
- Amount solid is pure
- Calculate mass of solid to be used
- Assume conc of HCl used between 0.1 o 1.0 moldm-3 e.g. let conc of HCl be 0.200moldm-3
 → calculate amount of HCl required

Draw diagram

Procedure

1) Using a measuring cylinder, introduce 60cm3 of 0.200 moldm-3 HCl(aq) into the clean and dry 250cm3 conical flask with a side arm

- 2) Using an analytical balance, weigh accurately about 0.32g of FA1 in a small test tube tied to a string
- 3) Set up the apparatus as shown in the diagram
- 4) Lower the test-tube with FA1 into the conical flask, taking care that the reagents do not mix. Stopper the conical flask
- 5) Check that the initial reading of the 100.0cm3 graduated gas syringe is set at the zero mark
- 6) At a suitable time, loosen the stopper slightly to release the string such that the FA1 in the test tube can react with HCl(aq). Stopper the conical flask immediately
- 7) Swirl the conical flask gently to ensure that the reagents are well mixed
- 8) Allow the reaction to progress until it has ceased, as indicated by a constant volume reading of the syringe
- 9) Record the final volume, Vcm3 on the graduated gas syringe
- 10) Repeat the experiment to get consistent results

Calculations

- Assume ideal gas law: 1 mol of gas occupies 24 dm3 at rtp

Possible measures to ensure that all gas given off was collected

- Ensure that the set-up is air-tight to minimise escape of gas (at rubber tubing juncture and at stopper). This is to ensure that an accurate volume of O2 produced is obtained

Ways to ensure reliability of results

- After reaction has ceased, time is allowed for equilibrium to be reached (pressure and or temperature) before measurement of volume
- The experiment is repeated to obtain consistent values of the volume of O2(g) collected

Energetics

- Measurable change in temperature, with the use of a calorimeter
- Common experiments
 - Determination of enthalpy change of reaction (neutralisation, solution)
 - o Determination of enthalpy change of combustion
- Definitions
 - Enthalpy change of reaction: energy absorbed or released when 1 mole of reactants react to form products at 1 atm and 298K
 - Enthalpy change of combustion: energy released when 1 mole of substance is completely burnt in oxygen at 1 atm and 298K
 - Enthalpy change of neutralisation: energy released when an amount of acid or base reacts to form 1 mole of water at 1 atm and 298K
 - Enthalpy change of solution: energy absorbed or released when 1 mole of solute is completely dissolved in a solvent to form an infinitely dilute solution at 1 atm and 298K
- Graph plotting (extrapolation to find minimum and maximum temp reached, vertical reading is the temp change
 - To account for heat lost/absorbed

- Monitor the temp of a solution at regular time intervals. At a suitable time, add the other reagent to the solution and take temp readings at 0.5 min intervals
- This can only be done if a stopwatch is stated in the apparatus list

Time/min	Temp/degC
0.0	
0.5	
1.0 (addition of reagent)	
1.5	
2.0	
2.5	

- \circ ~ To find unknown concentration of reagents used
 - Measure and record temp of solution. Take temp readings after adding small volumes of a reagent to the solution
 - When a max or min temp is reached, the corresponding volume is the optimal volume of reagent required. Total volume must be kept constant

Volume of solutions added/cm3		Temp/degC
Solution A Solution B		
5 45		
15 35		
25	25	
35	15	
45	5	

Examples

Calculations

- Assume density of water: 1.0gcm-3
 Let volume of water used be 50.0cm3
 mass of water 50.0g
 amount of water used: 2.778mol
- Amount of reactant used (2.778 mol divide by molar ratio of water to reactant)

- Use a pipette, transfer 50.0cm3 of deionised water into a clean and dry polystyrene cup supported in a 250cm3 beaker. Take the initial temperature of the water using the thermometer provided
- 2) Using an analytical balance, weigh accurately about 1.49g of NH4Cl in a weighing bottle. Record the total mass of the weighing bottle and solid NH4Cl
- 3) Empty the solid NH4Cl into the polystyrene cup and stir gently using the thermometer to dissolve all the NH4Cl. Take note of the lowest temperature reached
- 4) Weigh the emptied weighing bottle and record its mass
- 5) Repeat steps 1 to 4. Use the data obtained from each expt to calculate a value for deltaH. Take the average of these 2 values

Mass of weighing bottle and solid NH4Cl/g	
Mass of emptied weighing bottle/g	
Mass of solid NH4Cl used/g	

Lowest temperature reached/degC	
Initial temp/degC	
Change in temp/degC	

Assume specific heat capacity of solution to be 4.18Jcm-1K-1 Assume no heat absorbed from surroundings

Decrease in temp: -T/increase in temp: T

deltaH= -q/n

Neutralisation reaction

Procedure

- 1) Fill a 50.00cm3 burette with FA2
- 2) Support the Styrofoam cup in a 250cm3 beaker and pipette 25.0cm3 of FA1 into the Styrofoam cup
- 3) Use a thermometer to measure the steady initial temp of FA1. Record this temp
- 4) Run 3.00cm3 of FA2 into the cup. Stir and record the highest temp observed
- 5) Immediately add another 3.00cm3 of FA2 from the burette and repeat the temperature measurement
- 6) The end-point has been passed when the addition of FA2 no longer causes an increase in temp (or leads to a decrease in temp)
- 7) Repeat step 5 until a total of 48.00cm3 of FA2 has been added

Vol of FA2 added/cm3	Highest temp observed/degC	Change in temp/degC
0.00		
3.00		
6.00		
9.00		
48.00		

Plot graph and determine volume of acid required to reach highest temp (intersection of 2 graphs: one for increasing gradient, other for decreasing gradient)

Combustion of organic compound

- Need to use spirit lamp with wick partially immersed in fuel (lamp placed beneath calorimeter)

Mass of spirit lamp and fuel before	А
combustion/g	
Mass of spirit lamp and fuel after combustion/g	В
Mass of fuel	A-B
mcdeltaT: m is mass of fuel used	

Temp of water in calorimeter before	С
Temp of water in calorimeter after	D
combustion/degC	
Change in temp/degC	D-C

Procedure

- Using a 100cm3 measuring cylinder, place 100cm3 of water in the calorimeter provided. Record the initial temperature of the water in the calorimeter
- 2) Add X to the spirit lamp as fuel
- 3) Weigh the spirit lamp and the fuel a record the total mass
- 4) Place the spirit lamp under the calorimeter as shown in the diagram
- 5) Light the wick of the lamp. Stir the water in the calorimeter gently until there is a rise of about 5-10degC. Blow to extinguish the flame and record the final temperature of the water. Allow the spirit amp to cool sufficiently. Then, reweigh the spirit lamp and fuel and record the new total mass

Possible sources of errors

- Incomplete combustion of fuel
 - Increase oxygen content of surroundings (e.g. place a beaker of H2O2 solution mixed with a small amount of MnO2 beside the set-up)
- Rapid loss of fuel due to evaporation, especially immediately after the flame is extinguished
 Cover the wick with a small cap when the spirit lamp is not in use
 - Heat loss from flame due to draughts
 - Use a windshield to exclude draughts around the apparatus
- The use of a thermometer with divisions of 1degC to measure a small temp change of 5-10degC results in less accurately measured temperatures
 - Use a thermometer of higher precision (e.g. measures temp to 0.1degC) to improve the accuracy of temp readings. Or use a thermocouple/data logger with a temperature probe.
- The thermometer may have a long response time, e.g. due to a large bulb, leading to lower readings taken. As such max temp reached will be lower, making the calculated values of enthalpy changes lower as well
 - Use a thermometer with a shorter response time e.g. smaller bulb, or a thermocouple/data logger with a temperature probe, which would allow fast and easy access to the actual temp
- The heat capacity of the calorimeter was not taken into consideration
 - Calibrate the calorimeter used by first conducting a combustion experiment with another fuel for which its enthalpy change of combustion is known

Measuring via time intervals

- 1) With the use of a burette or measuring cylinder, add 25.00cme3 of FA2 into a polysterene cup supported in a glass beaker
- 2) Using a weighing bottle, weigh about 1.00g of FA1. Record the total mass of the weighing bottle and FA1
- 3) Measure and record the temperature of the dilute nitric acid at 30s intervals. At 3.0 min, add FA1 and stir with the thermometer. Measure and record the temperature at 30s intervals for a further 10 min.
- 4) Plot a graph of temperature against time. Form the graph, obtain the maximum temperature change by extrapolation OR measure and record the initial temperature of the dilute nitric acid. Add FA1 and stir with the thermometer. Measure and record the highest temperature reached
- 5) Reweigh the emptied weighing bottle. Calculate the mass of FA1 used.

In the reaction CO2 is given off, heat is lost during the evolution of a gas.

Kinetics

Using initial rates method (clock reaction)

Procedure

- 1) Using a measuring cylinder, add 5cm3 of KI and 5cm3 of Na2S2O3 to a 100cm3 beaker
- 2) Add 40cm3 of deionised water and 5 drops of starch to the same beaker
- 3) Place the beaker on a white tile
- 4) At an appropriate time, add 10cm3 of Na2S2O8 and start the stopwatch at the same time
- 5) Record the time taken for the solution to turn blue-black
- 6) Repeat steps 1 to 5 for experiments 2 to 5 with varying volumes of distilled water and Na2S2O8 according to the table below

Expt no.	Volume of	Volume of	Volume of	Volume of	Time, t/s	1/t/s-1
	KI/cm3	Na2S2O3/cm3	H2O/cm3	Na2S2O8,		
				V/cm3		
1	5	5	40	10		
2	5	5	30	20		
3	5	5	20	30		
4	5	5	10	40		
5	5	5	0	50		

Volume of S2O82- is proportional to [S2O82-]

- Since the same fixed conc of iodine is formed for each expt when the stopwatch is stopped
 Rate of reaction proportional to 1/t
- Since the total volume of the reaction mixture is kept constant for all the expts by adding appropriate volumes of water,
 - Initial [S2O82-] proportional to V

Calculations

- Compare expts 1 and 2: when V is doubled, 1/t does not change, rate does not change with change in conc of S2O82-, hence order of reaction wrt S2O82- = 0
- OR when V is doubled, 1/t is doubled: order of rxn=1
- OR when V is doubled, 1/t increased 4 times: order of rxn=2

Graphical method

- Plot graph of 1/t against V
 - Horizontal line: 0 order
 - Straight line with positive gradient, passes though origin: 1st order
- Plot graph of 1/t^2 against V^2
 - Straight line with a positive gradient and passes through origin: 2nd order

Quenching method

Uses of dilute cold sulfuric acid

- It acts as a quenching agent which stops the decomposition reaction of hydrogen peroxide
 - \circ $\;$ Cold: slows down the rate of decomposition reactions rapidly
 - o Dilute: dilutes the reaction mixture, slows down the rate of decomposition reaction

Procedure

- 1) Using a pipette, transfer 50.0cm3 of hydrogen peroxide into a 250cm3 beaker. To the same beaker, add 25cm3 of sodium hydroxide using a measuring cylinder
- 2) Place 50cm3 of iron (III) chloride into a second measuring cylinder
- 3) Add the solution of FeCl3 into the same beaker and start the stopwatch immediately. Stir the mixture using a glass to ensure even mixing
- 4) Using a pipette, withdraw 10.0cm3 of the reaction mixture into a conical flask. At time 2min, add 20cm3 of cold dilute sulfuric acid into t conical flask
- 5) Take further 10.0cm3 samples from the reaction mixture and transfer them into separate conical flasks, and add cold dilute sulfuric acid (as in step 4) at 4, 6, 8, 10 and 12 min respectively, after the initial mixing
- 6) Titrate the hydrogen peroxide present in each of the conical flasks obtained against aqueous potassium manganate (VII) placed in a 50.00cm3 burette.
- 7) Record the titration results in a suitable table.

Time from initial mixing/min	2	4	6	8	10	12
Final burette reading/cm3						
Initial burette reading/cm3						
Volume of KMnO4 used/cm3	V1	V2	V3	V4	V5	V6

Sketch graph of Volume of KMnO4 used against time

- Volume of KMnO4 used proportional to amt of KMnO4 reacted proportional to amt of H2O2 present in conical flask
- As volume fo aliquots taken were kept constant at 10.0cm3, amt of H2O2 present in the conical flask is proportional to [H2O2] in conical flask
- If graph is straight, downward sloping line, reaction is zero order wrt H2O2
- If graph is straight downward sloping curve with constant half-life, reaction is firs order wrt H2O2

Equilibrium

LCP: when a system in equilibrium is subjected to a change the system responds in a way which counteracts the effect of the change and re-establishes the equilibrium state

- Changes in concentration, pressure, temperature, addition of catalyst

Examples

- To determine Kc for hydrolysis of ester to give alcohol and acid
- Ka of a weakacid
- Ksp of sparingly soluble salt
- Kd (distribution coefficient) of a compound distributed between 2 immiscible solvents
- Kstab of a complex ion

Experimental methods

- Titrimetry (for acid-base, redox, or pptn)
- Colorimetry (colour intensity related to conc of a coloured species)

Examples

Ionisation constant Kin

Procedure

- 1) Use a 10cm3 measuring cylinder to transfer 5.0cm3 of bromophenol solution into a clean and dry test tube and add 1 drop of conc HCl. In this solution, the indicator exists almost entirely as HIn. Label this solution A
- 2) Use another 10 cm3 measuring cylinder to transfer 5.0 cm3 of bromophenol solution into a clean and dry test tube and add 1 drop of 4 moldm-3 sodium hydroxide solution. In this solution, the indicator exists almost entirely as In-. label this solution B
- 3) Arrange 18 test tubes in racks as shown int eh diagram below. Use a dropper to add drops of solutions A and B corresponding to the numbers in the diagram
- 4) Use a 10 cm3 measuring cylinder to add 10.0cm3 of deionised water to each tube and stir the contents to ensure that the colour is uniform from top to bottom
- 5) Use another 10cm3 measuring cylinder to transfer 10.0 cm3 of buffer solution into a testtube and add 10 drops of bromophenol blue solution. Shake the tube to mix the contents. Label this solution C
- 6) By holding the tube containing solution C alongside the pairs of tubes in the rack, find the pair of tubes whch give a colour that is closest to that of solution C. do this against a brightly lit white background. Record the value of [HIn(aq)]/[(In-(aq)) for that pair of tubes
- 7) The ionisation constant Kin can be calculated by substituting the pH value of the buffer and the value of [HIn(aq)]/[In-(aq)] determined in step 6 into the equation below (buffer equation)

Temperature of the contents in each test tube can be kept constant by partially submerging all test tubes in a water bath maintained at 25degC

Ksp

Preparing a saturated solution

- 1) Dissolve solid in water until some solid remains undissolved
- 2) Allow solution to stand for few hours to establish equilibrium

3) Filter to remove undissolved solid. The saturated solution is collected as the filtrate

Procedure

- 1) Prepare a saturated solution of X in water
- 2) Pipette 25.0 cm3 of the saturated solution X into a 250cm3 conical flask
- 3) Add 3 drops of MO indicator
- 4) Titrate with standard HCl placed in a 50.00cm3 burette until the indicator solution changed from yellow to orange
- 5) Repeat the expt to achieve consistent results
- 6) Repeat the expt using a saturated solution of X in water saturated with CO2 instead
- 7) Expected result: the volume HCl used for titration against a saturated solution of MgCO3 in water saturated with CO2 is greater compared to saturated solution of MgCO3

Kd

Determine value of Kd

- 1) Pour 50cm3 of 1moldm-3 aqueous ammonia into a separting funnel
- 2) Pour 50cm3 of organic solvent into the same separating funnel
- 3) Hold the tap firmly with one hand and the stopper with the other. Shake the separating funnel vigorously, with the tap pointing upwards for 15 seconds. Open the tap for a moment and then lose it.
 - a. Tap has to be opened momentarily as the vigorous shaking causes rapid vaporisation of organic solvent. The tap is opened momentarily to release the pressure inside the separating funnel
- 4) Repeat step 3 twice. Then set aside until 2 layers separate.
- 5) Transfer the organic layer to a beaker and the aqueous layer to another beaker.

Concentration of ammonia in each layer may be determined by titration

- 1) Fill up a burette with 50.00cm3 of 0.010moldm-3 of Hcl
- 2) Using a dry pipette, transfer 10.0cm3 of the organic layer into a 250cm3 conical flask
- 3) Then add 2-3 drops of methyl orange
- 4) Titrate the resulting mixture against 0.010 moldm-3 HCl until the colour of MO changes from yellow to orange
- 5) Repeat the titration until two sets of consistent readings are obtained

Electrochemistry

Measurement of standard electrode potential E

- 1) Set up the given half-cell and the SHE next to each other and ensure that all concentrations of solutions are 1moldm-3, temperature is 298K and pressure of gases is 1atm
- 2) Connect the two solutions in the two half-cells using a salt bridge, commonly made up of saturated potassium nitrate solution.
- 3) Measure the potential difference between the given half-cell and the SHE using a high-resistance voltmeter. The value shown on the voltmeter is the standard electrode potential (emf) of the half-cell concerned. (should take the first reading shown on the voltmeter the moment the 2 half-cells are connected as the actual emf bc the moment the circuit is completed, chemical reactions occurs and the conc of reactants will start to decrease and will no longer be at standard conditions.

Procedure

- 1) Assemble the apparatus, as shown in the diagram above, in a fume cupboard. The two graphite electrodes are inserted into a two-hole rubber bung and clamped to a retort stand.
- 2) Use a spatula to fill the crucible, almost to the brim, with powdered PbBr2
- 3) Insert the graphite rods into the solid PbBr2 such that the rods almost touch the bottom of the crucible
- 4) Light the Bunsen burner and use a non-luminous flame to heat the crucible strongly
- 5) When all the white solid has melted, switch on the power supply and adjust the current to 3.0A. Start a stopwatch at the same time. Continue heating the crucible and maintaining the current for 30 min
- 6) At the end of 30 min, switch off the power supply and remove the graphite rods.
- 7) Turn off the Bunsen burner and pour the molten PbBr2 into a second crucible while keeping the small bead of molten lead in the first crucible. Allow the contents in both crucibles to cool
- 8) When cooled, brush off the solid PbBr2 from the solidified bead of lead. Rinse the bead with hot deionised water
- 9) Dry the solid lead in a dessicator. Weigh the clean and dried bead of lead on an electronic balance.
- 10) Repeat the expt. Use the mass readings obtained to calculate 2 different values for avogadro's constant. Take the average o these 2 calculated values

Calculations

- Find out no. of e transferred
- Find out no. of Pb atoms
- Find out no. of moles of Pb in m grams
- Number of Pb atoms divided by no. of moles = avogadro's constant

Safety precautions

- Br2 gas which is toxic, is produced during the electrolysis. The expt must be carried out in a fume cupboard
- The crucible will be very hot after strong heating. Gloves or metal tongs should be used when handling the crucible.

Inorganic Qualitative analysis

Procedure

- Coherent and detailed sequence of plan

Solubility

- All sodium, potassium ammonium, nitrates are soluble
- All halides are soluble, except Pb and Ag. AgCl soluble in excess NH3
- All sulfates are soluble, except Ba and Pb
- All chromates are soluble, except P[b and Ba (yellow), copper and iron (brown), and silver (red)
- All carbonates and sulphites are insoluble
- All hydroxides are insoluble, except Gp1 and Gp 2 (except Mg). Hydroxides of Al, Pb, Zn, Cr are soluble in excess NaOH