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| **DEPARTMENT OF CHEMISTRY**  **FOURAH BAY COLLEGE – UNIVERSITY OF SIERRA LEONE** **CHEM 124**EXPERIMENTAL CHEMISTRY II **(to be taken alongside CHEM 121, CHEM 122 and CHEM 123)** | |
| **CREDIT HOURS** | **1.5** |
| **REQUIREMENTS** | C- or better in CHEM 114To be taken alongside CHEM 111, CHEM 122 and CHEM 123 |
| **REQUIRED FOR** | **CHEM 214** |

## INTRODUCTION

* CHEM 124 is a continuation of CHEM 114 and covers more of the core practical techniques used in Chemistry
* The course consists of a series of 10 practical experiments based on different chemical principles and introducing different practical techniques
* Working in small groups, students are required to perform a series of practical tasks during the course of each experiment
* Students are expected to keep a clear written record of all practical work carried out and all experiment results obtained.

## EQUIPMENT REQUIRED

Students must purchase the manuals required for CHEM 114 and CHEM 124 at the start of the academic year at a cost of Le 40,000; this cost includes a small caution fee used to cover the annual cost of replacing any broken glassware.

An official FBC Chemistry labcoat must be worn to every practical session. These can be purchased from the head technician at a cost of Le 70,000. Students will not be admitted into any practical session unless they are wearing an FBC Chemistry labcoat.

Safety glasses must also be worn to every practical session. These are widely available in town but can also be purchased from the head technician at a cost of Le 15,000. Students will be deducted marks if they are not wearing eye protection.

Students should also bring a pen, writing paper and a calculator to every practical session. Graph paper is also required for some reports.

**DRESS AND BEHAVIOUR EXPECTATIONS**

Students are not permitted to wear open footwear and long hair must be tied back. Casual, durable clothes and trainers are recommended.

Food and drink must not be consumed in any laboratory.

The chemicals and apparatus used in Chemistry can be harmful, especially if not handled correctly. Students are expected to act in a safe and responsible manner at all times. Ask the nearest staff member if you not sure how to handle apparatus or chemical safely.

If students break equipment with a value exceeding the annual contribution, the student is expected to replace the equipment at his/her own expense.

**ASSESSMENT**

**Continuous Assessment – 50%**

Students must submit a full report for each practical to the head technician by 12.00 pm on the Monday following the practical session. The report will only be accepted if the student has been marked present at both the beginning and the end of the practical and if their results have been signed by their allocated lecturer.

Most reports will be awarded a mark out of 10. Two reports per semester will be awarded a mark out of 20 and will be considered equivalent to two reports. The best eight reports achieved by each student over the semester will be added up to give a total out of 80. This mark will be scaled down to give a final continuous assessment mark out of 50.

**Practical Examination – 50%**

Students will sit a single practical examination at the end of the semester. This will consist of two practical exercises and some written questions. There are 70 marks available for the practical exam, which will be scaled down to give a final examination mark out of 50.

The mark from the continuous assessment and the mark from the practical examination are combined to give a total mark out of 100, which will determine the students’ overall grade for the module.

**PROCEDURE FOR PRACTICAL SESSIONS**

1. Students should arrive promptly for each session and sign in as they enter the laboratory; students may sign in from 08:30
2. At 09:00 the lecturers will introduce the main chemical concepts underlying the practical, demonstrate how to use any new apparatus and provide suggestions on how to best carry out the practical
3. At 09:30 the lecturers will ask the students to report to their work stations and check their equipment; each group will be briefed, issued with any additional equipment they need and be instructed to start the practical
4. Once all data has been collected, the students should ensure that their rough work is signed by the lecturer allocated to their work station and then clean and tidy their work station
5. Students should sign out with the head technician once their work station is clean and tidy; they should do this by 12:30
6. At 12:30 the lecturers will review the theory and provide guidance on completing the report
7. The session should finish promptly at 13:00

## REPORTS

* Each report should include a clear presentation of all observations, calculation and presentation of results and conclusions with all working shown clearly, and answers to any additional questions
* Rough work should be signed by a lecturer at the end of the session and should be submitted along with the report
* Marks will be awarded as follows:

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| Arrives by 9.00 am | 1 mark |
| Arrives before practical work begins | 1 mark |
| Arrives by 10.00 am | 1 mark |
| Arrives by 10.00 am and wears eye protection throughout practical session | 1 mark |
| Arrives by 10.00 am, wears appropriate clothing and behave appropriately throughout practical session | 1 mark |
| Report | 5 marks |
| TOTAL | 10 marks |

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| **Week** | **Experiment Title** |
| **1** | Organic I – Investigation of solubility and the presence of C=C bonds |
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| **3** | **Organic III – Investigation of carboxylic acids, esters and amines** |
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**Week 1 – Organic Chemistry I**

### Test 1: Solubility

**Introduction**

* The solubility of organic molecules in water and in non-polar solvents such as hexane varies considerably
* Molecules which contain “hydrophilic” groups such as -COOH, -OH and ionic groups tend to be soluble in water, as water molecules can form strong hydrogen bonds with these groups; the greater the abundance of these groups on the molecule, the greater the solubility in water
* Molecules which can form hydrogen bonds with water (and are therefore soluble in water) tend to have limited solubility in non-polar solvents, as these molecules cannot form hydrogen bonds with non-polar molecules and so bond to themselves in preference
* Molecules which contain “hydrophobic” groups such as large alkyl groups tend to have limited solubility in water, as water molecules cannot form strong hydrogen bonds with these groups and so bond to themselves in preference; the larger the alkyl groups, the lower the solubility in water

**Procedure**

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| **Test for solubility in water:**   * Place one drop (if liquid) or two grains (if solid) of the organic substance into a test tube * Add water, drop by drop, shaking well after each drop is added, until the organic substance has dissolved * Continue until a total of 6 drops of water has been added   **Test for solubility in non-polar substances:**   * Place one drop (if liquid) or two grains (if solid) of the organic substance into a test tube * Add hexane, drop by drop, shaking well after each drop is added, until the organic substance has dissolved * Continue until a total of 6 drops of hexane has been added   **Interpretation of Results:**   * if the substance dissolves in 1 or 2 drops of the solvent, it is very soluble in that solvent * if the substance requires 3 - 5 drops of the solvent before dissolving, it is partially soluble in that solvent * if the substance requires 6 or more drops the solvent before dissolving, it is sparingly soluble in that solvent |

1. You are provided with the following six organic compounds: propanone, pentan-3-one, cyclohexanone, ethanoic acid, butanoic acid and hexanoic acid. Test the solubility of each compound in water; record your results and conclusions in a suitable table.
2. You are provided with three bottles, labelled A, B and C. These three bottles contain ethanol, butan-1-ol and hexan-1-ol but it is not known which bottle contains which alcohol. Test the solubility of each compound in water; record your results and conclusions in a suitable table. Hence identify which alcohol is in which bottle.
3. You are provided with a further eight organic compounds: trichloromethane, oleic acid, benzoic acid, ethyl ethanoate, propan-1-ol, methylbenzene, phenol and butanal. Test the solubility of each compound in both water and hexane; record your results and conclusions in a suitable table.

### Test 2: Test for Unsaturation

**Introduction**

* Alkenes contain a C=C double bond; organic molecules which contain a C=C double bond are said to be unsaturated
* The C=C bond in alkenes can undergo an electrophilic addition reaction with Br2 to give a dibromoalkane; the orange colour of the Br2 is rapidly decolorised during this reaction and this can be used as a test to identify alkenes
* There are some molecules other than alkenes which decolorise bromine water; phenols, carbonyls and some other functional groups can undergo substitution reactions with bromine, and all molecules can undergo free radical substitution reactions with bromine in the presence of sunlight; these reactions, however, tend to take place more slowly and therefore result in the gradual decolorisation of bromine; this can be contrasted with the rapid decolorisation of bromine observed in electrophilic addition reactions

**Procedure**

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| **Test for unsaturation:**   * Place one drop (if liquid) or two grains dissolved in ethanol (if solid) of the organic substance into a test tube * Add one drop of bromine water and shake well   **Interpretation of Results:**   * if the mixture decolorises rapidly, the organic substance contains a C=C bond * if the mixture decolorises slowly, the organic substance is likely to contain a phenol or carbonyl compound |

1. You are provided with the following five organic compounds: hexane, cyclohexene, cinnamic acid, ethanol and maleic acid. Carry out the unsaturation test on each compound; record your results and conclusions in a suitable table.
2. You are provided with three further organic compounds: phenol, benzophenone and propanone. Carry out the unsaturation test on each compound; record your results and conclusions in a suitable table.
3. How did your observations for phenol, benzophenone and propanone compare with those for cyclohexene, cinnamic acid and maleic acid?

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| Report  1. Record all your observations and conclusions clearly in a series of tables 2. Draw the structures of all the organic compounds you tested 3. Use your structures to explain your observations and conclusions for each test |

**Week 2 – Organic Chemistry II**

Even-numbered groups should carry out tests 3, 4, 5 and 6 in any order and then carry out test 7.

Odd-numbered groups should carry out test 7 and then tests 3, 4, 5 and 6 in any order.

### Test 3: Oxidation of Primary Alcohols, Secondary Alcohols and Aldehydes

* Primary alcohols can be oxidised to aldehydes; secondary alcohols can be oxidised to ketones; tertiary alcohols cannot be oxidised: R1R2CHOH + [O] 🡪 R1COR2 + H2O
* Aldehydes can be further oxidised to carboxylic acids but ketones cannot be oxidised: R1CHO + [O] 🡪 R1COOH
* Acidified potassium dichromate is a good reagent to use for this reaction; during the reaction, the orange Cr2O72- ion is reduced to the dark green Cr3+ ion and this reaction is clearly visible in a test tube
* Acidified potassium dichromate can therefore be used as a test for primary alcohols, secondary alcohols and aldehydes (it cannot distinguish between these groups)

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| **Test 3 Procedure:**   * Pour 5 drops of the organic compound into a test-tube * Add 2 drops of potassium dichromate solution * Add 10 drops of dilute sulphuric acid and shake gently * Leave to stand in a beaker of hot water for 10 minutes   **Interpretation of Results:**   * if the mixture turns green or dark brown, the organic substance is either a primary alcohol, secondary alcohol or aldehyde |

### Tests 4 and 5: Oxidation of Aldehydes

* The oxidation of aldehydes to carboxylic acids can be achieved with much milder oxidising agents than those needed to oxidise alcohols to carbonyl compounds; mild oxidising agents such as Fehling’s solution and Tollen’s reagent are able to oxidise aldehydes to carboxylic acids without also oxidising alcohols; they are therefore effective as a specific test for aldehydes:
* In Fehling’s solution (Test 4) the blue Cu2+ ion is reduced to the orange precipitate of Cu2O
* In Tollen’s reagent (Test 5) the colourless Ag+ ion is reduced to metallic silver

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| **Test 4 Procedure:**   * Pour 1 cm3 of Fehling’s solution 1 into a test tube * Add Fehling’s solution 2 dropwise until the precipitate just dissolves * Add ten drops of the organic compound and shake gently * Leave to stand in a beaker of boiling water for 10 minutes   **Interpretation of Results:**   * if an orange precipitate appears, the organic compound contains an aldehyde group |
| **Test 5 Procedure:**   * Pour 1 cm3 of Tollen’s reagent into a test tube * Add two drops of the organic compound and shake gently * Leave to stand in a beaker of boiling water for 10 minutes   **Interpretation of Results:**   * if a silver mirror or black precipitate appears, the organic compound contains an aldehyde group |

**Test 6: Condensation reactions of aldehydes and ketones**

* Aldehydes and ketones react with 2,4-dinitrophenylhydrazine to give a yellow or orange precipitate known as a hydrazone derivative: R1COR2 + C6H3N2O4NHNH2 🡪 R1COR2 + C6H3N2O4NHNCR1R2 + H2O; the precise colour of the precipitate depends on the carbonyl compound used; this reaction is a good test for carbonyl compounds (it works on aldehydes and ketones but not alcohols)

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| **Test 6 Procedure:**   * Dissolve five drops of the organic substance in 0.5 cm3 of ethanol * add 3 cm3 of 2,4-dinitrophenylhydrazine solution * shake the mixture for a few minutes   **Interpretation of Results:**   * if an orange or yellow precipitate appears, an aldehyde or a ketone is present |

1. You are provided with four unknown organic compounds, labelled A, B, C and D. The compounds are butan-1-ol, methylpropan-2-ol, butanal and butanone. Carry out tests 3 – 6 on each compound and hence identify which bottle contains which compound.
2. You are provided with one more unknown organic compound; this will either be labelled E, F, G or H. The compound is either 4-methylpentan-2-one, phenylethanone, cyclohexanone, 2-hydroxybenzaldehyde and 3-methylbutanal. Carry out either Test 4 or Test 5 on your compound to determine whether it is an aldehyde or a ketone.

**Test 7: Identification of a carbonyl compound by measuring the melting point of its 2,4-DNP-hydrazone**

* The reaction of each different carbonyl compound with 2,4-DNPH gives a different precipitate; the melting point of each derivative varies considerably so if the precipitate can be purified (by recrystallisation) and its melting point measured, it can be identified by comparing its melting point to those of known derivatives
* The melting points of the derivatives of E, F, G, H and I are as follows:

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| Carbonyl | Melting point of derivative |
| 4-methylpentan-2-one | 124o |
| Phenylethanone | 248o |
| Cyclohexanone | 162o |
| 2-hydroxybenzaldehyde | 252o (it also decomposes) |
| 3-methylbutanal | 182o |

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| **Test 7 Procedure:**   * Dissolve 10 drops of the organic substance in 1 cm3 of ethanol in a test tube * add 5 cm3 of 2,4-dinitrophenylhydrazine solution * shake the mixture until a precipitate forms * filter the mixture and scrape the residue into a boiling tube * dissolve the residue in the minimum possible quantity of hot ethanol * allow the solution to cool until the derivative crystallises * filter the mixture again * dry the residue in more filter paper and place the residue into an oven until it is completely dry * take a small sample of the residue and place it into a capillary tube * measure its melting point   **Interpretation of Results:**   * the organic substance can be identified as that whose derivative has a melting point closest to the one you have observed |

1. You will be given one of E, F, H, H and I to identify. Carry out test 7 on the compound you are given and hence identify it

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| Report  1. Record all your observations and conclusions clearly in a series of tables 2. Identify A, B, C and D, giving your reasons clearly 3. Use Test 7 to identify E, F, G or H, giving your reasons clearly |

## Week 3 – Organic Chemistry III

**Test 8: The presence of H+ ions in carboxylic acids and esters**

* Carboxylic acids and phenols dissociate partially in water to give H+ ions and carboxylate ions:

R-COOHR-COO- + H+ (carboxylic acids)

C6H5OHC6H5O- + H+ (phenols)

The presence of H+ ions can be detected with a simple acid-base indicator; phenolphthalein is suitable for this purpose; it is colourless in acidic solutions and pink in alkaline solutions

* Esters are gradually hydrolysed in water to produce carboxylic acids and alcohols:

R1-COOR2 + H2O R1-COOH + R2-OH

As a result esters often contain enough H+ ions to decolorise phenolphthalein

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| **Test 8 Procedure:**   * Add 1 cm3 of the organic compound to a test tube (if solid, use a small spatula measure) * add 2 drops of phenolphthalein indicator and swirl the mixture   **Interpretation of Results:**   * if the purple colour disappears, an acid, phenol or ester is present   (Note: keep your sample for test 9) |

**Test 9: Distinguishing between carboxylic acids and esters**

* When hydroxide ions are added to carboxylic acids, they are converted into carboxylate salts:

R-COOH + OH- 🡪 R-COO- + H2O

This reaction is irreversible and results in the conversion of all of the carboxylic acid to the salt

* When hydroxide ions are added to esters, any R-COOH present in the ester is neutralised**;** if the mixture is then warmed, however, the ester will continue to be hydrolysed and will produce more carboxylic acid; as a result the purple colour will disappear

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| **Test 9 Procedure:**   * Add NaOH, drop by drop, to your solution from Test 8, until the mixture turns purple again * Warm the solution for a few minutes   **Interpretation of Results:**   * if the purple colour disappears again, an ester is present |

**Test 10: The presence of OH- ions in amines**

* Amines dissociate partially in water to give hydroxide ions and alkylammonium ions:

R-NH2 + H2O 🡪 R-NH3+ + OH-

The presence of OH- ions can be detected with a simple acid-base indicator; bromothymol blue is suitable for this purpose; it is yellow in acidic solutions and blue in alkaline solutions

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| **Test 10 Procedure:**   * Add 1 cm3 of the organic compound to a test tube (if solid, use a small spatula measure) * add 2 drops of bromothymol blue indicator and swirl the mixture   **Interpretation of Results:**   * if the blue colour appears, an amine is present |

You are given eight organic compounds, labelled A, B, C, D, E, F, G and H; in no particular order, they are phenol, ethanol, ethyl ethanoate, phenyl benzoate, cyclohexylamine, butylamine, ethanoic acid and benzoic acid

1. Carry out Test 8 on all eight compounds; if Test 8 is positive, carry out Test 9.
2. Carry out Test 10 on all eight compounds.
3. Hence draw any conclusions you can about the identity of compounds A – H.

**Test 11: Distinguishing between aromatic and aliphatic compounds by burning**

* Almost all organic compounds are flammable; the type of flame depends on the % by mass of carbon present in the compound; the higher the % carbon, the sootier the flame
* Aromatic compounds have a high % carbon and burn with a very sooty flame; aliphatic compounds have a lower % carbon and burn with a much less sooty flame

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| **Test 11 Procedure:**   * Take a few drops of the organic compound and place on a spatula (if solid, use a small spatula measure) * place the compound in a Bunsen flame until it ignites   **Interpretation of Results:**   * if the flame is very sooty, an aromatic compound is present; otherwise, an aliphatic compound is present |

1. Carry out Test 11 on methylbenzene, then on hexane, and then on compounds A, B, G and H
2. Hence draw any conclusions you can about the identity of compounds A – H.

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| Report  1. Record all your observations and conclusions clearly in a series of tables 2. Draw the structures of all of the organic compounds A - H 3. Use your structures to explain your observations and conclusions for each test 4. Identify compounds A – H as far as you can |

## Week 4 – Organic Chemistry IV

* In this final practical session on organic chemistry you will use a combination of the different tests you used in the first three sessions to identify the main functional groups present in a series of compounds; you will have the following tests available:

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| **Test 1: Solubility in water:**   * Place one drop (if liquid) or two grains (if solid) of the organic substance into a test tube * Add water, drop by drop, shaking well after each drop is added, until the organic substance has dissolved * Continue until a total of 6 drops of water has been added   **Interpretation of Results:**   * if the substance dissolves in 1 or 2 drops of the solvent, it is very soluble in that solvent * if the substance requires 3 - 5 drops of the solvent before dissolving, it is partially soluble in that solvent * if the substance requires 6 or more drops the solvent before dissolving, it is sparingly soluble in that solvent * soluble organic compounds tend to be alcohols or carboxylic acids with short hydrocarbon chains |

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| **Test 2: Unsaturation (C=C bond):**   * Place one drop (if liquid) or two grains dissolved in ethanol (if solid) of the organic substance into a test tube * Add one drop of bromine water and shake well   **Interpretation of Results:**   * if the mixture decolorises rapidly, the organic substance contains a C=C bond * if the mixture decolorises slowly, the organic substance is likely to contain a phenol or carbonyl compound |

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| **Test 3: Reducing agents**   * Pour 5 drops of the organic compound into a test-tube * Add 2 drops of potassium dichromate solution * Add 10 drops of dilute sulphuric acid and shake gently * Leave to stand in a beaker of hot water for 10 minutes   **Interpretation of Results:**   * if the mixture turns green or dark brown, the organic substance is either a primary alcohol, secondary alcohol or aldehyde |

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| **Test 4: Aldehydes**   * Pour 1 cm3 of Fehling’s solution 1 into a test tube * Add Fehling’s solution 2 dropwise until the precipitate just dissolves * Add ten drops of the organic compound and shake gently * Leave to stand in a beaker of boiling water for 10 minutes   **Interpretation of Results:**   * if an orange precipitate appears, the organic compound contains an aldehyde group |
| **Test 6: Carbonyls**   * Dissolve five drops of the organic substance in 0.5 cm3 of ethanol * add 3 cm3 of 2,4-dinitrophenylhydrazine solution * shake the mixture for a few minutes   **Interpretation of Results:**   * if an orange or yellow precipitate appears, an aldehyde or a ketone is present |

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| **Test 8: Carboxylic acids and Esters**   * Add 1 cm3 of the organic compound to a test tube (if solid, use a small spatula measure) * add 2 drops of phenolphthalein indicator and swirl the mixture   **Interpretation of Results:**   * if the purple colour disappears, an acid, phenol or ester is present   (Note: if the test is positive, keep your sample for test 9) |

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| **Test 9: Esters**   * Add NaOH, drop by drop, to your solution from Test 8, until the mixture turns purple again * Warm the solution for a few minutes   **Interpretation of Results:**   * if the purple colour disappears again, an ester is present |

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| **Test 10: amines**   * Add 1 cm3 of the organic compound to a test tube (if solid, use a small spatula measure) * add 2 drops of bromothymol blue indicator and swirl the mixture   **Interpretation of Results:**   * if the blue colour appears, an amine is present |

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| **Test 11: Combustion flame**   * Take a few drops of the organic compound and place on a spatula (if solid, use a small spatula measure) * place the compound in a Bunsen flame until it ignites   **Interpretation of Results:**   * if the flame is very sooty, an aromatic compound is present; otherwise, an aliphatic compound is present |

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| Task You are given ten unknown organic compounds, labelled A – J. Your task is to identify the functional group in each, indicate whether the compound is aromatic and suggest a possible chain length for each compound.  You do not have to carry every test out on every compound, but you must carry out at least enough test to identify the functional group in each. |

##### Week 5 – Calorimetry I

* The **enthalpy change of reaction** is the enthalpy change when reactants are converted into products in the molar quantities shown in the chemical equation:

Eg the enthalpy of reaction for

2AgNO3 + Zn 🡪 Zn(NO3)2 + 2Ag

Is the enthalpy change when two moles of AgNO3 react with one mole of Zn to produce one mole of Zn(NO3)2 and two moles of Ag

* The **enthalpy of neutralisation** is the enthalpy change when one mole of water is formed from the reaction between an acid and a base
* If a chemical reaction takes place in aqueous solution, the heat change can be measured directly using the following equation: q = m x c x ΔT
* q is the heat change in Joules
* m is the mass of the solution in grams (generally taken to be the mass of water, which is equal to the volume of water in cm3 x density of water (1.0 gcm-3))
* c is the **specific heat capacity** of the solution in Jg-1K-1, generally assumed to be equal to the specific heat capacity of water which is 4.18 Jg-1K-1
* ΔT is the temperature change in K or oC
* The **molar enthalpy change** can be calculated by dividing the heat change by the moles of any reactant or product in the equation with a stoichiometric coefficient of 1 (ΔH = q/n)
* A container used to measure the enthalpy change of a reaction is called a **calorimeter**; calorimeters should be made of insulating material so that the heat produced by the reaction is not lost to the surroundings; some of the energy is absorbed by the calorimeter itself and this can be taken into account if the mass and specific heat capacity of the calorimeter are known; for the purposes of this experiment, the calorimeter can be taken to have a specific heat capacity of 0.42 Jg-1K-1; using this value, a more accurate value for q can be calculated as follows: q = (mwcw + mccc)ΔT, where mw is the mass of water, c is the specific heat capacity of water, mc is the mass of the calorimeter and cc is the specific heat capacity of the calorimeter

**Note: First carry out the first two steps of Experiment 3. Then carry out Experiments 1 and 2 in any order, then complete the remainder of Experiment 3.**

**Experiment 1: measuring the enthalpy of neutralisation of 2.0 moldm-3 hydrochloric acid by 2.0 moldm-3 sodium hydroxide**

Equipment needed: calorimeter or polystyrene cup, two 25.0 cm3 pipettes, pipette filler, two small beakers, thermometer, access to 2.0 moldm-3 HCl, access to 2.0 moldm-3 NaOH

1. Weigh the empty calorimeter (if you are performing this experiment first)
2. Use a pipette to add 25 cm3 of 2.0 moldm-3 HCl to the calorimeter
3. Record the initial temperature of the solution
4. Use another pipette to add 25 cm3 of 2.0 moldm-3 NaOH to the calorimeter; replace the lid of the calorimeter and monitor the change in temperature; note the maximum temperature reached
5. Hence calculate the temperature change during the reaction

**Experiment 2: measuring the enthalpy of reaction between coper sulphate solution and zinc**

Equipment needed: calorimeter or polystyrene cup, 25.0 cm3 pipette, pipette filler, one small beakers, thermometer, access to 2.0 moldm-3 CuSO4, access to zinc powder with spatula, access to 2 dp mass balance

1. Weigh the empty calorimeter (if you are performing this experiment first)
2. Use a pipette to add 25 cm3 of 1.0 moldm-3 CuSO4 to the calorimeter
3. Record the initial temperature of the solution
4. Weigh out 2.0 – 2.5 g of zinc powder
5. Pour the zinc powder into the calorimeter; replace the lid of the calorimeter and monitor the change in temperature; note the maximum temperature reached

**Experiment 3: Measuring the relative atomic mass of an unknown metal**

* The Dulong-Petit law states that the heat capacity (C) of a solid element is approximately equal to 27 JK-1 per mole of that element; this law can be used to estimate the relative atomic mass of solid elements:

C/n = 27, but C = c x m so = 27 so c x mr = 27 so **mr =**  (mr = molar mass in gmol-1 and c = specific heat capacity in JK-1g-1)

* The relative atomic mass of a solid element can therefore be calculated if its specific heat capacity can be measured
* If a hot solid of known temperature (T1) is dropped into cold water of temperature T2, heat will be transferred from the solid to the water (and the calorimeter); the heat lost by the solid will be equal to the heat gained by the calorimeter and contents and the final temperature (T3) will be higher than T2 but lower than T1.

**mece(T1 – T3) = mccc(T3 – T2) + mwcw(T3 – T2)**

me = mass of element, ce = specific heat capacity of element

mc = mass of calorimeter, cc = specific heat capacity of calorimeter (0.42)

mw = mass of water, cw = specific heat capacity of water (4.18)

* If T1, T2, T3, m1 and m2 are measured, ce and hence mr can be calculated

Equipment needed: calorimeter or polystyrene cup, 50 cm3 measuring cylinder, thermometer, metal block, access to 2 dp mass balance, access to boiling water bath

1. Weigh the metal block you are provided with
2. Place the metal block in a beaker of boiling water and leave it in there for at least 30 minutes (in the meantime, carry out Experiments 1 and 2)
3. Use a measuring cylinder to add 50 cm3 of water to the calorimeter
4. Weigh the calorimeter again to calculate the exact mass of water added
5. Record the initial temperature of the water (T2)
6. Record the temperature of the boiling water in which the metal block is immersed (T1)
7. Remove the metal block from the boiling water and drop it into the calorimeter
8. Record the final temperature reached (T3)

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| **Report**   1. Calculate the heat change during Experiment 1 2. Calculate the number of moles of copper sulphate and zinc used, and hence deduce which is the limiting reactant 3. Hence calculate the molar enthalpy change for the reaction CuSO4(aq) + Zn(s) 🡪 ZnSO4(aq) + Cu(s) 4. Calculate the heat change during Experiment 2 5. Calculate the number of moles of sodium hydroxide and hydrochloric acid used, and hence deduce which is the limiting reactant 6. Hence calculate the molar enthalpy change for the reaction HCl(aq) + NaOH(aq) 🡪 NaCl(aq) + H2O(l) 7. Calculate the heat gained by the calorimeter and its contents during Experiment 3 8. Hence calculate the specific heat capacity, and the relative atomic mass, of the metal |

##### Week 6 – Calorimetry II

**Experiment 1: Determine the enthalpy change for the following reaction: CaCO3(s) 🡪 CaO(s) + CO2(g)**

* The enthalpy change for this decomposition reaction is difficult to measure directly; it is, however, possible to measure the enthalpy change of the following reactions directly:

CaCO3(s) + 2HCl(aq) 🡪 CaCl2(aq) + CO2(g) + H2O(l) (Reaction 1)

CaO(s) + 2HCl(aq) 🡪 CaCl2(aq) + H2O(l) (Reaction 2)

* From the measurement of these two enthalpy changes, it is possible to determine the enthalpy change for the decomposition of calcium carbonate using Hess’ Law
* The equipment you will need is: one 250 cm3 plastic cup, one 250 cm3 beaker, one thermometer, one 25.0 cm3 pipette, access to a mass balance (minimum 2 dp), access to 2 moldm-3 HCl, access to CaCO3 powder, access to CaO powder

1. Use a pipette to transfer 25 cm3 of 2 moldm-3 HCl into a clean dry plastic cup.

2. Place the plastic cup in a beaker and record the temperature of the acid to the nearest 0.5 oC.

3. Weigh out accurately between 2.50 and 3.00 g of calcium carbonate powder. Record your mass in a suitable table.

4. Add the calcium carbonate to the acid. Stir the mixture carefully and record the maximum temperature reached (if the temperature rises) or the minimum temperature reached (if the temperature drops) to the nearest 0.5 oC.

5. Repeat steps 1 – 4, but using 2.50 – 3.00 grams of calcium oxide instead of the calcium carbonate.

**Experiment 2: Determine the enthalpy change for the following reaction: 2NaHCO3(s) 🡪 Na2CO3(s) + CO2(g) + H2O(l)**

* The enthalpy change for this decomposition reaction is difficult to measure directly; it is, however, possible to measure the enthalpy change of the following reactions directly:

NaHCO3(s) + HCl(aq) 🡪 NaCl(aq) + CO2(g) + H2O(l) (Reaction 3)

Na2CO3(s) + 2HCl(aq) 🡪 2NaCl(aq) + CO2(g) + H2O(l) (Reaction 4)

* From the measurement of these two enthalpy changes, it is possible to determine the enthalpy change for the decomposition of calcium carbonate using Hess’ Law
* The equipment you will need is: one 250 cm3 plastic cup, one 250 cm3 beaker, one thermometer, one 25.0 cm3 pipette, access to a mass balance (minimum 2 dp), access to 2 moldm-3 HCl, access to NaHCO3 powder, access to anhydrous Na2CO3 powder

6. Repeat steps 1 – 4, but using 2.50 – 3.00 grams of sodium hydrogencarbonate instead of the calcium carbonate.

7. Repeat steps 1 – 4, but using 2.50 – 3.00 grams of anhydrous sodium carbonate instead of the calcium carbonate.

**Experiment 3: Determine the enthalpy change for the following reaction: Na2CO3.10H2O(s) 🡪 Na2CO3(s) + 10H2O(l)**

* The enthalpy change for this decomposition reaction is difficult to measure directly; it is, however, possible to measure the enthalpy change of the following reactions directly:

Na2CO3(s) + 2HCl(aq) 🡪 2NaCl(aq) + CO2(g) + H2O(l) (Reaction 4)

Na2CO3.10H2O(s) + 2HCl(aq) 🡪 2NaCl(aq) + CO2(g) + H2O(l) (Reaction 5)

* From the measurement of these two enthalpy changes, it is possible to determine the enthalpy change for the decomposition of calcium carbonate using Hess’ Law
* The equipment you will need is: one 250 cm3 plastic cup, one 250 cm3 beaker, one thermometer, one 25.0 cm3 pipette, access to a mass balance (minimum 2 dp), access to 2 moldm-3 HCl, access to hydrated Na2CO3 powder, access to anhydrous Na2CO3 powder

8. Repeat steps 1 – 4, but using 2.50 – 3.00 grams of hydrated sodium carbonate instead of the calcium carbonate.

**Experiment 4: Determine the enthalpy change for the following reaction: CuSO4(s).5H2O(l) 🡪 CuSO4(s) + 5H2O(l)**

* The enthalpy change for this decomposition reaction is difficult to measure directly; it is, however, possible to measure the enthalpy change of the following reactions directly:

CuSO4(s) 🡪 Cu2+(aq) + SO42-(aq) (Reaction 6)

CuSO4.5H2O(s) 🡪 Cu2+(aq) + SO42-(aq) + 5H2O(l) (Reaction 7)

* From the measurement of these two enthalpy changes, it is possible to determine the enthalpy change for the decomposition of calcium carbonate using Hess’ Law
* The equipment you will need is: one 250 cm3 plastic cup, one 250 cm3 beaker, one thermometer, one 50 cm3 measuring cylinder, access to a mass balance (minimum 2 dp), access to hydrated CuSO4 powder, access to anhydrous CuSO4 powder, access to distilled water

9. Weigh a clean dry plastic cup.

10. Using a measuring cylinder, pour approximately 25 cm3 of distilled water into the cup.

11. Weight the cup again with the water in it.

12. Place the plastic cup in a beaker and record the temperature of the acid to the nearest 0.5 oC.

13. Weigh out accurately between 1.00 and 1.50 g of anhydrous copper sulphate powder. Record your mass in a suitable table.

14. Add the anhydrous copper sulphate to the water. Stir the mixture carefully and record the maximum temperature reached (if the temperature rises) or the minimum temperature reached (if the temperature drops) to the nearest 0.5 oC.

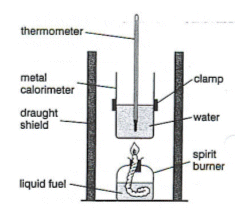
15. Repeat steps 9 – 14, but using 1.00 – 1.50 grams of hydrated copper sulphate instead of the anhydrous copper sulphate.

|  |
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| **Report**   * Your report should include all of your mass readings and temperature readings for all four experiments. * Your report should include calculations of the enthalpy changes of the following four reactions, with full working shown:   **CaCO3(s) 🡪 CaO(s) + CO2(g) (Experiment 1)**  **2NaHCO3(s) 🡪 Na2CO3(s) + CO2(g) + H2O(l) (Experiment 2)**  **Na2CO3.10H2O(s) 🡪 Na2CO3(s) + 10H2O(l) (Experiment 3)**  **CuSO4(s).5H2O(l) 🡪 CuSO4(s) + 5H2O(l) (Experiment 4)**  You should proceed as follows:   * Use the temperature change and the mass of the solution to calculate the heat transferred during each reaction (assume that the solutions behave as pure water with a density of 1.0 gcm-3 and a specific heat capacity of 4.18 JK-1 g-1) * For each reaction, calculate the number of moles of each reactant and hence assume which of the reactants is limiting * Hence calculate the molar enthalpy change for each of reactions 1 – 7 * Derive Hess’ law cycles to show how the molar enthalpy change for the four decomposition reactions above can be expressed in terms of the molar enthalpy changes for reactions 1 – 7 * Hence calculate the molar enthalpy change for each of the four decomposition reactions |

##### Week 7 – Calorimetry III

##### Measuring and Comparing the Enthalpies of Combustion of different alcohols

* The **enthalpy of combustion** of a substance is the heat energy change when one mole of a substance is completely burned in excess oxygen
* If a substance is readily flammable and liquid, its enthalpy of combustion can be measured directly using a spirit burner and a copper can
* Ethanol (C2H6O), propan-1-ol (C3H8O), butan-1-ol (C4H10O), pentan-1-ol (C5H12O) and hexan1-ol (C6H14O) can all be measured in this way:



* a copper can is used is it is a good conductor of heat, so heat can pass efficiently from the flame to the water
* the initial temperature of the water should be recorded
* the initial mass of the spirit burner and fuel should be recorded with the lid on
* the spirit burner can be lit and the fuel allowed to burn until the desired temperature change has been reached
* the spirit burner can be put out by putting the lid on
* the spirit burner can be re-weighed and the mass of fuel burned can be calculated
* the heat change q = mcΔT and the enthalpy change = q/n

Equipment list: 100 cm3 measuring cylinder, copper can, stand, clamp, boss, spirit burners containing ethanol, propan-1-ol, butan-1-ol, pentan-1-ol and hexan-1-ol, thermometer

1. Using a measuring cylinder, pour 100 cm3 of water into a copper calorimeter
2. Mount the calorimeter on a clamp stand and record its initial temperature
3. Take a spirit burner containing one of the alcohols, and record its mass to two decimal places with the lid on
4. Remove the lid, place the spirit burner under the calorimeter and surround it with a windshield to protect it from wind currents
5. Light the spirit burner and allow it to heat the copper calorimeter until the temperature of the water has increased by 30 oC
6. Put out the spirit burner by putting the lid back on
7. Record the final temperature of the water
8. Weigh the spirit burner again and record the loss in mass
9. Repeat steps 1 – 7
10. Repeat steps 1 – 8 using each of the other alcohols

|  |
| --- |
| **Report:**   1. Use the equation q = mc∆T to calculate the heat transferred from the spirit burner to the water in each experiment 2. Use the change in mass of the spirit burner and the molecular formula of the alcohol to calculate the number of moles of alcohol used 3. Hence calculate the enthalpy of combustion of each alcohol 4. Identify and explain the trend in the enthalpy of combustion of the alcohols 5. Estimate the percentage apparatus error in any **one** of your experiments 6. Find out the known values for the combustion of the different alcohols from a data book and hence deduce the percentage error in your answers 7. Compare the apparatus error to the error in your answer and comment on your results 8. Identify the main source of error other than apparatus error and suggest how the accuracy of your experiment could be improved |

##### Week 8 – Determining a Rate Equation Using the Initial Rates Method

#### Introduction

* Sodium thiosulphate reacts with hydrochloric acid as follows:

Na2S2O3(aq) + 2HCl(aq) 🡪 S(s) + SO2(g) + H2O(l)

The sulphur produced in this reaction forms a precipitate which turns the mixture opaque; the rate of this reaction can be monitored by measuring the time taken for a cross under the reaction mixture to cease to be visible

* The rate of reaction is inversely proportional to the time taken for the reaction to reach a particular stage:

Relative rate of reaction =

* The rate equation for a chemical reaction is an equation which links the rate of reaction to the concentrations of the reactants (eg rate = k[A]x[B]y)
* The aim of this experiment is to find the order of reaction with respect to Na2S2O3 and HCl; this can be done by carrying out the reaction using different molarities of Na2S2O3 and HCl and investigating how the rate of reaction changes in each case

**Background**

* The order of reaction with respect to each reactant can be deduced by plotting a graph of concentration vs initial rate (an **initial rate-concentration graph**)
* In first-order reactions, rate = k[A]; a graph of initial rate against initial concentration will be a straight line through the origin of gradient k:



* In second-order reactions, rate = k[A] 2; a graph of initial rate against initial concentration will be a curve through the origin:



* In zero order reactions, rate = k; a graph of initial rate against initial concentration will be a horizontal line:



**Procedure**

1. Take a piece of filter paper and use a thick pen to draw an X on it.
2. Take a 250 ml conical flask and place it on top of the X on the filter paper.
3. Take a 50 cm3 measuring cylinder and label it “HCl”. Take another 50 cm3 measuring cylinder and measure it “Na2S2O3”.
4. Measure 50 cm3 of 0.25 moldm-3 Na2S2O3 into the labelled measuring cylinder and then pour it into the conical flask.
5. Measure out 50 cm3 of 2.0 moldm-3 HCl into the other labelled measuring cylinder. Pour it into the conical flask and start the stopclock immediately.
6. Record the time taken for the cross to disappear. This is experiment 1.
7. Rinse out the conical flask and repeat the experiment using different concentrations of Na2S2O3 and HCl as follows:

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
|  | “HCl cylinder” | | | “Na2S2O3 cylinder” | | |
| Experiment Number | Volume of HCl/cm3 | Volume of water/cm3 | [HCl]/  moldm-3 | Volume of Na2S2O3/cm3 | Volume of water/cm3 | [Na2S2O3] /moldm-3 |
| 1 | 50 | 0 | 2.0 | 50 | 0 | 0.25 |
| 2 | 50 | 0 |  | 40 | 10 |  |
| 3 | 50 | 0 |  | 30 | 20 |  |
| 4 | 50 | 0 |  | 20 | 30 |  |
| 5 | 50 | 0 |  | 10 | 40 |  |
| 6 | 40 | 10 |  | 30 | 20 |  |
| 7 | 30 | 20 |  | 30 | 20 |  |
| 8 | 20 | 30 |  | 30 | 20 |  |
| 9 | 10 | 40 |  | 30 | 20 |  |

|  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| **Report**   1. Complete the table above to show the initial molarity of Na2S2O3 and HCl in each experiment 2. Using the results of your experiments, calculate 1/time taken for each experiment and put your results in a table as follows:  |  |  |  |  |  | | --- | --- | --- | --- | --- | | Experiment | [Na2S2O3] /moldm-3 | [HCl]/moldm-3 | Time taken/s | 1/time taken (s-1) | | 5 |  |  |  |  | | 4 |  |  |  |  | | 3 |  |  |  |  | | 2 |  |  |  |  | | 1 |  |  |  |  |  |  |  |  |  |  | | --- | --- | --- | --- | --- | | Experiment | [Na2S2O3] /moldm-3 | [HCl]/moldm-3 | Time taken/s | 1/time taken (s-1) | | 9 |  |  |  |  | | 8 |  |  |  |  | | 7 |  |  |  |  | | 6 |  |  |  |  | | 3 |  |  |  |  |  1. Plot a graph of rate of reaction against [Na2S2O3] using experiments 1 – 5 2. Plot a graph of rate of reaction against [HCl] using experiments 3 and 6 – 9 3. Use your graphs to deduce the orders of reaction with respect to HCl and Na2S2O3, write a rate equation for the reaction and calculate the rate constant. |

**Week 9 - Determining The Activation Energy Of Two Reactions**

**Introduction**

* **Experiment 1**
* Persulfate ions reacts with iodide ions to form iodine:

**S2O82-(aq) + 2I-(aq) 🡪 I2(aq) + 2SO42-(aq)**

* The iodine produced reacts with thiosulphate ions:

2S2O32-(aq) + I2(aq) 🡪 S4O62-(aq) + 2I-(aq)

- If persulfate ions react with iodide ions in the presence of a small quantity of thiosulfate ions, no iodine is detected initially as the iodine reacts instantly with the thiosulfate ions; iodine only appears when all of the thiosulfate is used up; if starch is present, the presence of iodine will turn the solution blue-black in colour

- The initial rate of reaction between iodide ions and persulfate ions can therefore be found by measuring the time taken to liberate sufficient iodine to react with the thiosulfate ions present and then produce a blue colour with starch solution

- The rate of reaction is proportional to 1/(time taken for blue colour to form)

* **Experiment 2**

- Manganate (VII) ions react with ethanedioate ions in acidic conditions:

**2MnO4- + 5C2O42- + 16H+ 🡪 2Mn2+ + 10CO2 + 8H2O**

- The reaction is initially slow but speeds up rapidly once enough Mn2+ has been produced to catalyse the reaction; as a result the solution remains purple (due to MnO4-) for some time before becoming rapidly decolorised; the initial rate of reaction can therefore be found by measuring the time taken for the solution to decolorise

- The rate of reaction is proportional to 1/(time taken for solution to decolorise)

* The activation energy of both of these reactions can be found by investigating how the rate of a reaction varies with temperature

**Background**

* The rate constant of a chemical reaction is related to the temperature (in K) and the activation energy according the following equation: **k = Aexp(-Ea/RT)**
* exp(-Ea/RT) is the fraction of collisions which are successful
* Ea = activation energy (in Jmol-1)
* R = molar gas constant (in Jmol-1K-1)
* T = temperature (in K)
* A is the Arrhenius constant for that reaction
* This equation can be rearranged as follows:

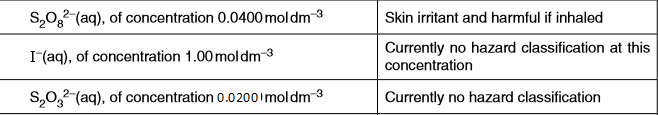
Log(k) = logA – (Ea/R)T

A graph of log(k) (or log 1/time taken) against 1/T (in K) will give a straight line of gradient Ea/R; the gradient can be used to determine the activation energy of the reaction

**Procedure**

**Experiment 1:**

* The following concentrations should be used:



1. Using suitable measuring cylinders, dd 5 cm3 of sodium persulfate solution, 5 cm3 of sodium thiosulphate solution and 2 cm3 of starch solution to a boiling tube
2. Use a suitable measuring cylinder to add 10 cm3 of potassium iodide solution to a test tube
3. Place a clean dry thermometer into the boiling tube
4. Pour the KI from the test tube into the boiling tube and start timing; shake the mixture to ensure that the contents are well mixed; record the time taken for the blue colour to appear and also record the initial and final temperature of the reaction mixture
5. Repeat steps (a) – (c) and then place the boiling tube and the test tube into a water bath until the temperature in the boiling tube has reached 30 oC; then carry out step (d)
6. Repeat steps (a) – (c) and then place the boiling tube and the test tube into a water bath until the temperature in the boiling tube has reached 40 oC; then carry out step (d)
7. Repeat steps (a) – (c) and then place the boiling tube and the test tube into a water bath until the temperature in the boiling tube has reached 50 oC; then carry out step (d)
8. Repeat steps (a) – (c) and then place the boiling tube and the test tube into a water bath until the temperature in the boiling tube has reached 60 oC; then carry out step (d)
9. Record your results in a table similar to the one below:

|  |  |  |  |
| --- | --- | --- | --- |
| Time taken for blue colour to appear (s) | Initial Temperature (K) | Final Temperature (K) | Average Temperature (K) |
|  |  |  |  |
|  |  |  |  |
|  |  |  |  |
|  |  |  |  |
|  |  |  |  |

**Experiment 2:**

* The following concentrations should be used:

|  |  |
| --- | --- |
| solution | molarity/ moldm-3 |
| KMnO4 | 0.010 |
| H2C2O4 | 0.050 |
| H2SO4 | 1.0 |

1. Using suitable measuring cylinders, add 5 cm3 of H2SO4 and 10 cm3 of H2C2O4 solution to a boiling tube
2. Use a suitable measuring cylinder to add 5 cm3 of KMnO4 solution to a test tube
3. Place a clean dry thermometer into the boiling tube
4. Pour the KMnO4 from the test tube into the boiling tube and start timing; shake the mixture to ensure that the contents are well mixed; record the time taken for the solution to decolorise and also record the initial and final temperature of the reaction mixture
5. Repeat steps (a) – (c) and then place the boiling tube and the test tube into a water bath until the temperature in the boiling tube has reached 30 oC; then carry out step (d)
6. Repeat steps (a) – (c) and then place the boiling tube and the test tube into a water bath until the temperature in the boiling tube has reached 40 oC; then carry out step (d)
7. Repeat steps (a) – (c) and then place the boiling tube and the test tube into a water bath until the temperature in the boiling tube has reached 50 oC; then carry out step (d)
8. Repeat steps (a) – (c) and then place the boiling tube and the test tube into a water bath until the temperature in the boiling tube has reached 60 oC; then carry out step (d)
9. Record your results in a table similar to the one below:

|  |  |  |  |
| --- | --- | --- | --- |
| Time taken for purple colour to disappear (s) | Initial Temperature (K) | Final Temperature (K) | Average Temperature (K) |
|  |  |  |  |
|  |  |  |  |
|  |  |  |  |
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| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| **Report**   1. For both experiments, record all your results clearly in a table similar to the following:  |  |  |  |  |  | | --- | --- | --- | --- | --- | | Time taken/s | (1/time taken)/s | Log(1/time taken) | Average Temperature/K | (1/temp)/K-1 | |  |  |  |  |  | |  |  |  |  |  | |  |  |  |  |  | |  |  |  |  |  |  1. Plot graphs of log (1/time taken) against (1/temperature) for each experiment 2. Deduce the gradient of your line of best fit for each graph and hence calculate the value of the activation energy for each reaction |

**Week 10 – Kinetics III**

#### Determination of the order of a reaction using a continuous monitoring method

#### Background: continuous monitoring method vs initial rates method

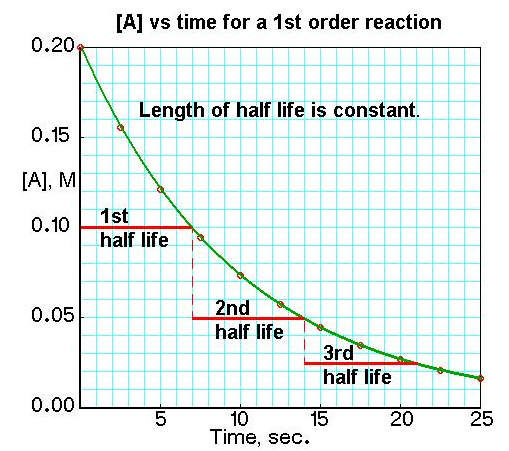
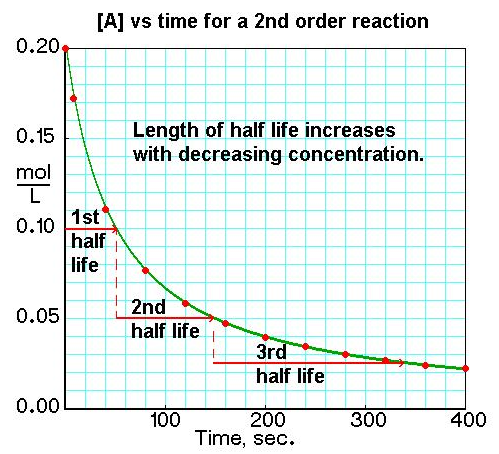
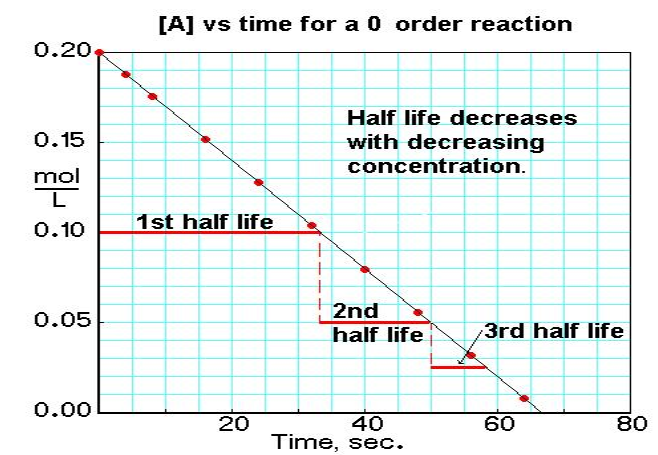
1. **The initial rates method (rate-concentration graphs)**

* The initial rates method involves carrying out the same reaction multiple times, using different initial concentrations of the reactants, and measuring the initial rate of reaction in each case; the order of reaction can be deduced from the shape of a graph of initial rate against initial concentration:

1. **The continuous monitoring method (concentration-time graphs)**

* The continuous monitoring method involves monitoring the concentration of a single reactant over time in a single reaction; it can only be used when only one reactant’s concentration is changing; the order of reaction can be deduced from the concentration-time graph:

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#### Introduction

* Iodine reacts with propanone in the presence of an acid catalyst as follows:

I2(aq) + CH3COCH3(aq) 🡪 CH3COCH3I(aq) + HI(aq)

* The quantity of iodine remaining in the reaction mixture can be monitored over time by removing small samples of the reaction mixture, “quenching” the reaction by adding a small quantity of NaHCO3 to remove the acid catalyst, and titrating the sample against Na2S2O3; the Na2S2O3 reacts with the iodine as follows: I2(aq) + 2S2O32-(aq) 🡪 S4O62-(aq) + 2I-(aq)
* From this titration, the moles of iodine in the sample, and hence the molarity of iodine in the main reaction mixture, can be deduced; if the sampling is repeated at various time intervals, it is possible to monitor how the concentration of iodine varies over time during the reaction
* If the reacting quantities are set to ensure that the propanone is in large excess, it can be assumed that the concentration of propanone does not change during this reaction, and therefore that iodine is the only reactant whose concentration changes during the reaction; a graph of iodine concentration against time can therefore be plotted and used to determine the initial rate of the reaction and the order of reaction with respect to iodine (continuous monitoring method)
* The experiment can then be repeated using different initial concentrations of propanone; in each case the variation of iodine concentration with time can be plotted; a graph of initial rate against initial propanone concentration can be plotted and used to determine the order of reaction with respect to propanone (initial rate method); alternatively, the ratio of initial rates can be compared to the ratio of initial concentrations

**Equipment List:** one 50 cm3 burette, one funnel,one 250 cm3 conical flask, two 25 cm3 measuring cylinders, one 25.0 cm-3 pipette, one 10 cm3 pipette, one pipette filler, one stopwatch

**Chemicals List:** access to1.0 moldm-3 H2SO4 (min) , 1.0 moldm-3 CH3COCH3 (min), 0.02 moldm-3 I2 (min), 0.005 moldm-3 Na2S2O3 (min), 0.50 moldm NaHCO3 (min) 1 cm3 starch solution with dropping pipette

**Procedure - Experiment 1:**

1. Prepare a burette for first use with 0.0050 moldm-3 sodium thiosulphate solution.
2. Using a measuring cylinder, pour 25 cm3 of 1.0 moldm-3 sulphuric acid into a conical flask. Measure the quantity of acid as accurately as possible. Label this flask A.
3. Using the same measuring cylinder, measure out 20 cm3 of 1.0 moldm-3 propanone. Add 5 cm3 of water (so that the total volume is 25 cm3) and pour the mixture into flask A. Measure the quantity of propanone as accurately as possible.
4. Using a 25 cm3 pipette, transfer 50.0 cm3 of 0.020 moldm-3 I2 into a 100 cm3 beaker.
5. Pour the iodine into the conical flask containing the sulphuric acid and the propanone. Start the timer as soon as you do this.
6. Measure out 25 cm3 of 0.5 moldm-3 NaHCO3 using a second measuring cylinder.
7. After the reaction has been going for four minutes, pipette 10 cm3 of the reaction mixture into a new conical flask, labelled flask B. Allow the rest of the reaction mixture in flask A to continue reacting.
8. Add the NaHCO3 from the measuring cylinder to flask B, noting the exact time at which you did so. Do not stop the timer.
9. Titrate the quenched sample of the reaction mixture (flask B) against the sodium thiosulphate. When the mixture is pale yellow, add 1 cm3 of starch solution (a blue colour will appear) using the dropping pipette provided and then continue the titration drop by drop until the blue colour disappears. Record your titre volume.
10. Rinse out flask B with distilled water.
11. Repeat steps 6 – 9 until a total of five samples have been withdrawn, taking care to ensure that the samples are quenched at approximately five-minute intervals (the fifth and final sample should therefore be quenched approximately 25 minutes after the start of the reaction).
12. Once you have quenched five samples, you may stop the timer, discard the remaining contents of flask A and rinse it with distilled water.
13. Record your results in a table similar to the one below:

|  |  |  |  |
| --- | --- | --- | --- |
| Time between start of reaction and sample quenching (s) | Final volume of Na2S2O3 (cm3) | Initial volume of Na2S2O3 (cm3) | Volume of Na2S2O3 required (cm3) |
|  |  |  |  |
|  |  |  |  |
|  |  |  |  |
|  |  |  |  |
|  |  |  |  |

**Procedure - Experiment 2:**

1. Repeat steps 2 – 13 above, but this time during step 3, use 10 cm3 of 1.0 moldm-3 propanone and 15 cm3 of water (so that the total volume is still 25 cm3) instead of 20 cm3 of propanone and 5 cm3 of water.

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| **Report:**   1. Complete two tables showing the times and titre volumes for each experiment (10 marks) 2. The titre volume of Na2S2O3 can be used to determine the iodine concentration at the time when the sample was quenched: [I2] = titre volume x 2.5 x 10-4; use this ratio to calculate the iodine concentration at each quenching time (4 marks) 3. Hence plot two graphs, on the same axes, showing how [I2] changes with time; include [I2] at t = 0, which is 0.01 moldm-3 (10 marks) 4. From the shape of your graphs, deduce the order of reaction with respect to iodine (4 marks) 5. Calculate the initial rate of reaction in both experiments (this is the initial gradient of the concentration-time graph) and hence deduce the order of reaction with respect to propanone (4 marks) |