

**DEPARTMENT OF CHEMISTRY
FOURAH BAY COLLEGE – UNIVERSITY OF SIERRA LEONE**

CHEM 124

PRACTICAL CHEMISTRY II

(to be taken alongside CHEM 121, CHEM 122 and CHEM 123)

CREDIT HOURS	1.5
REQUIREMENTS	C- or better in CHEM 114 To be taken alongside CHEM 111, CHEM 122 and CHEM 123
REQUIRED FOR	CHEM 214

CHEM 124

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; students only taking one of the modules must pay a cost of Le 20,000
- 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; students will not be admitted into any practical session unless they are wearing safety glasses
- Students should also bring a pen, writing paper and a calculator to every practical session; graph paper is also required for some reports
- Disposable gloves should be worn during organic chemistry practicals; these will be provided by the department

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 – 30%

- 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
- Each report is valued at either one credit (10 marks) or two credits (20 marks); the best eight credits achieved by each student over the semester will be added up to give a total out of 80; this mark will be divided by 4 to give a final continuous assessment mark out of 30

Practical Examination – 70%

- Students will sit a single practical examination at the end of each semester. This will consist of two practical exercises and some written questions; there are 70 marks available for the practical exam
- 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

- reports must be submitted by 12:00 on the first working day after the practical
- all rough work must be submitted along with the report
- the report should answer all questions
- some of the marks for each report may be awarded based on the group's performance during the practical session; students should work safely but quickly, involve the whole group in the practical work, avoid using more than the allocated quantity of chemicals and use all appropriate safety precautions

CONTENTS

Week	Experiment Title
1	Qualitative Analysis I – Identification of Cations
2	Qualitative Analysis II – Identification of Anions
3	Kinetics I: Determination of a rate equation using the initial rates method
4	Kinetics II: Determination of the activation energy of two reactions
5	Organic I – Investigation of solubility and the presence of C=C bonds
6	Organic II – Investigation of alcohols and carbonyls
7	Organic III – Investigation of carboxylic acids, esters and amines
8	Organic IV– Qualitative Analysis of Organic Compounds
9	Calorimetry II – Using Hess' Law
10	Kinetics III – Determination of an order of reaction using a continuous monitoring method

Week 1 – Qualitative Analysis of Cations in Solution

Qualitative Analysis is the identification of an unknown substance.

Cations in solution can be identified from:

- the colour of the solution
- whether or not they give a precipitate with certain reagents, the colour of that precipitate and the solubility of the precipitate in other reagents
- whether or not they form complex ions with certain reagents, observable by the solution turning a characteristic colour

In this experiment you will be given 20 solutions, labelled A – T. Each solution contains a different one of the following cations:

NH_4^+	Na^+	K^+	Mg^{2+}	Ca^{2+}	Sr^{2+}	Ba^{2+}	Al^{3+}	Sn^{2+}	Pb^{2+}
Cr^{3+}	Mn^{2+}	Fe^{2+}	Fe^{3+}	Co^{2+}	Ni^{2+}	Cu^{2+}	Zn^{2+}	Ag^+	Cd^{2+}

You are required to carry out a series of observations and simple chemical tests in order to identify which cation is in which solution.

The available tests are:

(a) hydrochloric acid followed by dilute ammonia

- dilute HCl will precipitate Ag^+ and Pb^{2+} as chlorides
- AgCl dissolves in dilute NH_3 to give a complex ion but PbCl_2 does not

Test: add 1 cm³ of dilute HCl to 1 cm³ of the unknown solution; if a precipitate is formed add dilute NH_3 dropwise until the test tube is half-full

(b) hydrogen sulphide

- H_2S in acidic conditions will precipitate Ag^+ , Pb^{2+} , Cu^{2+} , Sn^{2+} and Cd^{2+} as sulphides
- the colour of the precipitate can help identify the cation
- Fe^{3+} can oxidise H_2S to a yellow precipitate of sulphur

Test: bubble H_2S into 1 cm³ of the unknown solution for 10 seconds; if no precipitate is formed after 10 s, bubble H_2S into the solution for a further 20 s

(c) sodium hydroxide solution

- NaOH will precipitate all cations except NH_4^+ , Na^+ , K^+ , Ba^{2+} and Sr^{2+} as hydroxides, although $\text{Ca}(\text{OH})_2$ does not form instantly
- the colour of the precipitate can help identify the cation
- $\text{Pb}(\text{OH})_2$, $\text{Zn}(\text{OH})_2$, $\text{Cr}(\text{OH})_3$, $\text{Al}(\text{OH})_3$ and $\text{Sn}(\text{OH})_2$ are amphoteric and dissolve in excess sodium hydroxide to give hydroxoanions

Test: add a few drops of dilute NaOH to 1 cm³ of the unknown solution; continue to add NaOH , a few drops at a time, until the test tube is half-full

(d) ammonia solution

- NH_3 is alkaline and will precipitate all cations except NH_4^+ , Na^+ , K^+ , Ba^{2+} , Sr^{2+} and Ca^{2+} as hydroxides
- the colour of the precipitate can help identify the cation
- AgOH , $\text{Cu}(\text{OH})_2$, $\text{Cd}(\text{OH})_2$, $\text{Cr}(\text{OH})_3$, $\text{Ni}(\text{OH})_2$, $\text{Mn}(\text{OH})_2$, $\text{Zn}(\text{OH})_2$ and $\text{Co}(\text{OH})_2$ dissolve in excess dilute NH_3 to give complex ions
- the colour of the complex can help identify the cation

Test: add a few drops of dilute NH_3 to 1 cm³ of the unknown solution; continue to add NH_3 , a few drops at a time, until the test tube is half-full

(e) dilute sulphuric acid

- H_2SO_4 will precipitate Pb^{2+} , Ag^+ , Ba^{2+} and Sr^{2+} ions as sulphates; Ca^{2+} also forms a precipitate slowly

Test: add 1 cm³ of dilute H_2SO_4 to 1 cm³ of the unknown solution

You are NOT required to carry out all five of these tests on every solution; you are only required to carry out enough tests to identify all 20 cations; should these five tests be insufficient to identify a particular cation, there are some other tests available to you if you ask a technician:

- potassium dichromate and ethanoic acid precipitates Ag^+ , Pb^{2+} and Ba^{2+} as chromates; the colour of the precipitate can help identify the cation
- potassium hexacyanoferrate (II) solution precipitates Cu^{2+} , Fe^{2+} , Fe^{3+} , Zn^{2+} and Ca^{2+} as cyanoferrates; the colour of the precipitate can help identify the cation
- uranyl magnesium acetate solution precipitates Na^+ as a magnesium uranyl acetate

CHEM 124

The expected observations for each chemical test are shown below:

	Cation	Colour in solution	(a) HCl followed by NH ₃	(b) Acidic H ₂ S	(c) NaOH until in excess	(d) NH ₃ until in excess	(e) H ₂ SO ₄	Extra Test
1	Ag ⁺	Colourless	White ppt soluble in NH ₃	Black ppt	Brown ppt	brown ppt soluble in excess	White ppt	K ₂ CrO ₄ /CH ₃ COOH – red ppt
2	Pb ²⁺	Colourless	White ppt insoluble in NH ₃	Black ppt	White ppt soluble in excess	White ppt insoluble in excess	White ppt	K ₂ CrO ₄ /CH ₃ COOH – yellow ppt
3	Cu ²⁺	Pale blue	No reaction	Black ppt	Pale blue ppt	Pale blue ppt, soluble in excess to give deep blue solution	No reaction	K ₄ Fe(CN) ₆ – red/brown ppt
4	Sn ²⁺	Colourless	No reaction	Brown ppt	White ppt soluble in excess	White ppt insoluble in excess	No reaction	
5	Cd ²⁺	Colourless	No reaction	Yellow ppt	White ppt	White ppt soluble in excess	No reaction	
6	Fe ²⁺	Pale green	No reaction	No reaction	Dirty green ppt, turning orange on standing	Dirty green ppt, turning orange on standing	No reaction	K ₄ Fe(CN) ₆ – pale blue ppt
7	Fe ³⁺	Yellow/orange	No reaction	Yellow ppt	Red-brown ppt	Red-brown ppt	No reaction	K ₄ Fe(CN) ₆ – pale blue ppt
8	Cr ³⁺	Dark green	No reaction	No reaction	Green ppt, soluble in excess	Green ppt, soluble in excess	No reaction	
9	Ni ²⁺	Green	No reaction	No reaction	Green ppt	Green ppt soluble in excess	No reaction	
10	Mn ²⁺	Pale pink /colourless	No reaction	No reaction	White ppt, darkens on standing,	White ppt soluble in excess	No reaction	

CHEM 124

	Cation	Colour in solution	(a) HCl followed by NH ₃	(b) Acidic H ₂ S	(c) NaOH until in excess	(d) NH ₃ until in excess	(e) H ₂ SO ₄	Extra Test
11	Zn ²⁺	Colourless	No reaction	No reaction	White ppt soluble in excess	White ppt soluble in excess	No reaction	K ₄ Fe(CN) ₆ – white ppt
12	Al ³⁺	Colourless	No reaction	No reaction	White ppt Soluble in excess	White ppt	No reaction	
13	Co ²⁺	Pink	No reaction	No reaction	Blue ppt, turning pink on standing	Blue ppt, soluble in excess (yellow solution)	No reaction	
14	Ca ²⁺	Colourless	No reaction	No reaction	Faint white ppt in excess	No reaction	White ppt may form slowly	K ₄ Fe(CN) ₆ – white ppt
15	Sr ²⁺	Colourless	No reaction	No reaction	No reaction	No reaction	White ppt	
16	Ba ²⁺	Colourless	No reaction	No reaction	No reaction	No reaction	White ppt	K ₂ CrO ₄ /CH ₃ COOH – yellow ppt
17	NH ₄ ⁺	Colourless	No reaction	No reaction	Pungent gas evolved on warming	No reaction	No reaction	
18	Na ⁺	Colourless	No reaction	No reaction	No reaction	No reaction	No reaction	UMA – yellow ppt
19	K ⁺	Colourless	No reaction	No reaction	No reaction	No reaction	No reaction	
20	Mg ²⁺	Colourless	No reaction	No reaction	White ppt	White ppt	No reaction	

Report

- 1) Identify the cation present in solutions A – T and explain how you were able to identify each cation
 - Indicate which tests you carried out on each solution
 - Indicate your observations in full for each test
 - Indicate what you have concluded from each test

- 2) For the regular tests (a) – (e) which gave a result, write an equation for the reaction taking place.

- 3) Explain any problems you encountered with identifying the cation present in each solution.

Week 2 – Qualitative Analysis of Anions in Solution

Qualitative Analysis is the identification of an unknown substance.

Anions in solution can be identified from:

- whether or not they give a precipitate with certain reagents, the colour of that precipitate and the solubility of the precipitate in other reagents
- whether or not they evolve gases with certain reagents, and if so, which gas is evolved

In this experiment you will be given 10 solids, labelled A – J. Each solid contains a different one of the following ions as well as a K^+ or Na^+ ion:

CO_3^{2-}	HCO_3^-	SO_4^{2-}	SO_3^{2-}	S^{2-}	NO_3^-	NO_2^-	Cl^-	Br^-	I^-
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You are required to carry out a series of observations and simple chemical tests in order to identify which anion is in which solid.

The available tests are:

(a) hydrochloric acid

- H^+ will give an acid-base reaction with CO_3^{2-} and HCO_3^- to produce CO_2 , with SO_3^{2-} to produce SO_2 , with S^{2-} to produce H_2S , with NO_2^- to produce NO_2 ; in all cases effervescence will be observed
- The gas evolved can be tested as follows:

CO_2	bubble through limewater	Turns limewater cloudy
SO_2	dichromate paper bubble through limewater	Turns green Turns limewater cloudy
NO_2	brown gas	
H_2S	smelly gas (rotten eggs) dichromate paper lead ethanoate paper	Turns green Turns black

Test: add 1 cm³ of dilute HCl to a small spatula measure of the unknown solid; if a gas is evolved check the colour and smell; then add dichromate or lead ethanoate paper as appropriate; if both tests negative bubble through limewater

(b) barium chloride solution

- Ba^{2+} will give precipitates with HCO_3^- , CO_3^{2-} , SO_3^{2-} which dissolve in HCl with effervescence
- Ba^{2+} will give a precipitate with SO_4^{2-} which is insoluble in HCl

Test: dissolve a small spatula measure of the unknown solid in water; add 1 cm³ of $BaCl_2$; if a precipitate is formed add HCl gradually until in excess

(c) Silver nitrate solution

- Ag^+ will give precipitates with CO_3^{2-} , SO_3^{2-} , S^{2-} , Cl^- , Br^- and I^- and with excess NO_2^- and SO_4^{2-}
- Ag_2CO_3 and Ag_2SO_3 dissolve in HNO_3
- All precipitates except AgBr and AgI dissolve in dilute NH_3

Test: dissolve a small spatula measure of the unknown solid in water; add 1 cm³ of AgNO_3 ; if a precipitate is formed, split the precipitate into two portions; to the first add HNO_3 and to the second add NH_3

(d) Chlorine water

- Chlorine will oxidise I^- and Br^-
- the colours of the halogens formed can be used to identify the ion
- organic solvents give more clarity to the colour

Test: dissolve a small spatula measure of the unknown solid in water; add 1 cm³ of chlorine water; if the solution turns yellow, orange or brown add 1 cm³ of cyclohexane and shake well

You are NOT required to carry out all three of these tests on every anion; you are only required to carry out enough tests to identify all 10 anions; should these three tests be insufficient to identify a particular anion, there are some other tests available to you:

(i) magnesium sulphate solution

- Mg^{2+} will give a precipitate with CO_3^{2-} but not HCO_3^-

Test: Dissolve a small spatula measure of the unknown solid in water; add 1 cm³ of MgSO_4

(ii) Potassium manganate (VII) solution

- MnO_4^- is reduced and decolorised by SO_3^{2-} , NO_2^- and Cl^- it is also reduced by S^{2-} , Br^- and I^- but the resulting solutions are also coloured

Test: Dissolve a small spatula measure of the unknown solid in water; add 1 cm³ of acidified KMnO_4

CHEM 124

The expected observations for each chemical test are shown below:

	Cation	Colour in solution	(a) (i) HCl to solid (ii) test on gas	(b) (i) BaCl ₂ to solution (ii) add HCl	(c) (i) AgNO ₃ to solution (ii) add HNO ₃ or NH ₃	(d) (i) add Cl ₂ (ii) add C ₆ H ₁₂	Extra Test
1	CO ₃ ²⁻	Colourless	Effervescence (CO ₂)	white ppt soluble in HCl	White ppt Soluble in HNO ₃ and NH ₃	No reaction	White ppt with MgSO ₄
2	HCO ₃ ⁻	Colourless	Effervescence (CO ₂)	white ppt soluble in HCl	White ppt Soluble in HNO ₃ and NH ₃	No reaction	No reaction with MgSO ₄
3	SO ₄ ²⁻	Colourless	no effervescence	white ppt	Faint white ppt in excess	No reaction	
4	SO ₃ ²⁻	Colourless	Effervescence (SO ₂)	white ppt soluble in HCl	White ppt Soluble in HNO ₃ and NH ₃	No reaction	Decolorises KMnO ₄
5	NO ₃ ⁻	Colourless	no effervescence	no reaction	No reaction	No reaction	
6	NO ₂ ⁻	Colourless	effervescence (NO ₂)	no reaction	Faint white ppt in excess	No reaction	Decolorises KMnO ₄
7	S ²⁻	Colourless	effervescence (H ₂ S)	no reaction	Black ppt soluble in NH ₃	Yellow ppt	Decolorises KMnO ₄ ; yellow ppt
8	Cl ⁻	Colourless	no effervescence	no reaction	White curdy ppt soluble in NH ₃	No reaction	Decolorises KMnO ₄
9	Br ⁻	Colourless	no effervescence	no reaction	Cream ppt	Yellow/orange solution; turns cyclohexane orange	Yellow/orange solution with KMnO ₄
10	I ⁻	Colourless	no effervescence	no reaction	Yellow ppt	Yellow/brown solution; turns cyclohexane purple	Yellow/brown solution with KMnO ₄

Report

- 1) Identify the anion present in solutions A – J and explain how you were able to identify each anion
 - Indicate which tests you carried out on each solution
 - Indicate your observations in full for each test
 - Indicate what you have concluded from each test

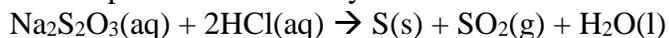
- 2) For the regular tests (a) – (d) which gave a result, write an equation for the reaction taking place.

- 3) Explain any problems you encountered with identifying the anion present in each solution.

Week 3 – Determining a Rate Equation Using the Initial Rates Method

Introduction

- Sodium thiosulphate reacts with hydrochloric acid as follows:



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

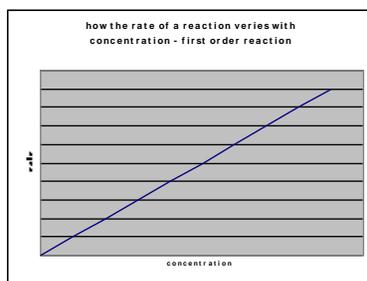
- The rate of reaction is inversely proportional to the time taken for the reaction to reach a particular stage:

$$\text{Relative rate of reaction} = \frac{1}{\text{time taken}}$$

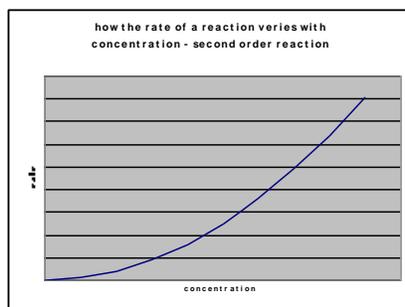
- The rate equation for a chemical reaction is an equation which links the rate of reaction to the concentrations of the reactants (eg rate = $k[\text{A}]^x[\text{B}]^y$)
- The aim of this experiment is to find the order of reaction with respect to $\text{Na}_2\text{S}_2\text{O}_3$ and HCl ; this can be done by carrying out the reaction using different molarities of $\text{Na}_2\text{S}_2\text{O}_3$ 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 = $k[\text{A}]$; a graph of initial rate against initial concentration will be a straight line through the origin of gradient k :

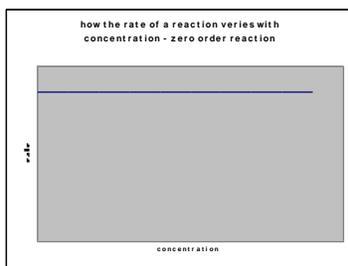


- In second-order reactions, rate = $k[\text{A}]^2$; a graph of initial rate against initial concentration will be a curve through the origin:



CHEM 124

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



Procedure

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

Experiment Number	“HCl cylinder”			“Na ₂ S ₂ O ₃ cylinder”		
	Volume of HCl/cm ³	Volume of water/cm ³	[HCl]/moldm ⁻³	Volume of Na ₂ S ₂ O ₃ /cm ³	Volume of water/cm ³	[Na ₂ S ₂ O ₃]/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 $\text{Na}_2\text{S}_2\text{O}_3$ and HCl in each experiment
2. Using the results of your experiments, calculate $1/\text{time taken}$ for each experiment and put your results in a table as follows:

Experiment	$[\text{Na}_2\text{S}_2\text{O}_3]$ / mol dm^{-3}	$[\text{HCl}]/\text{mol dm}^{-3}$	Time taken/s	$1/\text{time taken (s}^{-1}\text{)}$
5				
4				
3				
2				
1				

Experiment	$[\text{Na}_2\text{S}_2\text{O}_3]$ / mol dm^{-3}	$[\text{HCl}]/\text{mol dm}^{-3}$	Time taken/s	$1/\text{time taken (s}^{-1}\text{)}$
9				
8				
7				
6				
3				

3. Plot a graph of rate of reaction against $[\text{Na}_2\text{S}_2\text{O}_3]$ using experiments 1 – 5
4. Plot a graph of rate of reaction against $[\text{HCl}]$ using experiments 3 and 6 – 9
5. Use your graphs to deduce the orders of reaction with respect to HCl and $\text{Na}_2\text{S}_2\text{O}_3$, write a rate equation for the reaction and calculate the rate constant.

Week 4 - Determining The Activation Energy Of Two Reactions

Introduction

- **Experiment 1**

- Persulfate ions reacts with iodide ions to form iodine:

$$\text{S}_2\text{O}_8^{2-}(\text{aq}) + 2\text{I}^-(\text{aq}) \rightarrow \text{I}_2(\text{aq}) + 2\text{SO}_4^{2-}(\text{aq})$$
- The iodine produced reacts with thiosulphate ions:

$$2\text{S}_2\text{O}_3^{2-}(\text{aq}) + \text{I}_2(\text{aq}) \rightarrow \text{S}_4\text{O}_6^{2-}(\text{aq}) + 2\text{I}^-(\text{aq})$$
- 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 $1/(\text{time taken for blue colour to form})$

- **Experiment 2**

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

$$2\text{MnO}_4^- + 5\text{C}_2\text{O}_4^{2-} + 16\text{H}^+ \rightarrow 2\text{Mn}^{2+} + 10\text{CO}_2 + 8\text{H}_2\text{O}$$
- The reaction is initially slow but speeds up rapidly once enough Mn^{2+} has been produced to catalyse the reaction; as a result the solution remains purple (due to MnO_4^-) 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 $1/(\text{time taken for solution to decolorise})$
- 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: $k = A\exp(-E_a/RT)$
- $\exp(-E_a/RT)$ is the fraction of collisions which are successful
- E_a = activation energy (in Jmol^{-1})
- R = molar gas constant (in $\text{Jmol}^{-1}\text{K}^{-1}$)
- T = temperature (in K)
- A is the Arrhenius constant for that reaction

- This equation can be rearranged as follows:

$$\text{Log}(k) = \text{log}A - (E_a/R)T$$

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

Procedure

Experiment 1:

- The following concentrations should be used:

$\text{S}_2\text{O}_8^{2-}(\text{aq})$, of concentration $0.0400 \text{ mol dm}^{-3}$	Skin irritant and harmful if inhaled
$\text{I}^-(\text{aq})$, of concentration 1.00 mol dm^{-3}	Currently no hazard classification at this concentration
$\text{S}_2\text{O}_3^{2-}(\text{aq})$, of concentration $0.0200 \text{ mol dm}^{-3}$	Currently no hazard classification

- Using suitable measuring cylinders, dd 5 cm^3 of sodium persulfate solution, 5 cm^3 of sodium thiosulphate solution and 2 cm^3 of starch solution to a boiling tube
- Use a suitable measuring cylinder to add 10 cm^3 of potassium iodide solution to a test tube
- Place a clean dry thermometer into the boiling tube
- 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
- 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 $30 \text{ }^\circ\text{C}$; then carry out step (d)
- 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 $40 \text{ }^\circ\text{C}$; then carry out step (d)
- 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 $50 \text{ }^\circ\text{C}$; then carry out step (d)
- 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 $60 \text{ }^\circ\text{C}$; then carry out step (d)

CHEM 124

- i) 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
KMnO ₄	0.010
H ₂ C ₂ O ₄	0.050
H ₂ SO ₄	1.0

- Using suitable measuring cylinders, add 5 cm³ of H₂SO₄ and 10 cm³ of H₂C₂O₄ solution to a boiling tube
- Use a suitable measuring cylinder to add 5 cm³ of KMnO₄ solution to a test tube
- Place a clean dry thermometer into the boiling tube
- 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
- 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 °C; then carry out step (d)
- 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 °C; then carry out step (d)
- 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 °C; then carry out step (d)
- 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 °C; then carry out step (d)

CHEM 124

- i) 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)

Report

- a) 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

- b) Plot graphs of $\log(1/\text{time taken})$ against $(1/\text{temperature})$ for each experiment
- c) 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 5 – 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

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) In addition to solubility, do you notice any other trends in how the physical properties of organic compounds change along a homologous series? Make a note of any trends you observe.
- 4) 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

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

CHEM 124

- 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?

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 6 – 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: $R_1R_2CHOH + [O] \rightarrow R_1COR_2 + H_2O$
- Aldehydes can be further oxidised to carboxylic acids but ketones cannot be oxidised: $R_1CHO + [O] \rightarrow R_1COOH$
- Acidified potassium dichromate is a good reagent to use for this reaction; during the reaction, the orange $Cr_2O_7^{2-}$ ion is reduced to the dark green Cr^{3+} 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)

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 Cu^{2+} ion is reduced to the orange precipitate of Cu_2O
 - In Tollen's reagent (Test 5) the colourless Ag^+ ion is reduced to metallic silver

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: $R_1COR_2 + C_6H_3N_2O_4NHNH_2 \rightarrow R_1COR_2 + C_6H_3N_2O_4NHNCR_1R_2 + H_2O$; 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)

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:

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

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

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 7 – Organic Chemistry III

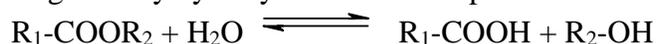
Test 8: The presence of H⁺ ions in carboxylic acids and esters

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



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:



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

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

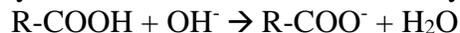
Interpretation of Results:

- 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:



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

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:

$$\text{R-NH}_2 + \text{H}_2\text{O} \rightarrow \text{R-NH}_3^+ + \text{OH}^-$$
 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

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

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

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

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 8 – 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:

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

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

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

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

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)

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

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

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

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.

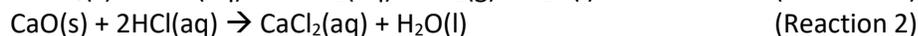
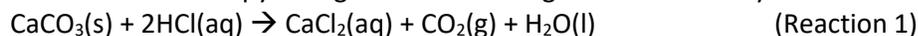
You do not have to carry every test out on every compound, but you must carry out at least enough test to identify the functional group in each.

You should present all test results and conclusions in your final report.

Week 9 – Calorimetry II

Experiment 1: Determine the enthalpy change for the following reaction: $\text{CaCO}_3(\text{s}) \rightarrow \text{CaO}(\text{s}) + \text{CO}_2(\text{g})$

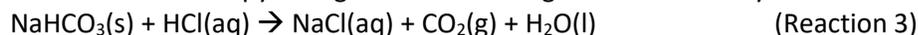
- The enthalpy change for this decomposition reaction is difficult to measure directly; it is, however, possible to measure the enthalpy change of the following reactions directly:



- 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
- Use a pipette to transfer 25 cm³ of 2 moldm⁻³ HCl into a clean dry plastic cup.
 - Place the plastic cup in a beaker and record the temperature of the acid to the nearest 0.5 °C.
 - Weigh out accurately between 2.50 and 3.00 g of calcium carbonate powder. Record your mass in a suitable table.
 - 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 °C.
 - 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: $2\text{NaHCO}_3(\text{s}) \rightarrow \text{Na}_2\text{CO}_3(\text{s}) + \text{CO}_2(\text{g}) + \text{H}_2\text{O}(\text{l})$

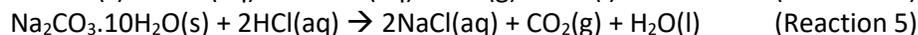
- The enthalpy change for this decomposition reaction is difficult to measure directly; it is, however, possible to measure the enthalpy change of the following reactions directly:



- 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
- Repeat steps 1 – 4, but using 2.50 – 3.00 grams of sodium hydrogencarbonate instead of the calcium carbonate.
 - 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: $\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}(\text{s}) \rightarrow \text{Na}_2\text{CO}_3(\text{s}) + 10\text{H}_2\text{O}(\text{l})$

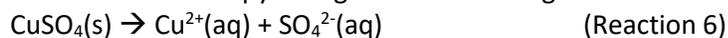
- The enthalpy change for this decomposition reaction is difficult to measure directly; it is, however, possible to measure the enthalpy change of the following reactions directly:



- 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: $\text{CuSO}_4(\text{s}) \cdot 5\text{H}_2\text{O}(\text{l}) \rightarrow \text{CuSO}_4(\text{s}) + 5\text{H}_2\text{O}(\text{l})$

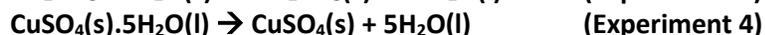
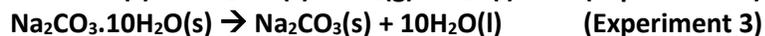
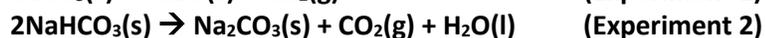
- The enthalpy change for this decomposition reaction is difficult to measure directly; it is, however, possible to measure the enthalpy change of the following reactions directly:



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

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:



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 g cm^{-3} and a specific heat capacity of $4.18 \text{ JK}^{-1} \text{ g}^{-1}$)
- 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

Marks will be awarded as follows:

Process	marks
Mass of solid used in each reaction correctly recorded	7
Mass of water used in reactions 6 and 7 correctly recorded	4
Initial and final temperature in each reaction correctly recorded	14
Heat change in each reaction correctly calculated	7
Moles of both reactants in reactions 1 – 5 correctly calculated	10
Limiting reagent correctly identified in reactions 1 – 5	5
Moles of solid used in reactions 6 and 7 correctly calculated	2
Molar enthalpy change of each reaction correctly calculated	7
Hess' Law cycles correctly deduced for each decomposition reaction	8
Molar enthalpy change of each decomposition reaction correctly calculated	8
TOTAL FOR REPORT	72

Your mark out of 72 will be scaled down to a mark out of 15; additional marks will be awarded as follows:

Achievement	marks
Signed in at 9.00 am	1
Signed in at 9.30 am	1
Present at start of practical work, wearing lab coat throughout and wearing suitable footwear	1
Present at start of practical work, wearing eye protection throughout	1
Present at start of practical work, using pipette safely and conducting all work safely and sensibly	1
TOTAL	5

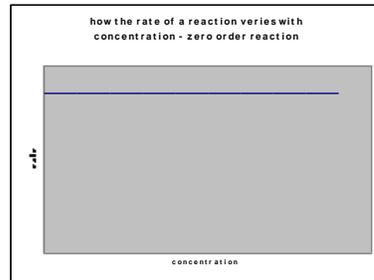
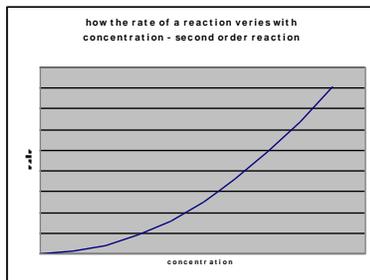
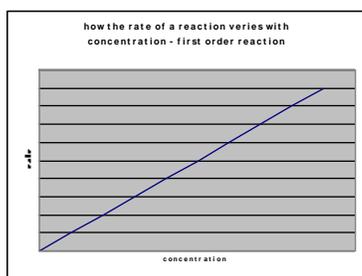
A total of 20 marks is available for this practical.

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

Background: continuous monitoring method vs initial rates method

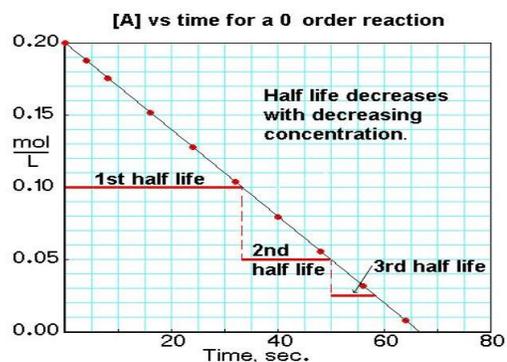
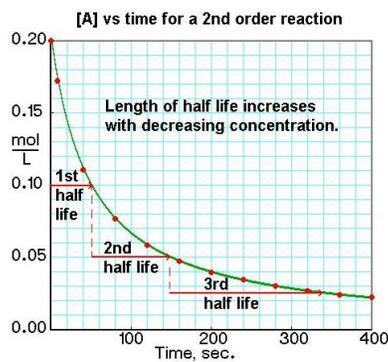
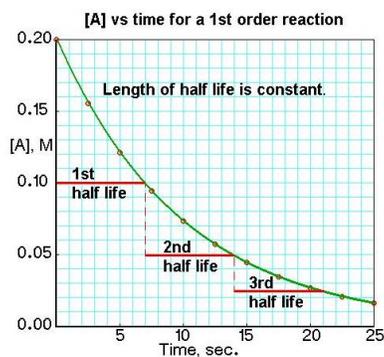
(a) The initial rates method (rate-concentration graphs)

- 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:



(b) 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:



Introduction

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

$$\text{I}_2(\text{aq}) + \text{CH}_3\text{COCH}_3(\text{aq}) \rightarrow \text{CH}_3\text{COCH}_2\text{I}(\text{aq}) + \text{HI}(\text{aq})$$
- 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 NaHCO_3 to remove the acid catalyst, and titrating the sample against $\text{Na}_2\text{S}_2\text{O}_3$; the $\text{Na}_2\text{S}_2\text{O}_3$ reacts with the iodine as follows:

$$\text{I}_2(\text{aq}) + 2\text{S}_2\text{O}_3^{2-}(\text{aq}) \rightarrow \text{S}_4\text{O}_6^{2-}(\text{aq}) + 2\text{I}^-(\text{aq})$$

CHEM 124

- 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 cm³ pipette, one 10 cm³ pipette, one pipette filler, one stopwatch

Chemicals List: access to 1.0 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.

CHEM 124

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 Na ₂ S ₂ O ₃ (cm ³)	Initial volume of Na ₂ S ₂ O ₃ (cm ³)	Volume of Na ₂ S ₂ O ₃ required (cm ³)

Procedure - Experiment 2:

14. 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.

Report:

- Your report should include two tables, showing the times and titre volumes for each experiment (10 marks)
- The titre volume of Na₂S₂O₃ can be used to determine the iodine concentration at the time when the sample was quenched: $[I_2] = \text{titre volume} \times 2.5 \times 10^{-4}$; use this ratio to calculate the iodine concentration at each quenching time (4 marks)
- Hence plot two graphs, on the same axes, showing how $[I_2]$ changes with time; include $[I_2]$ at $t = 0$, which is 0.01 moldm⁻³ (10 marks)
- From the shape of your graphs, deduce the order of reaction with respect to iodine (4 marks)
- 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)