GCE A LEVEL

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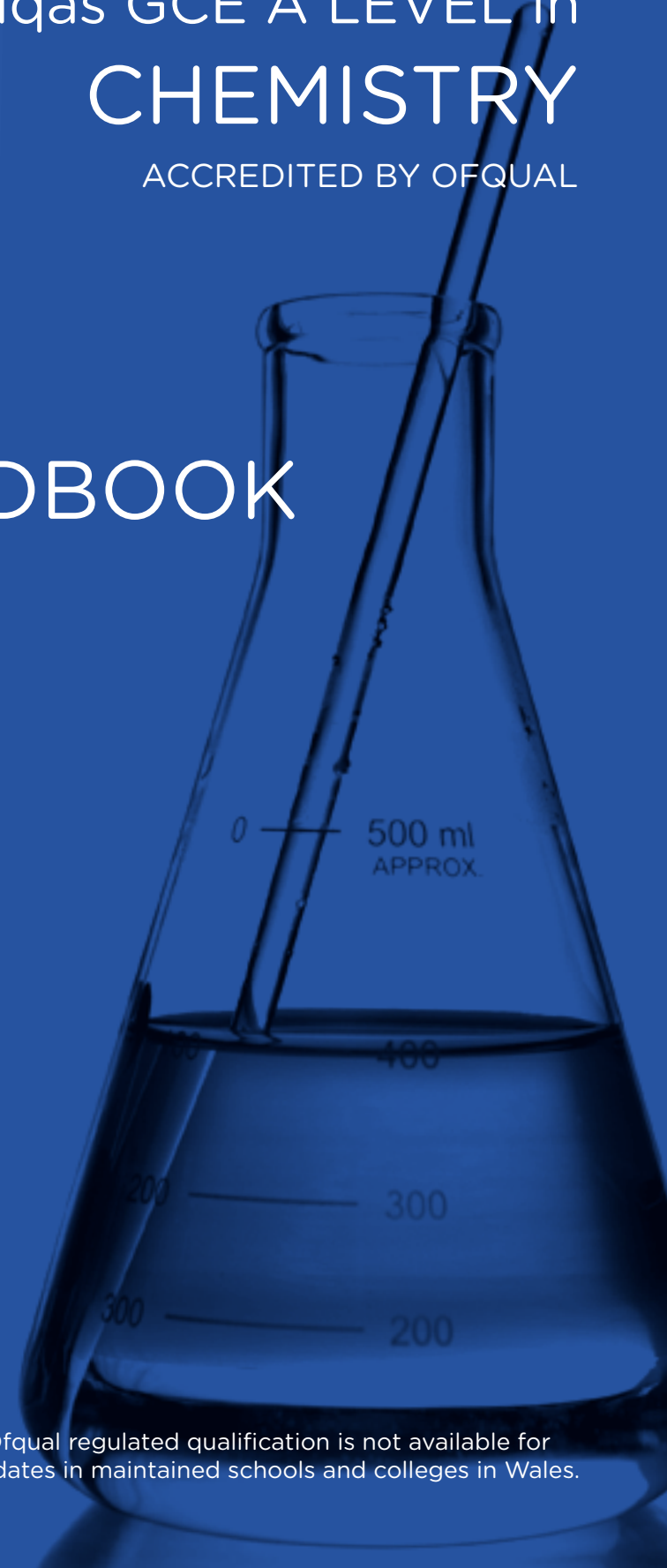
WJEC Eduqas GCE A LEVEL in

CHEMISTRY

ACCREDITED BY OFQUAL

TEACHER HANDBOOK

Teaching from 2015



wjec
cbac

This Ofqual regulated qualification is not available for candidates in maintained schools and colleges in Wales.

INTRODUCTION

The **WJEC Eduqas A level Chemistry** qualification, accredited by Ofqual for first teaching from September 2015, is available to:

- All schools and colleges in England and private schools in Wales
- Schools and colleges in independent regions such as Northern Ireland, Isle of Man and the Channel Islands

The A level will be awarded for the first time in summer 2017, using grades A*- E.

The qualification provides a broad, coherent, satisfying and worthwhile course of study. It encourages learners to develop confidence in, and a positive attitude towards, chemistry and to recognise its importance in their own lives and to society.

The specification lends itself to a variety of teaching and learning styles and offers learners of all abilities an enjoyable and positive learning experience.

Practical work is an intrinsic part of chemistry, and is greatly valued by higher education. It is imperative that practical skills are developed throughout the course and that an investigative approach is promoted wherever possible.

Additional ways that WJEC Eduqas can offer support:

- Specimen assessment materials
- Face-to-face CPD events
- Question paper database
- Examiners' reports on each question paper
- Free access to past question papers and mark schemes via the secure website
- Direct access to the subject officer
- Free online resources
- Exam Results Analysis
- Online Examination Review

If you have any queries please do not hesitate to contact:

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AIMS OF THE TEACHERS' GUIDE

The principal aim of the Teachers' Guide is to offer support to teachers in delivery of the new WJEC Eduqas **A level Chemistry** specification and to offer guidance on the requirements of the qualification and the assessment process.

The guide is **not intended as a comprehensive reference**, but as support for professional teachers to develop stimulating and exciting courses tailored to the needs and skills of their own learners in their particular institutions. The guide offers assistance to teachers with regard to the depth of coverage required, suggesting possible classroom activities and links to useful digital resources (both our own, freely available, digital materials and some from external sources) to provide ideas for engaging lessons.

EDUQAS – A LEVEL CHEMISTRY

CORE IDEAS, PRINCIPLES AND CONCEPTS

TOPIC C1 THE LANGUAGE OF CHEMISTRY AND STRUCTURE OF MATTER

C1.1 Formulae and equations

C1.2 Basic ideas about atoms

C1.3 Chemical calculations

C1.4 Bonding

C1.5 Solid structures

C1.6 The Periodic Table

TOPIC C2 CHEMICAL CHANGE

C2.1 Simple equilibria and acid-base reactions

C2.2 Thermochemistry

C2.3 Rates of reaction

C2.4 The wider impact of chemistry

TOPIC C3 CHEMISTRY OF CARBON COMPOUNDS

C3.1 Organic compounds

C3.2 Hydrocarbons

C3.3 Halogenoalkanes

C3.4 Alcohols and carboxylic acids

C3.5 Instrumental analysis

PHYSICAL AND INORGANIC CHEMISTRY

TOPIC PI ELECTROCHEMISTRY

PI1.1 Redox and standard electrode potential

PI1.2 Redox reactions

TOPIC PI2 MORE COMPLEX PATTERNS OF THE PERIODIC TABLE

PI2.1 Chemistry of the *p*-block

PI2.2 Chemistry of the *d*-block transition metals

TOPIC PI3 CHEMICAL KINETICS

TOPIC PI4 ENERGY CHANGES

PI4.1 Enthalpy changes for solids and solutions

PI4.2 Entropy and feasibility of reactions

TOPIC PI5 EQUILIBRIA

PI5.1 Equilibrium constants

PI5.2 Acid-base equilibria

ORGANIC CHEMISTRY AND ANALYSIS

TOPIC OA1 HIGHER CONCEPTS IN ORGANIC CHEMISTRY

OA1.1 Stereoisomerism

OA1.2 Aromaticity

TOPIC OA2 ORGANIC COMPOUNDS CONTAINING OXYGEN

OA2.1 Alcohols and phenols

OA2.2 Aldehydes and ketones

OA2.3 Carboxylic acids and their derivatives

TOPIC OA3 ORGANIC COMPOUNDS CONTAINING NITROGEN

OA3.1 Amines

OA3.2 Amino acids, peptides and proteins

TOPIC OA4ORGANIC SYNTHESIS AND ANALYSIS

CORE IDEAS, PRINCIPLES AND CONCEPTS

C1 THE LANGUAGE OF CHEMISTRY AND STRUCTURE OF MATTER

C1.1 FORMULAE AND EQUATIONS

Specification Statement		Comment
(a)	formulae of common compounds and common ions and how to write formulae for ionic compounds	Common compounds include the acids HCl, HNO_3, H_2SO_4, CH_3CO_2H, salts of these acids, Group 1 and Group 2 carbonates, hydroxides, oxides and gases such as CH_4, CO_2, NH_3, SO_2. Common ions are those from Groups 1, 2, 6 and 7 elements and compound ions such as NH_4^+, NO_3^-, OH^-, CO_3^{2-}, SO_4^{2-}.
(b)	oxidation numbers of atoms in a compound or ion	Candidates do not need to know the definition of oxidation number. Candidates should be able to apply the rules for assigning oxidation numbers in order to work out the oxidation number of an element in a compound or ion from its formula.
(c)	how to construct balanced chemical equations, including ionic equations, with appropriate use of state symbols	Candidates should be able to write balanced equations (full and ionic) for reactions studied. Candidates should be able to write balanced equations for unfamiliar reactions when reactants and products are supplied.

C1.2 BASIC IDEAS ABOUT ATOMS

	Spec Statement	Comment
(a)	nature of radioactive decay and the resulting changes in atomic number and mass number (including positron emission and electron capture)	Prior GCSE knowledge about atomic structure is expected. Candidates should be able to write an equation showing radioactive emission. For positron emission and electron capture no details of electron neutrinos are required.
(b)	behaviour of α-, β- and γ-radiation in electric and magnetic fields and their relative penetrating power	For the effect of magnetic fields, candidates should simply understand that α- and β- particles are deflected in opposite directions and by different amounts while γ- rays are unaffected.
(c)	half-life of radioactive decay	Candidates should be able to explain what is meant by half-life of a radioactive isotope and perform simple calculations involving integral numbers of half-lives.
(d)	adverse consequences for living cells of exposure to radiation and use of radioisotopes in many contexts, including health, medicine, radio-dating, industry and analysis	Candidates should know about the consequences of radiation from sources outside or inside the body. Candidates should be able to give an example and a brief outline of a beneficial use of radioactivity in each of these contexts.
(e)	significance of standard molar ionisation energies of gaseous atoms and their variation from one element to another	Knowledge of specific ionisation energy values is not required, only the trends down groups and across periods (up to Period 3).
(f)	link between successive ionisation energy values and electronic structure	Candidates should be able to use the successive ionisation energies of an element to predict the group to which it belongs, explaining their reasoning.
(g)	shapes of *s*- and *p*-orbitals and order of *s*-, *p*- and *d*-orbital occupation for elements 1–36	Candidates should be able to use sub-shell notation and 'arrows in boxes'.
(h)	origin of emission and absorption spectra in terms of electron transitions between atomic energy levels	Candidates should be able to relate emission or absorption spectra to energy diagrams.
(i)	atomic emission spectrum of the hydrogen atom	Candidates should understand the origin of the Lyman and Balmer series in the ultraviolet and visible regions of the spectrum.
(j)	relationship between energy and frequency (E = hf) and that between frequency and wavelength (f = c/λ)	Candidates should be able to use these equations in calculations. Candidates are not expected to recall the value of Planck's constant or the speed of light.
(k)	order of increasing energy of infrared, visible and ultraviolet	

	light	
(l)	significance of the frequency of the convergence limit of the Lyman series and its relationship with the ionisation energy of the hydrogen atom	Candidates should be able to calculate the first ionisation energy of an element given appropriate data.

C1.3 CHEMICAL CALCULATIONS

Specification Statement		Comment
(a)	relative mass terms (atomic, isotopic, formula, molecular)	Candidates should know that ^{12}C is used as the standard in comparing relative masses. Candidates should be able to use relative atomic masses to calculate relative formula masses.
(b)	principles of the mass spectrometer and its use in determining relative atomic mass and relative abundance of isotopes	Candidates will not be expected to draw a diagram of a mass spectrometer.
(c)	simple mass spectra, for example, that of chlorine gas	Candidates will be expected to use information about isotopes to deduce information about molecular ions and vice versa.
(d)	how empirical and molecular formulae can be determined from given data	
(e)	relationship between the Avogadro constant, the mole and molar mass	Calculations involving the use of the Avogadro constant may be set. Candidates are not expected to recall the value of the Avogadro constant.
(f)	relationship between grams and moles	
(g)	concept of concentration and its expression in terms of grams or moles per unit volume (including solubility)	
(h)	molar volume and correction due to changes in temperature and pressure	Formal definitions of Boyle's law and Charles' law will not be required. Candidates are not expected to recall molar volume at any given temperature
(i)	ideal gas equation ($pV = nRT$)	Candidates should know the equation and apply it to calculations for ideal gases, manipulating units as necessary.
(j)	concept of stoichiometry and its use in calculating reacting quantities, including in acid-base titrations	Candidates may be asked to deduce stoichiometric relationships from calculations. Calculations may be set on back titrations and double titrations.
(k)	concepts of atom economy and percentage yield	Candidates should be able to calculate atom economy and percentage yield.
(l)	how to estimate the percentage error in a measurement and use this to express numeric answers to the appropriate number of significant figures	The error in a measurement is one-half of the smallest division of the apparatus scale e.g. 0.05 cm3 on a burette or 0.005 g on a 2-decimal place digital balance. Recording a titre or measuring mass by difference requires two measurements therefore doubling the error. The error can be expressed as a percentage of the value measured. Values with approximately 1% error should be quoted to 3 significant figures whilst those with approximately 10% error should be quoted to 2 significant figures. Candidates should clearly show their logic when answering questions which test these skills. There will be no requirement to mathematically combine errors from more than one source.

C1.4 BONDING

Specification Statement		Comment
(a)	ionic bonding in terms of ion formation and the interaction between positive and negative ions in an ionic crystal	Candidates will be expected to predict the charges on ions and understand that ionic bonding is electrostatic attraction between oppositely charged ions.
(b)	covalent bonding, including coordinate bonding, in terms of the sharing of electrons and the balance of forces of attraction and repulsion within the molecule	Candidates should recognise that a covalent bond exists when a pair of electrons is shared and that, if both electrons are contributed by the same atom, this is co-ordinate bonding.
(c)	intermediate character of many bonds between purely ionic and purely covalent	
(d)	concepts of electronegativity and bond polarity	The meaning of electronegativity and the effect of differences in the electronegativities of bonding elements should be considered. The recall of electronegativity values is not required.
(e)	forces between molecules being much weaker than covalent (and ionic) bonds	
(f)	permanent and temporary dipoles and their relative effects on physical properties, such as boiling temperature and solubility	
(g)	hydrogen bonding and its effect on physical properties, such as boiling temperature and solubility	Candidates should know what elements are needed to form hydrogen bonds, that hydrogen bonds are much stronger than Van der Waals forces but much weaker than covalent or ionic bonds.
(h)	VSEPR principle and its use in predicting the shapes of simple molecules and ions	Candidates will be required to predict the shapes of molecules and ions using the number of pairs of electrons present. Species with up to 6 pairs of electrons could be used.
(i)	bond angles associated with linear, trigonal planar, tetrahedral and octahedral molecules and ions	Candidates should be able to quote bond angles for species with 2, 3, 4 and 6 pairs of electrons. This includes the effect on the bond angle of the presence of lone pairs.

C1.5 SOLID STRUCTURES

Specification Statement		Comment
(a)	crystal structures of sodium chloride and caesium chloride	Candidates should be able to quote the coordination numbers for the compounds, be able to draw 3D diagrams of each and recognise that the different structures are due to different cation size.
(b)	structures of diamond and graphite	Candidates should be able to draw a section of the structure of diamond and graphite and know that diamond is a giant tetrahedral structure whilst graphite is a hexagonal layer structure.
(c)	structures of iodine and ice	Candidates should recognise that solid iodine consists of molecules of iodine held in a lattice by weak intermolecular forces whilst ice is a giant tetrahedral structure containing stronger intermolecular hydrogen bonds.
(d)	‘electron sea’ model for bonding in metals	Metallic bonding should be considered as attraction between positive ions and a delocalised ‘electron sea’.
(e)	relationship between physical properties (for example, melting temperature, solubility, hardness and electrical conductance) and structure and bonding in ionic compounds, giant molecular substances, simple molecular substances and metals	Candidates must understand the effect of structure and bonding on physical properties. Questions could include prediction of properties based on structure and bonding or analysis of data to suggest structure and bonding.

C1.6 THE PERIODIC TABLE

Specification Statement		Comment
(a)	elements being arranged according to atomic number in the Periodic Table	
(b)	electronic structures of the elements relate to their position in the *s*-, *p*- and *d*-blocks of the Periodic Table	
(c)	oxidation and reduction in terms of electron transfer in reactions of *s*-, *p*- and *d*-block elements/compounds/ ions	Candidates should be able to interpret redox reactions in terms of electron loss/gain [and oxidation numbers 1.1(b)]
(d)	general trends in ionisation energy, melting temperature and electronegativity across periods and down groups	Candidates should be able to describe and explain these trends. No recall of actual values is required.
(e)	reactions of Group 2 elements with oxygen and water/steam	Candidates should be able to describe the reactions and recognise the trend.
(f)	reactions of the aqueous cations Mg^{2+}, Ca^{2+} and Ba^{2+} with OH^-, CO_3^{2-} and SO_4^{2-} ions	Candidates should be able to describe the reactions and write ionic equations for the formation of an insoluble salt.
(g)	characteristic flame colours shown by compounds of Li, Na, K, Ca, Sr and Ba (Mg compounds show no colour)	Candidates should recall this information and use it in qualitative analysis.
(h)	trend in general reactivity of Group 1 and Group 2 metals	Candidates should be able to explain the trend in reactivity in terms of increasing ease of cation formation.
(i)	trend in thermal stability of the Group 2 carbonates and hydroxides	Candidates should be able to describe the thermal decompositions. No explanation is required.
(j)	trends in solubility of Group 2 hydroxides and sulfates	Candidates are not required to remember any numerical values. No explanation is required.
(k)	basic character of the oxides and hydroxides of Group 1 and Group 2 metals	Candidates should appreciate that Group 1 oxides/hydroxides are stronger bases. Candidates should be able to explain everyday uses in terms of base strength e.g. why $Mg(OH)_2$ is used in indigestion remedies but $Ca(OH)_2$ is not.
(l)	trend in volatility of Group 7 elements (halogens)	Candidates should be able to explain the trend in terms of forces between molecules.
(m)	reactions of the halogens with metals	Candidates should be able to describe the reactions of halogens with sodium and iron.

(n)	trend in reactivity of the halogens in terms of relative oxidising power	Candidates should be able to explain the trend in terms of the oxidising ability decreasing down the group.
(o)	reaction between aqueous Ag^+ and halide ions followed by dilute aqueous NH_3	Candidates should be able to describe these precipitation reactions, including ionic equations. Complexes with ammonia are not required. Candidates should be able to use the precipitation reactions as a test for different halide ions.
(p)	displacement reactions of halogens in terms of redox	Candidates should be able to describe the reactions, including ionic equations, of the halogens with other halides.
(q)	use of chlorine and fluoride ions in water treatment and the related health and ethical issues	Candidates should be able to contrast the benefits of chlorine and fluoride ions in water treatment with associated risks.
(r)	soluble salt formation and crystallisation, insoluble salt formation by precipitation and simple gravimetric analysis	Candidates should be able to describe how soluble and insoluble salts are formed. Candidates could be expected to calculate the concentration of an ion in a solution by precipitating the ion and weighing.

GRAVIMETRIC ANALYSIS

SPECIFICATION REFERENCE: C1.6

AIM

Identification of an unknown metal chloride

APPARATUS AND CHEMICALS

- Access to balance – ideally 3 decimal place (minimum 2 decimal place)
- Access to drying oven

- 2 x 250 cm^3 beaker
- 2 x 100 cm^3 beaker
- spatula
- 10 cm^3 measuring cylinder
- 3 x dropping pipette
- Bunsen burner, tripod and gauze or electric hotplate
- filter funnel
- filter paper
- wash bottle

- sample of unknown chloride
- deionised water
- 0.5 mol dm^{-3} $AgNO_3$ solution
- 6 mol dm^{-3} HNO_3 solution
- 2 mol dm^{-3} HCl solution
- wash solution – 4 cm^3 of 6 mol dm^{-3} HNO_3 per dm^3 deionised water

SAFETY CONSIDERATIONS

0.5 mol dm^{-3} $AgNO_3$ solution	– corrosive
6 mol dm^{-3} HNO_3 solution	– corrosive
2 mol dm^{-3} HCl solution	– irritant





METHOD

1. Accurately weigh around 0.3 g of the unknown chloride and transfer to a 250 cm^3 beaker.

2. Dissolve the sample in approximately 100 cm^3 of deionised water and add 3 cm^3 of 6 mol dm^{-3} HNO_3.

3. Slowly add 20 cm^3 of $AgNO_3$ solution until silver chloride is seen to coagulate, then add a further 5 cm^3 of $AgNO_3$.

4. Heat the beaker carefully for 10 minutes without allowing the solution to boil.

5. Leave the solution to cool for at least 2 hours.

6. Accurately weigh a filter paper and place in a filter funnel.

7. Decant the liquid from the beaker into the funnel.

8. Wash the precipitate in the beaker with 3 cm^3 of wash solution.

9. Transfer all the precipitate to the filter funnel and wash with 20 cm^3 of wash solution.

10. Test the used wash solution by adding 3 drops of HCl solution. If a precipitate forms (indicating the presence of Ag^+ ions), wash with a further 10 cm^3 and re-test.

11. Dry the precipitate in the filter paper at 105 °C until constant mass is attained.

TEACHER/TECHNICIAN NOTES

Apparatus per laboratory

- 3 decimal place mass balance (minimum 2 decimal place)
- drying oven

Chemicals per laboratory

- deionised water
- wash solution - 4 cm^3 of 6 mol dm^{-3} HNO_3 per dm^3 deionised water

Apparatus per student

- 2 x 250 cm^3 beaker
- 2 x 100 cm^3 beaker
- 1 x spatula
- 10 cm^3 measuring cylinder
- 3 x dropping pipette
- 1 x Bunsen burner, tripod and gauze or electric hotplate
- 1 x filter funnel
- 1 x filter paper
- 1 x wash bottle

Chemicals per student

- ~ 0.3g sample of unknown chloride
- ~ 20 cm^3 0.5 mol dm^{-3} $AgNO_3$ solution
- ~ 3 cm^3 6 mol dm^{-3} HNO_3 solution
- ~ 1 cm^3 2 mol dm^{-3} HCl solution

Notes

Any soluble metal chloride may be used for this analysis. However, it is recommended to use metal chlorides that produce colourless solutions, such as group 1 and group 2 metal chlorides, to allow students to see the formation of AgCl more easily.

IDENTIFICATION OF UNKNOWN SOLUTIONS BY QUALITATIVE ANALYSIS

SPECIFICATION REFERENCE: C1.6

AIM

To plan and carry out a method to identify six inorganic salts by the interactions between their solutions

APPARATUS AND CHEMICALS

- 20 x test tube
- test tube rack
- 6 x dropping pipette

- solutions of the following salts, randomly labelled A–F
    - $Ba(NO_3)_2$
    - $Pb(NO_3)_2$
    - $MgSO_4$
    - KI
    - Na_2CO_3
    - $Zn(NO_3)_2$

SAFETY CONSIDERATIONS

$Ba(NO_3)_2$ - toxic
$Pb(NO_3)_2$ - toxic



PLANNING

1. Construct and complete a table to show the expected observations when each solution interacts with the other five.

2. Record a summary of the observations expected for each salt, e.g. two white precipitates, one yellow precipitate and two 'no visible change'.

3. Construct another similar table but this time labelled with the letters A–F rather than the names of the salts.

METHOD

1. Test approximately 2 cm^3 of each solution with a few drops of each of the other solutions in turn and record your observations in the second table.

TEACHER/TECHNICIAN NOTES

Apparatus per student

- 20 x test tube
- test tube rack
- 6 x dropping pipette

Chemicals per student

- ~ 10 cm^3 of solutions of the following salts, randomly labelled A–F
    - $Ba(NO_3)_2$
    - $Pb(NO_3)_2$
    - $MgSO_4$
    - KI
    - Na_2CO_3
    - $Zn(NO_3)_2$

C2 CHEMICAL CHANGE

C2.1 SIMPLE EQUILIBRIA AND ACID-BASE REACTIONS

Specification Statement		Comment
(a)	reversible reactions and dynamic equilibrium	
(b)	Le Chatelier's principle in deducing the effect of changes in temperature, concentration and pressure	A definition of Le Chatelier's principle is required.
(c)	equilibrium constant (K_c) and calculations involving given equilibrium concentrations	Candidates should realise that only a change in temperature can change the value of K_c.
(d)	acids as donors of $H^+(aq)$ and bases as acceptors of $H^+(aq)$	Candidates should be able to identify and write equations for species reacting as acids and bases.
(e)	relationship between pH and $H^+(aq)$ ion concentration ($pH = -\log[H^+(aq)]$)	Candidates will be expected to calculate pH values from given H^+ ion concentration and vice versa. Prior GCSE knowledge about pH scale is required.
(f)	acid-base titrations	Candidates will be expected to know details of experimental procedures, including apparatus and methods used.
(g)	difference between strong acids and weak acids in terms of relative dissociation	Candidates should be aware that strong and weak does not mean the same as concentrated and dilute.

PREPARATION OF A SOLUBLE SALT BY TITRATION

SPECIFICATION REFERENCE: C2.1

AIM

To prepare crystals of sodium chloride by using titration followed by evaporation

APPARATUS AND CHEMICALS

- 50 cm^3 burette
- funnel
- 25 cm^3 pipette and filler
- 100 cm^3 conical flasks
- evaporating basin

- 0.1 mol dm^{-3} NaOH solution
- 0.1 mol dm^{-3} HCl solution
- phenolphthalein indicator

SAFETY CONSIDERATIONS

0.1 mol dm^{-3} NaOH solution	- irritant
0.1 mol dm^{-3} HCl solution	- irritant
phenolphthalein indicator	- flammable



METHOD

1. Using a pipette, measure 25.00 cm^3 of NaOH solution and pour it into a conical flask.
2. Add two drops of phenolphthalein.
3. Pour the HCl solution into a burette.
4. Record the initial volume of HCl solution.
5. Add the hydrochloric acid from the burette into the conical flask a little at the time while swirling the conical flask.
6. When the phenolphthalein starts to turn from pink to colourless, add the HCl solution a drop at the time until one drop is sufficient to turn the solution colourless. Record the volume of HCl solution needed.
7. Carry out the titration again using 25.00 cm^3 of NaOH solution and exactly the same volume of HCl solution.
8. Do not add indicator this time.
9. Gently heat the solution from the conical flask in an evaporating basin until its volume decreases by around a half.
10. Leave the evaporating basin to cool allowing crystals to form.

TEACHERS/TECHNICIAN NOTES

Chemicals per laboratory

- phenolphthalein indicator

Apparatus per student

- 1 x 50 cm^3 burette
- 1 x funnel
- 1 x 25 cm^3 pipette and filler
- 1 x 100 cm^3 conical flasks
- 1 x evaporating basin

Chemicals per student

- ~ 100 cm^3 0.1 mol dm^{-3} NaOH solution
- ~ 100 cm^3 0.1 mol dm^{-3} HCl solution

STANDARDISATION OF AN ACID SOLUTION

SPECIFICATION REFERENCE: C2.1

AIM

To find the concentration of hydrochloric acid using a standard solution of anhydrous sodium carbonate

APPARATUS AND CHEMICALS

Part 1

- access to a 3 decimal place digital balance (minimum 2 decimal place)
- access to deionised water

- weighing bottle/boat
- 250 cm^3 volumetric flask and funnel
- 250 cm^3 beaker and glass rod
- labels
- wash bottle containing distilled water
- Pasteur pipette

- anhydrous Na_2CO_3

Part 2

- 50cm^3 burette and funnel
- burette clamp and stand
- 25 cm^3 bulb/volumetric pipette with safety filler
- 2 x 250 cm^3 conical flask
- white tile

- 0.2 mol dm^{-3} HCl solution
- methyl orange indicator

SAFETY CONSIDERATIONS

Na_2CO_3 - irritant
0.2 mol dm^{-3} HCl solution - irritant



METHOD

Part 1 – Making up the standard solution

1. Accurately weigh out approximately 2.75 g of anhydrous Na_2CO_3 into a weighing bottle.
2. Record the mass with the correct number of significant figures.
3. Tip the solid into a 250 cm^3 beaker and reweigh the weighing bottle and traces.
4. Dissolve the solid in deionised water, stirring with a glass rod. Ensure you do not add more than 150cm^3 of distilled water at this stage.
5. Pour the solution into the 250 cm^3 volumetric flask via a funnel.
6. Rinse the beaker and glass rod at least three times, transfer the washings into the flask each time.
7. Make the solution up to the mark with distilled water so that the bottom of the meniscus is level with the graduation mark. The flask and graduation mark must be at eye level when the final drops are added using a Pasteur pipette.
8. Add the stopper and shake the mixture thoroughly.
9. Label the volumetric flask ready for use during the second part of the experiment.

Part 2 – Titrating the standard solution with hydrochloric acid

1. Using a funnel, pour a small volume of the HCl solution into the burette. Rinse the burette with this solution to remove any water left behind after washing.
2. Now fill the burette with the HCl solution and record the initial burette reading.
3. Rinse the volumetric pipette with a small volume of sodium carbonate solution.
4. Accurately pipette 25.0 cm^3 of the Na_2CO_3 solution into a conical flask add 3 drops of methyl orange indicator.
5. The standard solution is titrated with the acid until on addition of one drop of acid the indicator changes colour from yellow to pink/red. Use the white tile to help you see the colour change.
6. Record the burette reading and repeat until the results are concordant.

TEACHER/TECHNICIAN NOTES

Apparatus per laboratory

- 3 decimal place digital balance (minimum 2 decimal place)
- labels

Chemicals per laboratory

- deionised water
- methyl orange indicator

Apparatus per student

- 1 x weighing bottle/boat
- 1 x 250 cm^3 volumetric flask and funnel
- 1 x 250 cm^3 beaker and glass rod
- 1 x wash bottle containing distilled water
- 1 x Pasteur pipette
- 1 x 50cm^3 burette and funnel
- 1 x burette clamp and stand
- 1 x 25 cm^3 bulb/volumetric pipette with safety filler
- 2 x 250 cm^3 conical flask
- 1 x white tile

Chemicals per student

- ~ 2.75g anhydrous Na_2CO_3
- ~ 100 cm^3 0.2 mol dm^{-3} HCl solution

BACK TITRATION

SPECIFICATION REFERENCE: C2.1

AIM

Determination of the percentage of calcium carbonate in limestone

APPARATUS AND CHEMICALS

- access to a 3 decimal place digital balance (minimum 2 decimal place)

- 25 cm^3 bulb/volumetric pipette with safety filler
- pestle and mortar
- 250 cm^3 conical flask
- hot plate/hot water bath/Bunsen burner
- 50cm^3 burette and funnel
- burette clamp and stand
- 250 cm^3 conical flask
- white tile

- limestone chips (or antacid tablets)
- 0.1 mol dm^{-3} NaOH solution
- 0.5 mol dm^{-3} HCl solution
- phenolphthalein indicator

SAFETY CONSIDERATIONS

0.1 mol dm^{-3} NaOH solution	- irritant
0.1 mol dm^{-3} HCl solution	- irritant
phenolphthalein indicator	- flammable



PROCEDURE

1. Using a pestle and mortar, grind the limestone chips (or antacid tablet) into a fine powder.

2. Accurately weight approximately 0.5 g of limestone powder and transfer to a conical flask.

3. Record the mass of limestone added.

4. Using a 25 cm^3 pipette, add 25 cm^3 HCl solution.

5. Stir the reaction mixture vigorously with a stirring rod and heat gently with regular stirring.

6. When the limestone has dissolved completely, cool to room temperature.

7. Add a few drops of phenolphthalein indicator and titrate against the standardised NaOH solution.

8. Record the volume of NaOH solution needed to neutralise the excess HCl solution and use this to calculate the percentage $CaCO_3$ in limestone.

TEACHER/TECHNICIAN NOTES

Apparatus per laboratory

- 3 decimal place digital balance (minimum 2 decimal place)

Chemicals per laboratoty

- phenolphthalein indicator

Apparatus per student

- 1 x 25 cm^3 bulb/volumetric pipette with safety filler
- 1 x pestle and mortar
- 1 x 250 cm^3 conical flask
- 1 x hot plate/hot water bath/Bunsen burner
- 1 x 50cm^3 burette and funnel
- 1 x burette clamp and stand
- 1 x 250 cm^3 conical flask
- 1 x white tile

Chemicals per student

- ~ 0.5g limestone chips (or antacid tablets)
- ~ 50cm^3 0.1 mol dm^{-3} NaOH solution
- ~ 25cm^3 0.5 mol dm^{-3} HCl solution

DOUBLE TITRATION

SPECIFICATION REFERENCE: C2.1

AIM

To determine the concentration and the mass of sodium hydroxide and sodium carbonate in a mixed solution

APPARATUS AND CHEMICALS

- 50cm^3 burette and funnel
- burette clamp and stand
- 25 cm^3 bulb/volumetric pipette with safety filler
- 2 x 250 cm^3 conical flask
- white tile

- 0.1 mol dm^{-3} HCl solution
- $NaOH/Na_2CO_3$ mixed solution
- phenolphthalein indicator
- methyl orange indicator

SAFETY CONSIDERATIONS

mixed $NaOH/Na_2CO_3$ solution	- irritant
0.1 mol dm^{-3} HCl solution	- irritant
phenolphthalein indicator	- flammable
methyl orange indicator	- flammable



METHOD

1. Titrate a 25.00 cm^3 sample of the mixed solution against HCl solution using phenolphthalein as the indicator. Do not agitate the flask any more than the minimum necessary to mix the acid.

2. Record the volume used at the phenolphthalein end-point.

3. Add methyl orange and continue titrating until its end-point.

4. Record the total volume of hydrochloric acid added to this end-point.

5. Repeat as necessary until the titration values obtained agree within 0.20 cm^3 and separately average results for the two values.

6. Calculate the concentration of NaOH and Na_2CO_3 in a mixed solution and then the mass in grams.

TEACHER/TECHNICIAN NOTES

Chemicals per laboratory

- phenolphthalein indicator
- methyl orange indicator

Apparatus per student

- 1 x 50cm^3 burette and funnel
- 1 x burette clamp and stand
- 1 x 25 cm^3 bulb/volumetric pipette with safety filler
- 2 x 250 cm^3 conical flask
- 1 x white tile

Chemicals per student

- ~ 100cm^3 0.1 mol dm^{-3} HCl solution
- ~ 25cm^3 NaOH/Na_2CO_3 mixed solution

Notes

Any concentration of NaOH and Na_2CO_3 can be used to prepare the mixed solution.

However, for ease of use, a solution of maximum concentration 0.08 mol dm^{-3} of both NaOH and Na_2CO_3 should be used to ensure a full titration can be carried out with a 50 cm^3 burette of HCl solution.

To prepare this solution, dissolve 3.2g of NaOH and 8.48g of Na_2CO_3 in 1 dm^3 of deionized water.

C2.2 THERMOCHEMISTRY

Specification Statement		Comment
(a)	enthalpy change of reaction, enthalpy change of combustion and standard molar enthalpy change of formation, $\Delta_f H^{\ominus}$	Candidates should understand that reactions can be endothermic or exothermic. Standard conditions are 25 °C (298 K) and 1 atm (100 kPa). Candidates should know what is meant by these enthalpy change terms and be able to use them in calculations.
(b)	Hess's law and energy cycles	Candidates should know Hess's law and be able to use it in calculations.
(c)	concept of average bond enthalpies and how they are used to carry out simple calculations	A formal definition of average bond enthalpy is not required. Calculations to determine average bond enthalpies from given data may also be set.
(d)	how to calculate enthalpy changes	Candidates should be able to calculate enthalpy change from the temperature change in a reaction using the equation $q = mc\Delta T$ and be able to determine molar enthalpy changes from experimental data.
(e)	simple procedures to determine enthalpy changes	Candidates should know how to: • directly determine enthalpy changes for simple reactions between a solid and an aqueous solution, e.g. Zn and $CuSO_4$, and between two solutions, e.g. HCl and NaOH; • indirectly determine enthalpy changes of reaction; • determine enthalpy changes of combustion.

INDIRECT DETERMINATION OF AN ENTHALPY CHANGE OF REACTION

SPECIFICATION REFERENCE: C2.2

AIM

Indirect determination of the enthalpy change of reaction of magnesium oxide and carbon dioxide to form magnesium carbonate

APPARATUS AND CHEMICALS

- access to a 3 decimal place digital balance (minimum 2 decimal place)

- thermometer
- simple calorimeter
- 25 cm^3 bulb/volumetric pipette with safety filler
- stopwatch
- spatula
- weighing boat

- 2 mol dm^{-3} HCl solution
- dry MgO
- dry $MgCO_3$

SAFETY CONSIDERATIONS

2 mol dm^{-3} HCl solution - irritant



METHOD

1. Collect the equipment listed above.
2. Measure 50 cm^3 of HCl solution into the calorimeter using the pipette to measure out 2 measurements of 25 cm^3.
3. Place the thermometer into the HCl solution and leave it in to allow the temperature reading to stabilise.
4. Accurately weigh out approximately 0.90 g of MgO in a weighing boat.
5. Accurately weigh the weighing boat and MgO. Record the mass.
6. Record the temperature of the acid. At the same time, start the stopwatch.
7. Record the temperature of the acid every 30 seconds for 2 minutes and 30 seconds.
8. When the stopwatch reaches 3 minutes, add the MgO to the HCl solution and mix it together thoroughly. Keep the bulb of the thermometer submerged in the reaction mixture.
9. When the stop watch reaches 3 minutes 30 seconds, record the temperature of the reaction mixture.
10. Continue to record the temperature of the reaction mixture every 30 seconds until the temperature drops for 5 consecutive readings.
11. Weigh the weighing boat again. Record the mass. Use this mass and the mass obtained in step 5 to calculate the mass of MgO added to the calorimeter.
12. Use the data to construct an appropriate graph and calculate the enthalpy change of the reaction between MgO and HCl.
13. Repeat steps 2 – 12 using approximately 3.5 g of $MgCO_3$.
14. Use the values of ΔH obtained for each reaction to calculate the enthalpy change for the following reaction:

$$MgO_{(s)} + CO_{2(g)} \rightarrow MgCO_{3(s)}$$

TEACHER/TECHNICIAN NOTES

Apparatus per laboratory

- 3 decimal place digital balance (minimum 2 decimal place)

Apparatus per student

- 1 x thermometer
- 1 x simple calorimeter
- 1 x 25 cm^3 bulb/volumetric pipette with safety filler
- 1 x stopwatch
- 1 x spatula
- 1 x weighing boat

Chemicals per student

- ~ 100 cm^3 2 mol dm^{-3} HCl solution
- ~ 0.9g dry MgO
- ~ 3.5g dry $MgCO_3$

Notes

A simple calorimeter can be made using a polystyrene drinking cup placed inside a glass beaker.

Students can be asked to evaluate the effectiveness of the calorimeter and suggest possible improvements based on their observations.

DETERMINATION OF AN ENTHALPY CHANGE OF COMBUSTION

SPECIFICATION REFERENCE: C2.2

AIM

Determination of the enthalpy change of combustion of methanol and ethanol

APPARATUS AND CHEMICALS

- access to deionised water
- access to a 3 decimal place digital balance (minimum 2 decimal place)

- clamp stand
- 250cm^3 conical flask
- spirit burner
- heatproof mat
- thermometer

- CH_3OH
- CH_3CH_2OH

SAFETY CONSIDERATIONS

CH_3CH_2OH - highly flammable
CH_3OH - highly flammable, toxic





METHOD

1. Add 100 cm^3 of deionised water to a 250 cm^3 conical flask.
2. Clamp the conical flask to a stand at a suitable height so that the spirit burner can be placed below it.
3. Weigh a spirit burner and lid containing methanol using a mass balance and record the mass.
4. Record the initial temperature of the water using the thermometer.
5. Place the spirit burner under the conical flask and light the wick.
6. Allow the flame to heat the water by around 40 °C.
7. Replace the cap to extinguish the flame.
8. Record the final temperature of the water.
9. Re-weigh the spirit burner and cap and record in your table.
10. Calculate the mass of alcohol used.
11. Repeat steps 1-10 for ethanol.
12. Determine the energy released by each alcohol and calculate the enthalpy change of combustion - $\Delta_c H^{\ominus}$.

TEACHER/TECHNICIAN NOTES

Apparatus per laboratory

- 3 decimal place digital balance (minimum 2 decimal place)

Apparatus per student

- clamp stand
- 250cm^3 conical flask
- spirit burner
- heatproof mat
- thermometer

Chemicals per laboratory

- deionised water
- CH_3OH
- CH_3CH_2OH

C2.3 RATES OF REACTION

Specification Statement		Comment
(a)	collision theory in explaining the effects of changing conditions on reaction rate	
(b)	how to calculate rates from experimental data and how to establish the relationship between reactant concentrations and rate	Candidates should be able to use numerical data, plot data and interpret graphs.
(c)	concepts of energy profiles and activation energy	Candidates should be able to draw and interpret energy profiles for endothermic and exothermic reactions.
(d)	rapid increase in rate with temperature in terms of changes in the Boltzmann energy distribution curve	Candidates should be able to draw and interpret energy distribution curves for different temperatures.
(e)	characteristics of a catalyst	Candidates should know the different types of catalysts and why industry relies on catalysts (including enzymes) in terms of green chemistry.
(f)	how catalysts increase reaction rates by providing alternative routes of lower activation energy	Candidates should be able to show this on energy profile diagrams and energy distribution curves.
(g)	how colorimetry can be used in studies of some reaction rates	Candidates should know that colorimetry is used when one of the reactants or products in a reaction is coloured, meaning that there is a change of colour as the reaction proceeds. The intensity of the colour at any given time is related to the concentration of the coloured substance.
(h)	measurement of reaction rate by gas collection and precipitation methods and by an 'iodine clock' reaction	Candidates should be able to outline how each of these methods would be carried out and to select an appropriate method to follow the rate of a given reaction.

INVESTIGATION OF A RATE OF REACTION BY A GAS COLLECTION METHOD

SPECIFICATION REFERENCE: C2.3

AIM

Determination of the rate of reaction of hydrochloric acid and calcium carbonate

APPARATUS AND CHEMICALS

- access to a 3 decimal place digital balance (minimum 2 decimal place)

- 250 cm^3 conical flask
- 100 cm^3 gas syringe with delivery tube and rubber stopper
- 50 cm^3 measuring cylinder
- weighing boat
- spatula
- stopwatch
- clamp and stand

- HCl solutions of different concentrations
- $CaCO_3$ powder

SAFETY CONSIDERATIONS

HCl solution - irritant



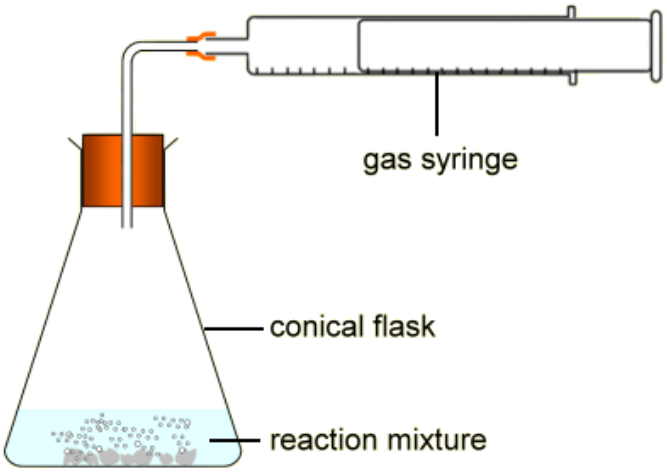
METHOD

1. Measure 50 cm^3 of HCl solution of known concentration into the conical flask using the 50 cm^3 measuring cylinder.

2. Measure out 2 grams of $CaCO_3$ powder using a weighing boat.

3. Set up the gas syringe in a clamp and stand.

4. Put the $CaCO_3$ powder into the conical flask and then quickly put the rubber stopper and delivery tube into the neck of the conical flask. At the same time, start the stopwatch.



5. Stop the stopwatch as soon as the volume of gas in the syringe reaches 100 cm^3.

6. Record this result in an appropriate table and calculate the average rate for this reaction in cm^3s^{-1}.

7. Repeat steps 2-7 using a different concentration of HCl solution.

8. Use your data to plot a graph of rate (1/t) against concentration of HCl solution.

TEACHER/TECHNICIAN NOTES

Apparatus per laboratory

- 3 decimal place digital balance (minimum 2 decimal place)

Apparatus per student

- 250 cm^3 conical flask
- 100 cm^3 gas syringe with delivery tube and rubber stopper
- 50 cm^3 measuring cylinder
- weighing boat
- spatula
- stopwatch
- clamp and stand

Chemicals per student

- HCl solutions of different concentrations
- $CaCO_3$ powder

STUDY OF AN 'IODINE CLOCK' REACTION

SPECIFICATION REFERENCE: C2.3

AIM

To study the kinetics of the oxidation of iodide ions by hydrogen peroxide in acid solution

APPARATUS AND CHEMICALS

- access to deionised water
- stopwatch
- 4 x 10 cm^3 measuring cylinders
- 25 cm^3 bulb/volumetric pipette with safety filler
- 50cm^3 burette and funnel
- burette clamp and stand
- 5 x 250 cm^3 conical flasks

- 0.1 mol dm^{-3} H_2O_2 solution
- 1 mol dm^{-3} H_2SO_4 solution
- 0.1 mol dm^{-3} KI solution
- 0.005 mol dm^{-3} $Na_2S_2O_3$
- starch solution

SAFETY CONSIDERATIONS

1 mol dm^{-3} H_2SO_4 solution - irritant



PLANNING

1. Decide what volumes of H_2O_2 solution and deionised water you will mix together to get at least 5 different concentrations of H_2O_2. The total volume must not exceed 5 cm^3.

GENERAL METHOD

1. In separate conical flasks, make up the solutions according to the table below. Do not add the hydrogen peroxide yet.

2. Add 1 cm^3 starch solution to each flask and mix thoroughly.

3. Rapidly add the hydrogen peroxide to flask 1, starting the stopwatch immediately after this addition.

4. Swirl to mix the reaction mixture thoroughly.

5. Stop timing when the solution turns blue-black and record the time.

6. Repeat the experiment for flasks 2-5.

Flask	Volume H_2SO_4 (cm^3)	Volume $Na_2S_2O_3$ (cm^3)	Volume KI (cm^3)	Volume H_2O (cm^3)	Volume H_2O_2 (cm^3)
1	10	10	25		
2	10	10	25		
3	10	10	25		
4	10	10	25		
5	10	10	25		

TEACHER/TECHNICIAN NOTES

Apparatus per student

- 1 x stopwatch
- 4 x 10 cm^3 measuring cylinders
- 1 x 25 cm^3 bulb/volumetric pipette with safety filler
- 1 x 50cm^3 burette and funnel
- 1 x burette clamp and stand
- 5 x 250 cm^3 conical flasks

Chemicals per laboratory

- deionised water

Chemicals per student

- ~ 15 cm^3 0.1 mol dm^{-3} H_2O_2 solution
- ~ 50 cm^3 1 mol dm^{-3} H_2SO_4 solution
- ~ 125 cm^3 0.1 mol dm^{-3} KI solution
- ~ 50 cm^3 0.005 mol dm^{-3} $Na_2S_2O_3$
- ~ 5 cm^3 starch solution

C2.4 THE WIDER IMPACT OF CHEMISTRY

Specification Statement		Comment
(a)	social, economic and environmental impact of chemical synthesis and the production of energy	Candidates will be expected to apply general principles in order to discuss unfamiliar situations. Knowledge of specific examples/ scenarios is not required.
(b)	role of green chemistry in improving sustainability in all aspects of developments	Candidates should focus on the key principles of green chemistry rather than detailed recall of specific examples.

C3 CHEMISTRY OF CARBON COMPOUNDS

C3.1 ORGANIC COMPOUNDS

Specification Statement		Comment
(a)	how to represent simple organic compounds using shortened, displayed and skeletal formulae	A shortened formula must show the compound unambiguously whilst a displayed formula must show **all** bonds including, for example, that between the O and H in an alcohol.
(b)	nomenclature rules relating to alkanes, alkenes, halogenoalkanes, alcohols and carboxylic acids	Candidates should be able to name and draw molecules in each of these homologous series.
(c)	effect of increasing chain length and the presence of functional groups on melting/boiling temperature and solubility	Candidates should be aware of the effect of temporary dipole (or induced dipole-induced dipole) forces, permanent dipole interactions and hydrogen bonding on melting/boiling temperature and solubility.
(d)	concept of structural isomerism	Candidates should be able to name/draw all isomers corresponding to a particular molecular formula.
(e)	description of species as electrophiles, nucleophiles and radicals and bond fission as homolytic or heterolytic	

C3.2 HYDROCARBONS

Specification Statement		Comment
(a)	combustion reaction of alkanes and benefits and drawbacks relating to the use of fossil fuels, including formation of carbon dioxide, acidic gases and carbon monoxide	Links should be made to social/environmental/green issues.
(b)	C—C and C—H bonds in alkanes as σ-bonds	σ-bonds are 'normal' covalent bonds. Bonds that involve s-s orbital overlap, end-on overlap of a p-orbital with an s-orbital and end-on overlap of two p-orbitals can all be considered as σ-bonds. Reference to orbital hybridisation is not required.
(c)	mechanism of radical substitution, such as the photochlorination of alkanes	
(d)	difference in reactivity between alkanes and alkenes in terms of the C=C bond as a region of high electron density	
(e)	C=C bond in ethene and other alkenes as comprising π-bond and σ-bond	π-bonds involve sideways overlap of p-orbitals above and below the plane of the molecule.
(f)	*E*–*Z* isomerism in terms of restricted rotation about a carbon-carbon double bond	Candidates should understand the significance of the relative atomic mass of the atom joined to the carbon atom involved in the double bond but it is not necessary for them to be able to use the details of the Cahn-Ingold-Prelog rules.
(g)	mechanism of electrophilic addition, such as in the addition of Br_2 to ethene, as a characteristic reaction of alkenes	Curly arrows and the inclusion of dipoles, lone pairs and charges are expected in mechanisms.
(h)	bromine/bromine water and potassium manganate(VII) tests for alkenes	The product of the reaction of alkenes with bromine is required but the nature of the diol formed from the reaction between an alkene and potassium manganate(VII) is not.
(i)	orientation of the normal addition of HBr to propene in terms of the relative stabilities of the possible carbocations involved	The relative stabilities can be explained by considering the electron releasing characteristics of the alkyl groups present.
(j)	conditions required for the catalytic hydrogenation of ethene and the relevance of this reaction	The mechanism of catalytic hydrogenation can be considered to be similar to that of the addition of bromine.
(k)	nature of addition polymerisation and the	The mechanism of addition polymerisation is not required.

	economic importance of the polymers of alkenes and substituted alkenes	

C3.3 HALOGENOALKANES

Specification Statement		Comment
(a)	elimination reaction of halogenoalkanes forming alkenes, for example, HBr eliminated from 1-bromopropane to form propene	The mechanism of this reaction is not required.
(b)	mechanism of nucleophilic substitution, such as in the reaction between OH^-(aq) and primary halogenoalkanes	Curly arrows and the inclusion of dipoles, lone pairs and charges are expected in mechanisms.
(c)	effect of bond polarity and bond enthalpy on the ease of substitution of halogenoalkanes	Candidates should know the **relative** effects of bond polarity and bond enthalpy on the rate of nucleophilic substitution reactions of halogenoalkanes.
(d)	hydrolysis/Ag^+(aq) test for halogenoalkanes	It is expected that if aqueous sodium hydroxide is used in the hydrolysis that this will be neutralised by the addition of dilute nitric acid before adding aqueous silver nitrate.
(e)	halogenoalkanes as solvents, anaesthetics and refrigerants, and tight regulation of their use due to toxicity or adverse environmental effects	Links should be made to social/environmental/green issues.
(f)	adverse environmental effects of CFCs and the relevance of the relative bond strengths of C–H, C–F and C–Cl in determining their impact in the upper atmosphere	
(g)	how to carry out a reflux (for example, for nucleophilic substitution reaction of halogenoalkanes with hydroxide ions)	Candidates should not spend too much time drawing diagrams of apparatus. The basic requirements are that all apparatus is clearly identifiable (labelling is helpful) and that it is shown to be assembled correctly. Condensers should show one tube inside another and labelling should show the direction of water flow.

NUCLEOPHILIC SUBSTITUTION REACTION

SPECIFICATION REFERENCE: C3.3

AIM

To prepare a pure sample of butan-1-ol from 1-bromobutane and sodium hydroxide

APPARATUS AND CHEMICALS

- 100 cm^3 round bottom flask
- 100 cm^3 beaker
- 100 cm^3 conical flask
- 25 cm^3 measuring cylinder
- reflux condenser
- thermometer
- specimen tube
- labels/suitable pen
- anti-bumping granules
- heating mantle / Bunsen burner with water bath
- clamp stand

- 0.1 mol dm^{-3} NaOH solution
- $CH_3CH_2CH_2CH_2Br$

SAFETY CONSIDERATIONS

$CH_3CH_2CH_2CH_2Br$	- flammable, irritant
0.1 mol dm^{-3} NaOH solution	- irritant





METHOD

1. Measure 25 cm^3 of $CH_3CH_2CH_2CH_2Br$ using a measuring cylinder and decant to a round bottomed flask. This step should be performed in the fume cupboard.

2. Add 25 cm^3 of 0.1 mol dm^{-3} NaOH solution and a few anti-bumping granules in to the round bottom flask. This step should be performed in the fume cupboard.

3. Swirl the flask gently to mix the reagents and set up the reflux condenser apparatus for refluxing (diagram 1).

4. Turn on the water to run through the condenser.

5. Turn on the heating mantle (or heat the water bath with a Bunsen burner) and heat the solution to reflux for 15 minutes.

6. Rearrange the apparatus for distillation (diagram 2).

7. Distil off the $CH_3CH_2CH_2CH_2OH$ produced and collect in a clean, dry 100 cm^3 conical flask.

8. Record the temperature at which the liquid product is collected.

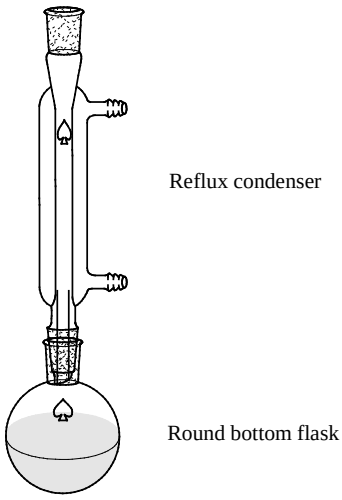


diagram 1

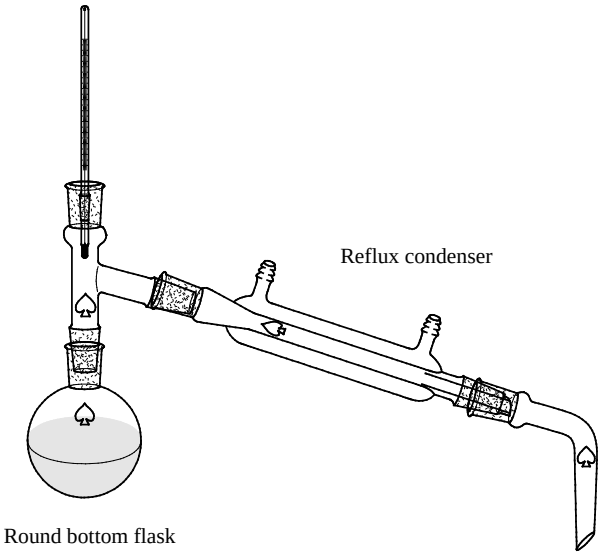


diagram 2

Boiling point of butan-1-ol	

TEACHER/TECHNICIAN NOTES

Apparatus per laboratory

- anti-bumping granules
- fume cupboard

Apparatus per pupil

- 1 x 100 cm^3 round bottom flask
- 1 x 100 cm^3 beaker
- 1 x 100 cm^3 conical flask
- 1 x 25 cm^3 measuring cylinder
- 1 x reflux condenser
- 1 x thermometer
- 1 x specimen tube
- 1 x label
- 1 x heating mantle / Bunsen burner with water bath
- 1 x clamp stand

Chemicals per pupil

- ~ 25 cm^3 0.1 mol dm^{-3} NaOH solution
- ~ 25 cm^3 $CH_3CH_2CH_2CH_2Br$

C3.4 ALCOHOLS AND CARBOXYLIC ACIDS

Specification Statement		Comment
(a)	industrial preparation of ethanol from ethene	
(b)	preparation of ethanol and other alcohols by fermentation followed by distillation, and issues relating to the use of biofuels	This could be linked with work on the wider impact of the uses of biofuels.
(c)	dehydration reactions of alcohols	
(d)	classification of alcohols as primary, secondary and tertiary	
(e)	oxidation of primary alcohols to aldehydes/carboxylic acids and secondary alcohols to ketones	Candidates should use [O] as the oxidising agent when balancing equations. Candidates should be able to draw the aldehydes/carboxylic acids and ketones formed and name them by applying the basic nomenclature rules for organic compounds.
(f)	dichromate(VI) test for primary/secondary alcohols and sodium hydrogencarbonate test for carboxylic acids	It is expected that candidates, as part of their practical experience, will be familiar with test-tube scale experiments designed to identify unknown organic substances.
(g)	reactions of carboxylic acids with bases, carbonates and hydrogencarbonates forming salts	Candidates will be expected to write equations for these reactions and recall observations where applicable.
(h)	esterification reaction that occurs when a carboxylic acid reacts with an alcohol	Candidates will be expected to write equations for esterification reactions. Candidates should be able to draw esters and name those formed from straight chain acids and alcohols.
(i)	separation by distillation	Candidates should not spend too much time drawing diagrams of apparatus. The basic requirements are that all apparatus is clearly identifiable (labelling is helpful) and that it is shown to be assembled correctly. Condensers should show one tube inside another and labelling should show the direction of water flow.

PREPARATION OF AN ESTER AND SEPARATION BY DISTILLATION

SPECIFICATION REFERENCE: C3.4

AIM

To prepare a pure sample of ethyl ethanoate from ethanoic acid and ethanol

APPARATUS AND CHEMICALS

- 100 cm^3 round bottom flask
- 100 cm^3 beaker
- 100 cm^3 conical flask
- 10 cm^3 measuring cylinder
- 25 cm^3 measuring cylinder
- reflux condenser
- thermometer
- specimen tube
- labels/suitable pen
- anti-bumping granules
- warm water bath (approximately 50 °C)
- clamp stand

- CH_3CH_2OH
- CH_3COOH
- concentrated H_2SO_4 solution

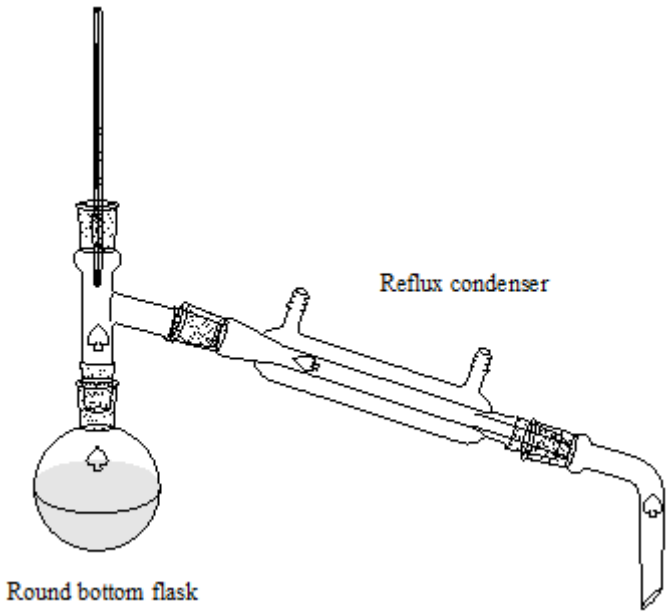
SAFETY CONSIDERATIONS

CH_3CH_2OH	- flammable
CH_3COOH	- irritant
H_2SO_4 solution	- corrosive



METHOD

1. Measure 25 cm^3 of CH_3COOH using a measuring cylinder and decant to a round bottomed flask.
2. Add 10 cm^3 of CH_3CH_2OH solution and a few anti-bumping granules in to the round bottom flask.
3. Add 10 drops of concentrated H_2SO_4 solution.
4. Swirl the flask gently to mix the reagents.
5. Warm the reaction mixture gently with the warm water bath for 15 minutes.
6. Set up the distillation apparatus (see diagram).
7. Distil off the $CH_3COOCH_2CH_3$ produced and collect in a clean, dry 100 cm^3 conical flask.
8. Record the temperature at which the liquid product is collected.



Boiling point of ethyl ethanoate	

TEACHER/TECHNICIAN NOTES

Apparatus per laboratory

- labels
- anti-bumping granules
- warm water bath (approximately 50 °C)

Apparatus per student

- 1 x 100 cm^3 round bottom flask
- 1 x 100 cm^3 beaker
- 1 x 100 cm^3 conical flask
- 1 x 10 cm^3 measuring cylinder
- 1 x 25 cm^3 measuring cylinder
- 1 x reflux condenser
- 1 x thermometer
- 1 x specimen tube
- 1 x clamp stand

Chemicals per student

- ~ 10 cm^3 CH_3CH_2OH
- ~ 25 cm^3 CH_3COOH
- ~ 1 cm^3 concentrated H_2SO_4 solution

C3.5 INSTRUMENTAL ANALYSIS

Specification Statement		Comment
(a)	use of mass spectra in identification of chemical structure	Detailed knowledge of the instrument used in analytical techniques is not required. Candidates should be able to **interpret** given spectra and **predict** the spectra for given compounds.
(b)	use of IR spectra in identification of chemical structure	
(c)	use of ^{13}C and low resolution ^{1}H MR spectra in identification of chemical structure	

PHYSICAL AND INORGANIC CHEMISTRY

PI1 ELECTROCHEMISTRY

PI1.1 REDOX AND STANDARD ELECTRODE POTENTIAL

Specification Statement		Comment
(a)	redox reactions in terms of electron transfer	This is linked to AS section 1.6.
(b)	how to represent redox systems in terms of ion/electron half-equations and as half-cells in cell diagrams	Candidates should be able to write and use half-equations and cell diagrams for familiar oxidising or reducing agents such as those referred to in sections 3.2, 3.3 and 3.4. They should be able to write and use unfamiliar half-equations from information given, including those that require the inclusion of other species such as H^+ and H_2O to balance.
(c)	concept of standard electrode potential and role of the standard hydrogen electrode	Candidates should recall the exact nature of the standard hydrogen electrode – its constituents and the conditions required.
(d)	how simple electrochemical cells are formed by combining electrodes (metal/metal ion electrodes and electrodes based on different oxidation states of the same element)	Electrodes based on different oxidation states include cases where there are two ions in solution such as Fe^{2+}/Fe^{3+} or $Cr^{3+}/Cr_2O_7^{2-}$, but also cases where the oxidation state of one species is zero, such as Cl_2/Cl^-.
(e)	concept of cell EMF and its significance in terms of the feasibility of reactions	Candidates should be able to predict and explain feasibility in terms of the sign of the cell EMF but also by comparing standard electrode potentials.
(f)	principles of the hydrogen fuel cell and its benefits and drawbacks	The benefits and drawbacks should be linked to chemical principles.

CONSTRUCTION OF ELECTROCHEMICAL CELLS AND MEASUREMENT OF E_{CELL}

SPECIFICATION REFERENCE: PI 1.1

AIM

Construction of a copper/zinc electrochemical cell and the determination of E_{cell}

APPARATUS AND CHEMICALS

- wires
- crocodile clips
- voltmeter
- 2 x 100 cm^3 beakers
- filter paper cut into a long strip

- Cu foil strip
- Zn foil strip
- 1 mol dm^{-3} $CuSO_4$ solution
- 1 mo ldm^{-3} $ZnSO_4$ solution
- saturated KNO_3 solution

SAFETY CONSIDERATIONS

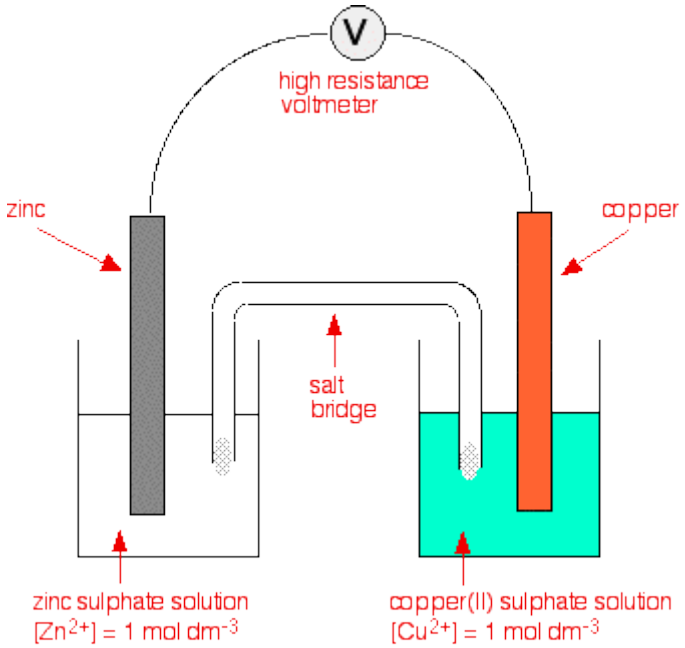
1 mol dm^{-3} $CuSO_4$ solution	- harmful, dangerous to environment
1 mol dm^{-3} $ZnSO_4$ solution	- irritant, dangerous to environment
Saturated KNO_3 solution	- oxidising



METHOD

1. Measure 50 cm^3 of $CuSO_4$ solution into one of the beakers.
2. Measure 50 cm^3 of $ZnSO_4$ solution into the other beaker.
3. Place the copper foil in the $CuSO_4$ solution.
4. Place the zinc foil in the $ZnSO_4$ solution.
5. Connect the zinc foil strip and Cu foil strip to the ammeter.
6. Connect the voltmeter across the Zn foil and copper foil connections.
7. Place the two beakers directly next to each other.
8. Soak the filter paper in the saturated KNO_3 solution and place it across the two beakers.
9. Ensure that the ends of the filter paper are in direct contact with the solutions in the two beakers. This is called a salt bridge.
10. Measure the potential difference of the cell using the voltmeter.

DIAGRAM



Potential difference of cell	

TEACHER/TECHNICIAN NOTES

Apparatus per student

- 2 x electrical wire
- 2 x crocodile clip
- 1 x high resistance voltmeter
- 2 x 100 cm^3 beaker
- 1 x filter paper cut into a long strip

Chemicals per student

- 1 x Cu foil strip
- 1 x Zn foil strip
- ~ 50cm^3 1 mol dm^{-3} $CuSO_4$ solution
- ~ 50cm^3 1 mo ldm^{-3} $ZnSO_4$ solution
- ~ 5cm^3 saturated KNO_3 solution

PI1.2 REDOX REACTIONS

Specification Statement		Comment
(a)	how to construct ion/electron half-equations, for example, for the reduction of acidified $Cr_2O_7^{2-}$ to Cr^{3+} and acidified MnO_4^- to Mn^{2+} and the oxidation of $S_2O_3^{2-}$ to $S_4O_6^{2-}$	Candidates should be able to construct unfamiliar ion/electron half-equations when given appropriate information.
(b)	how to combine half-equations to give a stoichiometric redox equation	Candidates should be able to combine both familiar and unfamiliar half-equations.
(c)	redox reaction between Cu^{2+} and I^- and the determination of the liberated iodine with $S_2O_3^{2-}$	Candidates should recall the products of the reaction which liberates I_2 and be able to show the 1≡1 equivalence of $S_2O_3^{2-}$ and Cu^{2+} ions.
(d)	how to carry out a redox titration	Candidates will be required to recall the practical details and principles relating to redox titrations in general. They should understand the difference between redox and acid-base titrations. They will acquire this knowledge and understanding and develop their skills by completing the specified practical work included in this section.

SIMPLE REDOX TITRATION

SPECIFICATION REFERENCE: PI 1.2

AIM

To determine the relative molecular mass of an iron(II) salt by titration with standard potassium manganate(VII) solution

APPARATUS AND CHEMICALS

- access to 3 decimal place mass balance (minimum 2 decimal place)

- 50 cm^3 burette and funnel
- 25 cm^3 pipette and filler
- 250 cm^3 conical flasks
- 250 cm^3 volumetric flask

- unknown iron(II) salt
- 1.0 mol dm^{-3} H_2SO_4 solution
- standardised $KMnO_4$ solution (approximately 0.02 mol dm^{-3})

SAFETY CONSIDERATIONS

1.0 mol dm^{-3} H_2SO_4 solution	- irritant
$KMnO_4$ solution	- harmful, oxidising



METHOD

1. Weigh out accurately, about 9.8 g of the iron(II) salt provided and record the mass.
2. Make the salt up to 250 cm^3 of a standard solution in H_2SO_4 solution.
3. Titrate 25 cm^3 portions of this solution against the standardised $KMnO_4$ solution.
4. Use your results to calculate the relative molecular mass of the iron(II) salt.

TEACHER/TECHNICIAN NOTES

Apparatus per classroom

- 3 decimal place mass balance (minimum 2 decimal place)

Apparatus per student

- 1 x 150 cm^3 burette and funnel
- 1 x 25 cm^3 pipette and filler
- 1 x 250 cm^3 conical flasks
- 1 x 250 cm^3 volumetric flask

Chemicals per student

- unknown iron(II) salt
- ~ 250 cm^3 1.0 mol dm^{-3} H_2SO_4 solution
- ~ 100 cm^3 standardised $KMnO_4$ solution (~0.02 mol dm^{-3})

Notes

Any soluble iron(II) salt can be used. Suitable examples include iron(II) sulfate and iron(II) ammonium sulfate.

ESTIMATION OF COPPER IN COPPER(II) SALTS

SPECIFICATION REFERENCE: PI 1.2

AIM

To determine the percentage by mass of copper in copper(II) sulfate crystals

APPARATUS AND CHEMICALS

- access to a 3 decimal place mass balance (minimum 2 decimal place)
- weighing boat
- 2 x 50 cm^3 beakers
- 250 cm^3 beaker
- 3 x 250 cm^3 conical flask
- 250 cm^3 volumetric flask with stopper
- 25 cm^3 bulb pipette
- pipette filler
- burette, funnel and stand
- white tile
- wash bottle of de-ionised water
- 2 x Pasteur pipettes

- $CuSO_4.5H_2O$ crystals
- 0.1 mol dm^{-3} $Na_2S_2O_3$ solution
- 0.2% starch solution
- KI powder

SAFETY CONSIDERATIONS

$CuSO_4.5H_2O$ crystals - harmful, dangerous to the environment
KI powder - irritant





METHOD

1. Accurately weigh out about 6 g copper(II) sulfate crystals into a weighing boat.
2. Use the copper(II) sulfate crystals to make up 250 cm^3 of standardised copper(II) sulfate solution.
3. Pipette 25 cm^3 of this solution into a conical flask.
4. Add 1.5 g potassium iodide and swirl thoroughly.
5. Titrate this solution with standard 0.1 mol dm^{-3} $Na_2S_2O_3$ in a burette. When the iodine colour fades, add 1 cm^3 starch indicator.
6. Use your titration data to calculate the percentage by mass of copper in the copper(II) sulfate crystals.

TEACHER/TECHNICIAN NOTES

Apparatus per laboratory

- 3 decimal place mass balance (minimum 2 decimal place)

Chemicals per laboratory

- deionised water

Apparatus per student

- 2 x weighing boat
- 2 x 50 cm^3 beakers
- 1 x 250 cm^3 beaker
- 3 x 250 cm^3 conical flask
- 1 x 250 cm^3 volumetric flask with stopper
- 1 x 25 cm^3 bulb pipette
- 1 x pipette filler
- 1 x burette, funnel and stand
- 1 x white tile
- 1 x wash bottle of de-ionised water
- 2 x Pasteur pipettes

Chemicals per student

- ~ 6g $CuSO_4.5H_2O$ crystals
- ~ 50 cm^3 0.1 mol dm^{-3} $Na_2S_2O_3$ solution
- ~ 1 cm^3 0.2% starch solution
- ~ 1.5g KI powder

PI2 MORE COMPLEX PATTERNS OF THE PERIODIC TABLE

PI2.1 CHEMISTRY OF THE *P*-BLOCK

Specification Statement		Comment
(a)	amphoteric behaviour of *p*-block elements as demonstrated by the reactions of Al^{3+}/Al and Pb^{2+}/Pb	
(b)	increasing stability of the inert pair cations on descent of Groups 3, 4 and 5	
(c)	how some Group 3 elements can form compounds with fewer than eight electrons in their valence shells and some elements of Groups 5, 6 and 7 can form compounds with more than eight	
(d)	structure and bonding in Al_2Cl_6 and formation of donor-acceptor compounds such as $NH_3.BF_3$	Candidates should be able to differentiate between standard covalent and co-ordinate bonds in dot-and-cross diagrams.
(e)	bonding and structure in hexagonal and cubic boron nitride and how these relate to their properties and uses	The recall of the structures of both forms of boron nitride needs to focus on the key similarities and differences when compared with graphite and diamond. The differences between hexagonal boron nitride and graphite are: • atoms in adjacent layers in register for BN and out of register for graphite; • π-electrons relatively evenly delocalised in graphite but more localised in BN – this makes BN an insulator; • electronegativity difference between B and N whereas there is no electronegativity difference in C.
(f)	change in relative stability of oxidation states II and IV down Group 4 as shown by reactions of CO as a reducing agent with oxides and Pb(IV) as an oxidising agent in the reaction of PbO_2	
(g)	nature, physical properties and acid-base properties of CO_2 and PbO	These properties should be linked to the differing bonding types present.
(h)	change in the types of bonding down Group 4 as shown by the	

	chlorides CCl_4, $SiCl_4$ and $PbCl_2$ and their reactions with water	
(i)	reactions of $Pb^{2+}(aq)$ with aqueous NaOH, Cl^- and I^-	The reaction with small amounts and excess OH^- are required.
(j)	reactions of Cl_2 with both cold and warm aqueous NaOH and the various disproportionation reactions involved	
(k)	bleaching and bactericidal action of Cl_2 and chlorate(I) (ClO^-) resulting from their oxidising power	
(l)	differences in behaviour of NaCl, NaBr and NaI with concentrated sulfuric acid (formation and subsequent reactions of HX)	Balanced equations for the redox reactions that occur for NaBr and NaI are not required.

PI2.2 CHEMISTRY OF THE *D*-BLOCK TRANSITION METALS

Specification Statement		Comment
(a)	how the *d*-block elements attain various oxidation states in their compounds	
(b)	the most important oxidation states of Cr, Mn, Fe, Co and Cu and the colours of aqueous solutions of compounds containing Cr^{3+}, CrO_4^{2-}, $Cr_2O_7^{2-}$, MnO_4^-, Co^{2+}, Fe^{2+}, Fe^{3+} and Cu^{2+}	Candidates should recall all stable oxidation states for Cr, Mn, Fe, Co and Cu.
(c)	bonding in tetrahedral and octahedral complexes	
(d)	origin of colour in transition metal complexes, as exemplified by octahedral 6-coordinate species such as $[Cu(H_2O)_6]^{2+}$ and $[Fe(H_2O)_6]^{3+}$, in terms of the splitting of d-orbitals	Candidates do not need to discuss high and low spin complexes but need to be aware that different ligands lead to different splittings and therefore different colours.
(e)	idea of ligand exchange and how this can lead to a change in coordination number as exemplified by the reactions of $[Cu(H_2O)_6]^{2+}$ and $[Co(H_2O)_6]^{2+}$ with concentrated HCl	Candidates are not required to discuss the trans-effect for complexes containing mixed ligands.
(f)	colours and formulae of the approximately octahedral complex ions $[Cu(H_2O)_6]^{2+}$, $[Cu(NH_3)_4(H_2O)_2]^{2+}$ and $[Co(H_2O)_6]^{2+}$ and the approximately tetrahedral ions $[CuCl_4]^{2-}$ and $[CoCl_4]^{2-}$	Reference to Jahn-Teller distortion is not required.
(g)	catalytic properties of many transition metals and their compounds; heterogeneous catalysis as a result of surface adsorption and homogeneous catalysis as a result of variable oxidation state	
(h)	nickel and iron as the catalysts used in the hydrogenation of alkenes and the Haber process respectively	

(i)	vanadium(V) oxide as the catalyst used in the contact process and manganese(IV) oxide as an effective catalyst for the decomposition of hydrogen peroxide	
(j)	reactions of Cr^{3+}, Fe^{2+}, Fe^{3+} and Cu^{2+} with excess aqueous OH^-	The reaction with small amounts and excess OH^- are required.

PI3 CHEMICAL KINETICS

Specification Statement		Comment
(a)	principles underlying the measurement of reaction rate by sampling and quenching	This approach may be linked to any analytical technique in the specification.
(b)	how reaction order is found from experimental results	
(c)	the general rate equation and how it is used	
(d)	concept of a rate determining step	
(e)	link between reaction kinetics and mechanism	Candidates should be able to suggest rate determining steps and mechanisms for familiar and unfamiliar reactions.
(f)	the Arrhenius equation to show the effect of changing temperature and the use of a catalyst on the rate constant, and to find the activation energy and frequency factor of a reaction	This is new to the specification. Candidates will be required to apply the Arrhenius equation in the form $k = Ae^{(-E_a/RT)}$ and undertake relevant calculations, which may involve rearrangement of the exponential. Interpretation or plotting of relevant graphs will be required.

DETERMINATION OF THE ORDER OF A REACTION

SPECIFICATION REFERENCE: PI 3

AIM

To determine of the order of reaction for the oxidation of iodide ions by hydrogen peroxide in acid solution

APPARATUS AND CHEMICALS

- stopwatch
- 100 cm^3 conical flask
- stirring rod
- 4 x 10 cm^3 measuring cylinder
- 5 cm^3 measuring cylinder
- 1 cm^3 measuring cylinder

- 0.1 mol dm^{-3} H_2O_2 solution
- 1.0 mol dm^{-3} H_2SO_4 solution
- 0.1 mol dm^{-3} KI solution
- 0.005 mol dm^{-3} $Na_2S_2O_3$ solution
- starch solution

SAFETY CONSIDERATIONS

H_2O_2 solution	- harmful, oxidising
H_2SO_4 solution	- irritant



PLANNING

1. Decide what volumes of H_2O_2 solution and deionised water you will mix together to get at least 5 different concentrations of H_2O_2. The total volume must not exceed 5 cm^3.

GENERAL METHOD

1. Prepare the reaction mixture by adding the following reagents to a 100 cm^3 conical flask.

    - 10.0 cm^3 H_2SO_4 solution
    - 10.0 cm^3 $Na_2S_2O_3$ solution
    - 15.0 cm^3 KI solution
    - 1.0 cm^3 starch solution
    - 9.0 cm^3 deionised water

2. Be ready with the stopwatch. Rapidly add 5.0 cm^3 of H_2O_2 solution to the reaction mixture and simultaneously start the stopwatch. Ensure the reaction mixture is thoroughly mixed.

3. Stop the watch immediately when the blue colour appears and record the time.

4. Repeat steps 1-3 using the other four concentrations of H_2O_2 solution.

5. Calculate the rate of reaction for each experiment.

6. Plot a graph of rate of reaction against [H_2O_2] solution and use this to calculate the order of reaction with respect to [H_2O_2].

TEACHER/TECHNICIAN NOTES

Chemicals per laboratory

- deionised water

Apparatus per student

- stopwatch
- 100 cm^3 conical flask
- stirring rod
- 4 x 10 cm^3 measuring cylinder
- 5 cm^3 measuring cylinder
- 1 cm^3 measuring cylinder

Chemicals per student

- ~ 15 cm^3 0.1 mol dm^{-3} H_2O_2 solution
- ~ 50 cm^3 1.0 mol dm^{-3} H_2SO_4 solution
- ~ 75 cm^3 0.1 mol dm^{-3} KI solution
- ~ 50 cm^3 0.005 mol dm^{-3} $Na_2S_2O_3$ solution
- ~ 5 cm^3 starch solution

PI4 ENERGY CHANGES

PI4.1 ENTHALPY CHANGES FOR SOLIDS AND SOLUTIONS

Specification Statement		Comment
(a)	enthalpy changes of atomisation, lattice formation and breaking, hydration and solution	
(b)	how solubility of ionic compounds in water (enthalpy change of solution) depends on the balance between the enthalpy change of lattice breaking and the hydration enthalpies of the ions	
(c)	processes involved in the formation of simple ionic compounds as described in a Born-Haber cycle	Candidates will be expected to construct and use Born-Haber cycles.
(d)	exothermicity or endothermicity of $\Delta_f H^{\ominus}$ as a qualitative indicator of a compound's stability	

PI4.2 ENTROPY AND FEASIBILITY OF REACTIONS

Specification Statement		Comment
(a)	entropy, *S*, of a system as a measure of the freedom possessed by particles within it and the increase in entropy towards a maximum for all natural changes	
(b)	particles in a solid having much less freedom than those in a gas and that, other factors being equal, entropy increasing in the sequence S(gas) > S(liquid) > S(solid)	
(c)	how to calculate an entropy change from absolute entropy values, $\Delta S = S_{final} - S_{initial}$	This calculation which was previously implied in the specification is now stated explicitly.
(d)	concept of Gibbs free energy change and how it is calculated using the relationship, $\Delta G = \Delta H - T\Delta S$	
(e)	spontaneous reactions having a negative value for ΔG and how the effect of entropy change explains the spontaneous occurrence of endothermic processes	This concept may be used in a range of calculations, including those identifying the minimum temperature for a reaction to be spontaneous.

PI5 EQUILIBRIA

PI5.1 EQUILIBRIUM CONSTANTS

Specification Statement		Comment
(a)	effect of temperature on K_p and K_c for exothermic and endothermic reactions	
(b)	how to calculate values of K_p and K_c and quantities present at equilibrium from given data	Questions involving degree of dissociation will not be set.
(c)	significance of the magnitude of an equilibrium constant and how this relates to the position of equilibrium	

DETERMINATION OF AN EQUILIBRIUM CONSTANT

SPECIFICATION REFERENCE: PI 5.1

AIM

To determine the equilibrium constant for the reaction of ethanol with ethanoic acid

APPARATUS AND CHEMICALS

- communal burette of glacial CH_3COOH
- communal burette of CH_3CH_2OH
- communal burette of deionised H_2O
- communal burette of 1.0 mol dm^{-3} of HCl solution

- 2 x 100 cm^3 conical flask with stopper
- 2 x 250 cm^3 conical flask
- 50 cm^3 burette and funnel
- burette clamp and stand
- 1 cm^3 measuring cylinder (or 1 cm^3 pipette with filler)
- 100 cm^3 measuring cylinder

- deionised water in wash bottle
- standard solution of 0.1 $moldm^{-3}$ NaOH solution
- phenolphthalein indicator

SAFETY CONSIDERATIONS

1.0 mol dm^{-3} HCl solution	- irritant
glacial CH_3COOH	- irritant
CH_3CH_2OH	- flammable
0.1 $moldm^{-3}$ NaOH solution	- irritant



METHOD PART 1

1. Prepare the control flask and reaction flask by mixing the appropriate chemicals in the quantities listed in the table below:

All volumes in cm^3

Reagent	Density/g cm^{-3}	The control	The mixture
glacial ethanoic acid	1.05	0.0	6.0
ethanol	0.79	0.0	6.0
water	1.00	18.0	6.0
1 mol dm^{-3} hydrochloric acid	1.00	2.0	2.0
Total volume		20.0	20.0

2. Stopper, label and shake both flasks and leave them for at least one week.

3. Using the densities and volumes given for each reagent, calculate the initial amount of CH_3COOH, CH_3CH_2OH and H_2O in moles. You should assume that 2.0 cm^3 of the 1 mol dm^{-3} HCl solution adds an extra 2.0 cm^3 of H_2O to the mixture.

METHOD PART 2

1. Empty the contents of the control flask mixture into a 250 cm^3 conical flask.

2. Add 100 cm^3 of deionised H_2O and 2 drops of phenolphthalein to the flask.

3. Titrate the mixture against the standard solution of NaOH. Record the results of this titration.

4. Transfer 1.0 cm^3 of the reaction mixture to a 250 cm^3 conical flask.

5. Add 100 cm^3 of deionised water and 2 drops of phenolphthalein to the flask.

6. Swirl the flask and titrate the mixture against the standard solution of sodium hydroxide solution as quickly as possible.

7. Repeat steps 4 to 6 with further 1.0 cm^3 samples of the equilibrium mixture until you have three concordant results.

8. Use the results of the titrations to calculate the equilibrium constant of the reaction.

TEACHER/TECHNICIAN NOTES

Apparatus per laboratory

- 4 x burette, clamp and stand

Chemicals per laboratory

- glacial CH_3COOH
- CH_3CH_2OH
- deionised water
- 1.0 mol dm^{-3} of HCl solution
- phenolphthalein indicator

Apparatus per student

- 2 x 100 cm^3 conical flask with stopper
- 2 x 250 cm^3 conical flask
- 1 x 50 cm^3 burette and funnel
- 1 x burette clamp and stand
- 1 x 1 cm^3 measuring cylinder (or 1 cm^3 pipette with filler)
- 1 x 100 cm^3 measuring cylinder

Chemicals per student

- ~ 50 cm^3 0.1 $moldm^{-3}$ NaOH solution

PI5.2 ACID-BASE EQUILIBRIA

Specification Statement		Comment
(a)	Lowry-Bronsted theory of acids and bases	Candidates should be familiar with the ideas of conjugate acids and bases. Other theories of acids and bases are not required.
(b)	differences in behaviour between strong and weak acids and bases in terms of the acid dissociation constant, K_a	
(c)	significance of the ionic product of water, K_w	
(d)	how to use pH, K_w, K_a and pK_a in calculations involving strong and weak acids and pH and K_w in calculations involving strong bases	
(e)	shapes of the titration curves for strong acid/strong base, strong acid/weak base, weak acid/strong base and weak acid/weak base systems	These curves should be linked to pH calculations for pure substances as well as buffer solutions.
(f)	mode of action of buffer solutions and how to use pH, K_w, K_a and pK_a in appropriate calculations	
(g)	importance of buffer solutions in living systems and industrial processes	
(h)	concept of hydrolysis of salts of a strong acid/strong base, a strong acid/weak base and a weak acid/strong base	Only a qualitative description is required here.
(i)	how suitable indicators are selected for acid-base titrations	

TITRATION USING A pH PROBE

SPECIFICATION REFERENCE: PI 5.2

AIM

To obtain titration curves for strong and weak acids and alkalis

APPARATUS AND CHEMICALS

- access to deionised water

- 25 cm^3 bulb/volumetric pipette with safety filler
- 50cm^3 burette and funnel
- burette clamp and stand
- 100 cm^3 beaker
- 250 cm^3 conical flask
- pH meter
- data logger (optional)

- pH 4 buffer solution
- pH 9 buffer solution
- 0.1 mol dm^{-3} HCl solution
- 0.1 mol dm^{-3} CH_3COOH solution
- 0.1 mol dm^{-3} NaOH solution
- 0.1 mol dm^{-3} NH_3 solution

SAFETY CONSIDERATIONS

pH 4 buffer solution	- irritant
pH 9 buffer solution	- irritant
0.1 mol dm^{-3} HCl solution	- irritant
0.1 mol dm^{-3} CH_3COOH solution	- irritant
0.1 mol dm^{-3} NaOH solution	- irritant
0.1 mol dm^{-3} NH_3 solution	- irritant



METHOD

1. Calibrate the pH meter by immersing the electrodes in the pH 4 buffer solution, swirl the beaker, and adjust the reading to 4.0. Remove the electrode, wash with distilled water and immerse in the pH 9 buffer solution. The pH should read 9.0.

2. Using the pipette and filler, transfer 25.0cm^3 of acid to the conical flask and measure the pH.

3. Add the alkali, 5cm^3 at a time up to 50cm^3, and measure the pH after each 5cm^3 addition. Wash the electrode with distilled water between measurements.

4. If there is time available, repeat the procedure for all combinations of acid and alkali. If not, collaborate with other students so that your class has at least one set of results for each of the following combinations:

    - HCl and NaOH
    - CH_3COOH and NaOH
    - HCl and NH_3
    - CH_3COOH and NH_3

5. Plot graphs of volume of alkali against pH. Read off the pH at the equivalence point for each titration.

TEACHER/TECHNICIAN NOTES

Chemicals per laboratory

- deionised water

Apparatus per student

- 1 x 25 cm^3 bulb/volumetric pipette with safety filler
- 1 x 50cm^3 burette and funnel
- 1 x burette clamp and stand
- 1 x 100 cm^3 beaker
- 1 x 250 cm^3 conical flask
- 1 x pH meter
- 1 x data logger (optional)

Chemicals per student

- ~ 25 cm^3 pH 4 buffer solution
- ~ 25 cm^3 pH 9 buffer solution
- ~ 150 cm^3 0.1 mol dm^{-3} HCl solution
- ~ 150 cm^3 0.1 mol dm^{-3} CH_3COOH solution
- ~ 150 cm^3 0.1 mol dm^{-3} NaOH solution
- ~ 150 cm^3 0.1 mol dm^{-3} NH_3 solution

Notes

The volumes of the acids and alkalis are based on each student performing the four different titrations individually. If students work collaboratively then a lower volume of each solution will be required.

ORGANIC CHEMISTRY AND ANALYSIS

OA1 HIGHER CONCEPTS IN ORGANIC CHEMISTRY

OA1.1 STEREOISOMERISM

Specification Statement		Comment
(a)	how stereoisomerism is distinct from structural isomerism and that stereoisomerism encompasses *E*–*Z* isomerism and optical isomerism	Stereoisomerism is concerned with the arrangements of atoms in space. A clear distinction should be made between this type of isomerism and structural isomerism.
(b)	the terms chiral centre, enantiomer, optical activity and racemic mixture	Questions may be set where there is more than one chiral centre in the molecule. However, knowledge of diastereoisomerism and the chirality of alicyclic compounds are not required.
(c)	optical isomerism in terms of an asymmetric carbon atom	Optical isomerism involving atoms other than carbon, e.g. silicon and transition metals, will not be set without detailed information being provided.
(d)	effect of an enantiomer on plane-polarised light	Candidates should know that enantiomers rotate the plane of polarised light in opposite directions.

OA1.2 AROMATICITY

Specification Statement		Comment
(a)	structure of and bonding in benzene and other arenes	The term 'other arenes' refers to benzene derivatives, e.g. methylbenzene and phenol. It will not be extended to the consideration of fused ring systems such as naphthalene and anthracene.
(b)	resistance to addition reactions shown by aromatic compounds such as benzene	A comparison should be made between addition to alkenes and to arenes.
(c)	mechanism of electrophilic substitution, such as in the nitration, halogenation and Friedel-Crafts alkylation of benzene, as the characteristic reaction of arenes	Electrophilic substitution reactions could be extended to substitution in alkylbenzenes and other benzene derivatives. Knowledge of directional effects in electrophilic substitution is not required.
(d)	interaction between benzene and substituent groups, as exemplified by the increase in C—Cl bond strength in chlorobenzene when compared with a chloroalkane	The greater bond strength in chlorobenzene is believed to result from overlap between *p*-electrons on the chlorine and the delocalised π-system in the ring to give some π-character to the bond and thus strengthen it.

OA2 ORGANIC COMPOUNDS CONTAINING OXYGEN

OA2.1 ALCOHOLS AND PHENOLS

Specification Statement		Comment
(a)	methods of forming primary and secondary alcohols from halogenoalkanes and carbonyl compounds	This section includes the nucleophilic substitution of OH^- ions on halogenoalkanes. There is no requirement to study the mechanism in terms of unimolecular or bimolecular processes (S_N1 and S_N2). Candidates should know that $LiAlH_4$ (and not $NaBH_4$) is able to reduce carboxylic acids to alcohols. The mechanism of this reaction will not be set without adequate guidance being given.
(b)	reactions of primary and secondary alcohols with hydrogen halides and ethanoyl chloride and carboxylic acids	The reaction of primary and secondary alcohols with hydrogen chloride requires anhydrous zinc chloride as a catalyst. For the preparation of the bromo-compounds from the corresponding alcohol, a mixture of potassium bromide and concentrated sulfuric acid is often used (an in-situ reaction).
(c)	acidity of phenol and its reactions with bromine and ethanoyl chloride	Phenol is only very weakly acidic, reacting with NaOH but not with Na_2CO_3. Candidates should recall the observations made when bromine reacts with phenol and be able to write an equation for the reaction.
(d)	$FeCl_3(aq)$ test for phenols	The observation is all that is required here. Knowledge of the chemistry involved in the reaction is not needed.

OA2.2 ALDEHYDES AND KETONES

Specification Statement		Comment
(a)	formation of aldehydes and ketones by the oxidation of primary and secondary alcohols respectively	Acidified dichromate(VI) or acidified manganate(VII) solutions are the usual oxidising agents. The relevant colour changes that occur are required. Any equation will use [O] as the formula of the oxidising agent.
(b)	how aldehydes and ketones may be distinguished by their relative ease of oxidation using Tollens' reagent and Fehling's reagent	The observations for these reactions are important but the full chemical equations are not needed.
(c)	reduction of aldehydes and ketones using $NaBH_4$	If equations are required, then [H] will be acceptable as the formula of the reducing agent.
(d)	mechanism of nucleophilic addition, such as in the addition of HCN to ethanal and propanone, as a characteristic reaction of aldehydes and ketones	Candidates should be aware that these reactions lead to an increase in the C—C chain length ('ascending the homologous series') and that hydrolysis of hydroxynitriles leads to important hydroxyacids.
(e)	reaction of aldehydes and ketones with 2,4-dinitrophenylhydrazine and its use as a test for a carbonyl group and in identifying specific aldehydes and ketones	Full details of the mechanism for this reaction are not required but candidates should be aware that this is an addition-elimination reaction (condensation).
(f)	triiodomethane (iodoform) test and its use in identifying CH_3CO— groups or their precursors	The equation for this reaction is not required. Both I_2/NaOH and KI/NaOCl are acceptable reagents.

IDENTIFICATION OF ALDEHYDES/KETONES BY THEIR REACTION WITH 2,4-DINITROPHENYLHYDRAZINE

SPECIFICATION REFERENCE: OA 2.2

AIM

To use 2,4-dinitrophenylhydrazine (2,4-DNPH) to identify aldehydes and ketones

APPARATUS AND CHEMICALS

- 5 x Pasteur pipette
- 2 x spatula
- 4 x boiling tube
- test-tube rack
- 100 cm^3 beaker
- 250 cm^3 beaker
- 10 cm^3 measuring cylinder
- 100 cm^3 conical flask
- hot water bath
- Buchner funnel
- suction apparatus
- filter paper
- melting point apparatus (or Thiele tube)
- capillary tube

- 2,4-DNPH
- 1.0 mol dm^{-3} H_2SO_4
- butanone ($CH_3CH_2COCH_3$)
- benzaldehyde (C_6H_5CHO)
- CH_3CH_2OH

SAFETY CONSIDERATIONS

2,4-DNPH	- flammable, toxic
$CH_3CH_2COCH_3$	- flammable, irritant
C_6H_5CHO	- harmful
1.0 mol dm^{-3} H_2SO_4	- irritant
CH_3CH_2OH	- highly flammable



METHOD

1. Add about 1 cm depth of $CH_3CH_2COCH_3$ to a boiling tube.
2. Using a measuring cylinder, carefully add 8 cm^3 of 2,4-DNPH solution to the boiling tube.
3. If a precipitate does not form, carefully add dilute sulfuric acid dropwise until a precipitate is formed.
4. Filter the mixture under reduced pressure to isolate the solid derivative.
5. Purify the derivative by recrystallisation from the minimum amount of hot ethanol.
6. Filter the purified derivative under reduced pressure.
7. Dry a small sample of the derivative by pressing between filter papers.
8. Obtain a melting point for this sample.
9. Repeat steps 1-8 with C_6H_5CHO.

Melting point of $CH_3CH_2COCH_3$ derivative	
Melting point of C_6H_5CHO derivative	

TEACHER/TECHNICIAN NOTES

Apparatus per pupil

- 5 x Pasteur pipette
- 2 x spatula
- 4 x boiling tube
- 1 x test-tube rack
- 1 x 100 cm^3 beaker
- 1 x 250 cm^3 beaker
- 1 x 10 cm^3 measuring cylinder
- 1 x 100 cm^3 conical flask
- 1 x hot water bath
- 1 x Buchner funnel
- 1 x suction apparatus
- 1 x filter paper
- 1 x melting point apparatus (or Thiele tube)
- 1 x capillary tube

Chemicals per pupil

- ~ 16 cm^3 2,4-DNPH
- ~ 1 cm^3 1.0 mol dm^{-3} H_2SO_4
- ~ 5 cm^3 butanone ($CH_3CH_2COCH_3$)
- ~ 5 cm^3 benzaldehyde (C_6H_5CHO)
- ~ 20 cm^3 CH_3CH_2OH

OA2.3 CARBOXYLIC ACIDS AND THEIR DERIVATIVES

Specification Statement		Comment
(a)	order of relative acidity of carboxylic acids, phenols, alcohols and water and how these can be demonstrated	The emphasis is on the pH of aqueous solutions of these compounds and their reactions (if any) with aqueous sodium hydrogencarbonate and sodium hydroxide.
(b)	formation of carboxylic acids by the oxidation of alcohols and aldehydes	
(c)	reduction of carboxylic acids using $LiAlH_4$	Details of the mechanism for this reduction are not required. [H] is acceptable as the formula of the reducing agent in any equations.
(d)	formation of aromatic carboxylic acids by the oxidation of methyl side-chains	Candidates should be aware that longer side chains, e.g. ethyl groups, are similarly oxidised to a —COOH group. Alkaline manganate(VII) is the most effective reagent followed by acidification to produce the aromatic carboxylic acid.
(e)	decarboxylation of carboxylic acids	The usual decarboxylating reagents are NaOH or (preferably) sodalime. Decarboxylation is often carried out using the sodium salt of the acid rather than the acid itself and this is generally more effective.
(f)	conversion of carboxylic acids to esters and acid chlorides and the hydrolysis of these compounds	The role of concentrated sulfuric acid as a catalyst in esterification should be known. The use of gaseous hydrogen chloride as a catalyst results in better yields, as no alkene side products are formed. Sulfur dichloride dioxide (thionyl chloride) is the preferred reagent in making acid chlorides from carboxylic acids as only gaseous co-products are formed. Other acceptable reagents include PCl_3 and PCl_5 but the separation of the acid chloride is more difficult. Candidates should be aware of the relatively rapid hydrolysis of acid chlorides when compared with ester hydrolysis.
(g)	conversion of carboxylic acids to amides and nitriles	An amide is made from a carboxylic acid by conversion to its corresponding ammonium salt which is then heated. The dehydration of an amide by heating with phosphorus(V) oxide (P_4O_{10}) gives the corresponding nitrile.
(h)	formation of nitriles from halogenoalkanes and hydroxynitriles from aldehydes and ketones	Aliphatic nitriles are prepared from halogenoalkanes and potassium cyanide (dissolved in ethanol). For hydroxynitriles see section 4.4(d).
(i)	hydrolysis of nitriles and amides	The hydrolysis of an amide by a dilute acid leads to a carboxylic acid (or its sodium salt if sodium hydroxide is used). The hydrolysis of a nitrile is a similar process to the hydrolysis of an amide.
(j)	reduction of nitriles using	

	$LiAlH_4$	

OA3 ORGANIC COMPOUNDS CONTAINING NITROGEN

OA3.1 AMINES

Specification Statement		Comment
(a)	formation of primary aliphatic amines from halogenoalkanes and nitriles	Aliphatic amines are made by heating halogenoalkanes with an excess of ammonia dissolved in ethanol inside a sealed tube. Candidates should be aware that amine salts are the usual products, e.g. $CH_3CH_2NH_3^+Cl^-$. Treatment of the amine salt with an alkali will release the amine. $LiAlH_4$ reduces nitriles to amines.
(b)	formation of aromatic amines from nitrobenzenes	The preferred reducing agent continues to be metallic tin and concentrated hydrochloric acid, although other valid alternatives are acceptable.
(c)	basicity of amines	Candidates should be aware that the reaction with acids can lead to amine salts, e.g. phenylammonium chloride ($C_6H_5NH_3^+Cl^-$).
(d)	ethanoylation of primary amines using ethanoyl chloride	Questions involving both aliphatic and aromatic primary amines will be set.
(e)	reaction of primary amines (aliphatic and aromatic) with cold nitric(III) acid	Candidates should be aware that at room temperature both aliphatic and aromatic primary amines give alcohols and phenols respectively. Only primary aromatic amines at temperatures below 10 °C give benzenediazonium compounds.
(f)	coupling of benzenediazonium salts with phenols and aromatic amines	Naphthalen-2-ol produces a red azo-dye. An alkaline solution is necessary for coupling to occur. The formulae of these simple azo-dyes should be known.
(g)	role of the —N=N— chromophore in azo dyes	
(h)	origin of colour in terms of the wavelengths of visible light absorbed	

OA3.2 AMINO ACIDS, PEPTIDES AND PROTEINS

Specification Statement		Comment
(a)	general formula and classification of α-amino acids	Only α-amino acids should be considered. Candidates should be aware that all α-amino acids (apart from aminoethanoic acid) contain a chiral centre.
(b)	amphoteric and zwitterionic nature of amino acids and the effect on melting temperature and solubility	Candidates should be able to interchange between the formulae for the zwitterion form and for derivatives in acidic and basic solutions. The nature of the isoelectric point is not required.
(c)	combination of α-amino acids to form dipeptides	Dipeptides can be produced from two molecules of the same amino acid as well as from one molecule of each of two different amino acids.
(d)	formation of polypeptides and proteins	The combination of more than two amino acid molecules gives rise to polypeptides which can then lead to protein formation.
(e)	basic principles of primary, secondary and tertiary protein structure	Only an outline of protein structure is needed. No questions will be set that require a detailed understanding of this topic. Candidates should be aware that the primary structure is the sequence of amino acids making up the protein chain. The secondary structure is concerned with how parts of the protein can fold up to form an α-helix that is held in place by hydrogen bonds or a β-pleated sheet in which the amino acids form a shape like a piece of paper stabilised by hydrogen bonds between amino acids in different polypeptide chains. The tertiary structure refers to the protein as a whole and is the way in which the α-coils or β-pleated sheets of the protein fold with respect to each other.
(f)	essential role of proteins in living systems, for example, as enzymes	Protein is an essential component of a healthy diet. The functions of proteins are many. They are necessary for structural functions in living organisms, e.g. in nails, feathers, skin and the collagen of cartilage. Proteins are enzymes – biological catalysts. Some proteins act as hormones, e.g. insulin. Candidates should recall a minimum of two examples of proteins and their importance.

OA4 ORGANIC SYNTHESIS AND ANALYSIS

Specification Statement		Comment
(a)	synthesis of organic compounds by a sequence of reactions	The sequence of reactions for organic synthesis will generally be limited to a maximum of three or four stages.
(b)	principles underlying the techniques of manipulation, separation and purification used in organic chemistry	Separation and purification procedures that are expected include distillation (including steam distillation), the use of a separating funnel (including simple solvent extraction), filtration, crystallisation and chromatographic processes.
(c)	distinction between condensation polymerisation and addition polymerisation	
(d)	how polyesters and polyamides are formed	Candidates should recall details of the synthesis of PET, Nylon-6 and Nylon-66.
(e)	use of melting temperature as a determination of purity	
(f)	use of high resolution ^{1}H NMR spectra (alongside the other spectral data specified in C3.5) in the elucidation of structure of organic molecules	Candidates will not be required to interpret high resolution ^{1}H NMR spectra of compounds where splitting occurs as a result of protons on more than one adjacent carbon atom, e.g. $CH_3CH_2CH_2Cl$.
(g)	use of chromatographic data from TLC/paper chromatography, GC and HPLC to find the composition of mixtures	Questions will be set that involve both qualitative and quantitative data. The theory of chromatography and technical details of instruments are not required.

SYNTHESIS OF A LIQUID ORGANIC PRODUCT

SPECIFICATION REFERENCE: OA 4

AIM

To synthesise 2-chloro-2-methylpropane using 2-methylpropan-2-ol and concentrated hydrochloric acid and purify the product using a separating funnel followed by distillation

APPARATUS AND CHEMICALS

- access to deionised water
- access to 2 decimal place mass balance

- 100 cm^3 round bottom flask
- 100 cm^3 conical flask
- 100 cm^3 conical flask with stopper
- 10 cm^3 measuring cylinder
- 50 cm^3 measuring cylinder
- 50 cm^3 separating funnel
- ice bath
- reflux condenser
- thermometer
- sample vial
- labels/suitable pen
- anti-bumping granules
- heating mantle / Bunsen burner with water bath
- clamp stand
- filter funnel with cotton wool

- $(CH_3)_3COH$
- concentrated HCl solution
- granular anhydrous $CaCl_2$
- saturated $NaHCO_3$ solution
- anhydrous $MgSO_4$ powder

SAFETY CONSIDERATIONS

$(CH_3)_3COH$	- flammable
concentrated HCl solution	- corrosive
$CaCl_2$	- irritant



METHOD

1. Add 30 cm^3 of concentrated HCl solution to a stoppered conical flask. This step should be performed in a fume cupboard.
2. Cool the HCl solution in the ice bath.
3. When the HCl solution has cooled add 2.5 g anhydrous $CaCl_2$ to the solution.
4. Add 10 cm^3 of $(CH_3)_3COH$ to the reaction mixture and mix thoroughly.
5. Remove the mixture from the ice bath and allow it to slowly warm up to room temperature. Note that a gaseous side product can be formed at this stage and so the stopper should be released periodically to alleviate any build-up of pressure.
6. Transfer the reaction mixture to the separating funnel.
7. Stopper the funnel and invert it a few times to thoroughly mix the reaction mixture. You may need to alleviate the pressure in the funnel by holding it upside down and opening the tap.
8. Run off and discard the aqueous layer. If you are unsure which layer is the aqueous layer, add deionised water to funnel and see which layer increases in volume.
9. Add 10 cm^3 of saturated $NaHCO_3$ solution to the organic layer and mix thoroughly in the separating funnel. CO_2 gas is formed in this step so it is important to alleviate the pressure in the funnel periodically.
10. Run off and discard the aqueous layer.
11. Repeat steps 9 and 10 to wash the product for the second time.
12. Repeat steps 9 and 10 using 15 cm^3 of deionised water to wash the product for a third time.
13. Transfer the organic product to a conical flask, add anhydrous $MgSO_4$ and swirl vigorously. Keep adding $MgSO_4$ until it no longer clumps together.
14. Transfer the organic product to the round bottomed flask through a filter funnel with a plug of cotton wool to remove the $MgSO_4$.
15. Set up the distillation apparatus.
16. Distil off the $(CH_3)_3CCl$ produced and collect in a clean, dry, pre-weighed 100 cm^3 conical flask.
17. Record the temperature at which the liquid product is collected.
18. Calculate the mass of the product and use this information to calculate the product yield (Note that the density of $(CH_3)_3COH$ is 0.775 g cm^{-3})

Boiling point of 2-chloro-2-methylpropane	

TEACHER/TECHNICIAN NOTES

Apparatus per laboratory

- 2 decimal place mass balance
- labels
- anti-bumping granules

Chemicals per laboratory

- deionised water
- anhydrous $MgSO_4$ powder

Apparatus per student

- 1 x 100 cm^3 round bottom flask
- 1 x 100 cm^3 conical flask
- 1 x 100 cm^3 conical flask with stopper
- 1 x 10 cm^3 measuring cylinder
- 1 x 50 cm^3 measuring cylinder
- 1 x 50 cm^3 separating funnel
- 1 x ice bath
- 1 x reflux condenser
- 1 x thermometer
- 1 x sample vial
- 1 x heating mantle / Bunsen burner with water bath
- 1 x clamp stand
- 1 x filter funnel with cotton wool

Chemicals per student

- ~ 10 cm^3 $(CH_3)_3COH$
- ~ 30 cm^3 concentrated HCl solution
- ~ 2.5 g granular anhydrous $CaCl_2$
- ~ 10 cm^3 saturated $NaHCO_3$ solution

SYNTHESIS OF A SOLID ORGANIC PRODUCT

SPECIFICATION REFERENCE: OA 4

AIM

To synthesise 2-acetoxybenzenecarboxylic acid (aspirin) from 2-hydroxybenzenecarboxylic acid and ethanoic anhydride, purify the product using recrystallization and determine its melting point

APPARATUS AND CHEMICALS

- access to deionised water
- access to 3 decimal place mass balance (minimum 2 decimal place)

- 25 cm^3 pear shaped flask
- 10 cm^3 measuring cylinder
- hot water bath
- ice bath
- glass stirring rod
- Buchner funnel
- suction apparatus
- watch glass
- sample vial
- labels/suitable pen
- filter paper
- melting point apparatus / Thiele tube
- capillary tube

- 2-hydroxybenzenecarboxylic acid
- $(CH_3CO)_2O$
- concentrated H_3PO_4 solution

SAFETY CONSIDERATIONS

2-hydroxybenzenecarboxylic acid	- harmful
$(CH_3CO)_2O$	- flammable, corrosive
concentrated H_3PO_4 solution	- corrosive



METHOD

1. Weigh out 1.0 g of 2-hydroxybenzenecarboxylic acid and transfer to a pear shaped flask.

2. Add 2 cm^3 of $(CH_3CO)_2O$ and 8 drops of concentrated H_3PO_4 solution.

3. Connect the reflux condenser and place in a fume hood.

4. Warm the mixture in a hot water bath until all of the solid dissolves then warm for a further 5 minutes.

5. Carefully add 5 cm^3 of cold deionised water to the solution and stand the flask in a bath of iced water until precipitation appears to be complete. It may be necessary to stir vigorously with a glass rod to start the precipitation process.

6. Filter the mixture under reduced pressure to obtain the impure derivative and wash with a little cold water.

7. Using a boiling tube in a water bath, dissolve the impure product in the minimum amount of warm ethanol.

8. Add 5 cm^3 of warm water drop-wise. If the solution becomes cloudy, heat until it becomes clear again.

9. Place the boiling tube into ice water for 15 minutes or until the crystals stop forming.

10. Filter the purified derivative under reduced pressure.

11. Dry the purified product using filter paper.

12. Collect your sample in a dry, pre-weighed sample vial and calculate the mass of the product. Use this information to calculate the percentage yield of the product.

13. Measure the melting point of the product.

Melting point of 2-acetoxybenzenecarboxylic acid	

TEACHER/TECHNICIAN NOTES

Apparatus per laboratory

- 3 decimal place mass balance (minimum 2 decimal place)
- labels

Chemicals per laboratory

- deionised water

Apparatus per student

- 1 x 25 cm^3 pear shaped flask
- 1 x 10 cm^3 measuring cylinder
- 1 x hot water bath
- 1 x ice bath
- 1 x glass stirring rod
- 1 x Buchner funnel
- 1 x suction apparatus
- 1 x watch glass
- 1 x sample vial
- 1 x filter paper
- 1 x melting point apparatus / Thiele tube
- 1 x capillary tube

Chemical per student

- ~ 1g 2-hydroxybenzenecarboxylic acid
- ~ 2 cm^3 $(CH_3CO)_2O$
- ~ 1 cm^3 concentrated H_3PO_4 solution

TWO-STEP SYNTHESIS

SPECIFICATION REFERENCE: OA 4

AIM

To synthesise 3-nitrobenzenecarboxylic acid via nitration of methyl benzenecarboxylate followed by alkaline hydrolysis of the ester functional group

APPARATUS AND CHEMICALS

- access to cold deionised water
- access to a 3 decimal place mass balance (minimum 2 decimal place)
- access to ice

- spatula
- weighing boat
- 2 x 100 cm^3 conical flask
- 25 cm^3 measuring cylinder
- ice bath
- glass Pasteur pipette with rubber teat
- glass stirring rod
- 250 cm^3 beaker
- Buchner funnel
- suction apparatus
- filter paper
- 10 cm^3 measuring cylinder
- hot water bath
- round bottomed flask
- reflux condenser
- thermometer
- sample vial
- labels/suitable pen
- anti-bumping granules
- heating mantle / Bunsen burner with water bath
- clamp stand
- melting point apparatus / Thiele tube
- capillary tube

- concentrated H_2SO_4 solution
- $C_6H_5COOCH_3$
- concentrated HNO_3 solution
- CH_3OH
- NaOH
- concentrated HCl solution
- 0.1 mol dm^{-3} HCl solution

SAFETY CONSIDERATIONS

concentrated H_2SO_4 solution	- corrosive
$C_6H_5COOCH_3$	- flammable
concentrated HNO_3 solution	- corrosive
CH_3OH	- flammable, toxic
NaOH	- corrosive
concentrated HCl solution	- corrosive
0.1 mol dm^{-3} HCl solution	- irritant



METHOD PART 1

1. Carefully add 20 cm^3 of concentrated H_2SO_4 solution to a conical flask.

2. Add 10.2 g of $C_6H_5COOCH_3$ to the conical flask.

3. Cool the reaction mixture to 0 °C in an ice bath.

4. Slowly add, with stirring, 12.5 cm^3 of a 1:1 mixture of concentrated H_2SO_4 solution and concentrated HNO_3 solution. Monitor the temperature of the reaction with a thermometer as you perform this step and ensure that the temperature does not rise above 10 °C.

5. Pour the reaction mixture slowly onto ice in a beaker.

6. When the ice has melted, filter the mixture under reduced pressure.

7. Wash the solid once with cold deionised water and twice with cold CH_3OH.

8. Recrystallise the solid product from hot methanol using a hot water bath.

9. Dry the recrystallized product, record its mass and measure its melting point.

10. Use the mass of the product to calculate the percentage yield.

METHOD PART 2

1. To a round bottomed flask add 9.0 g of your product from part 1, 20 cm^3 of deionised water and 4.0 g of NaOH.

2. Attach a condenser to the round bottomed flask and heat the reaction mixture under reflux for 5-10 minutes.

3. Carefully pour the reaction mixture, with stirring, into 12.5 cm^3 of concentrated HCl solution. This step should be performed in a fume cupboard.

4. Cool the reaction mixture to room temperature using an ice bath.

5. Filter the mixture under reduced pressure and wash with cold deionised water.

6. Recrystallise the solid from hot 0.1 mol dm^{-3} HCl solution using a hot water bath.

7. Dry the recrystallized product, record its mass and measure its melting point.

8. Use the mass of the product to calculate the percentage yield.

Melting point of 3-nitrobenzenecarboxylate	
Melting point of 3-nitrobenzenecarboxylic acid	

TEACHER/TECHNICIAN NOTES

Apparatus per laboratory

- access to a 3 decimal place mass balance (minimum 2 decimal place)
- labels
- anti-bumping granules
- fume cupboard

Chemicals per laboratory

- deionised water
- ice

Apparatus per student

- 1 x spatula
- 1 x weighing boat
- 2 x 100 cm^3 conical flask
- 1 x 25 cm^3 measuring cylinder
- 1 x ice bath
- 1 x glass Pasteur pipette with rubber teat
- 1 x glass stirring rod
- 1 x 250 cm^3 beaker
- 1 x Buchner funnel
- 1 x suction apparatus
- 1 x filter paper
- 1 x 10 cm^3 measuring cylinder
- 1 x hot water bath
- 1 x round bottomed flask
- 1 x reflux condenser
- 1 x thermometer
- 1 x sample vial
- 1 x heating mantle / Bunsen burner with water bath
- 1 x clamp stand
- 1 x melting point apparatus / Thiele tube
- 1 x capillary tube

Chemicals per student

- ~ 26.5 cm^3 concentrated H_2SO_4 solution
- ~ 10.2g $C_6H_5COOCH_3$
- ~ 6.5 cm^3 concentrated HNO_3 solution
- ~ 10 cm^3 CH_3OH
- ~ 4g NaOH
- ~ 12.5 cm^3 concentrated HCl solution
- ~ 10 cm^3 0.1 mol dm^{-3} HCl solution

PLANNING A SEQUENCE OF TESTS TO IDENTIFY ORGANIC COMPOUNDS

SPECIFICATION REFERENCE: OA 4

AIM

Plan and then carrying out a sequence of tests to identify organic compounds from a given list

APPARATUS AND CHEMICALS

- access to deionised water
- access to disposable gloves (or suitable long-term use gloves such as nitriles)

- test tubes with stoppers
- boiling tubes
- small beakers
- test tube racks
- red and blue litmus or similar indicator papers

- 2 mol dm^{-3} NaOH solution
- 1 mol dm^{-3} H_2SO_4 solution
- $NaHCO_3$
- 2,4-DNPH
- materials for preparing Tollens' reagent
- materials for preparing the reagent for the iodoform test
- $NaNO_2$

SAFETY CONSIDERATIONS

2 mol dm^{-3} NaOH solution	- irritant
1 mol dm^{-3} H_2SO_4 solution	- irritant
2,4-DNPH	- flammable, toxic
0.1 mol dm^{-3} NH_3 solution	- irritant
0.1 mol dm^{-3} $AgNO_3$ solution	- harmful
$NaNO_2$	- oxidising, toxic, dangerous to the environment
$CH_3CH_2CH_2CH_2NH_2$	- flammable, harmful
C_6H_5CHO	- flammable, harmful
$CH_3COCH_2CH_3$	- flammable, irritant
$C_6H_5COC_6H_5$	- irritant
$C_6H_5COOC_2H_5$	- irritant
CH_3CONH_2	- carcinogenic
C_6H_5CN	- harmful, irritant



GENERAL METHOD

1. You are provided with a sample of 6 unknown organic chemicals. These chemicals may be any six from the following eight chemicals.

1-aminobutane	$CH_3CH_2CH_2CH_2NH_2$
benzenecarbaldehyde	C_6H_5CHO
benzenecarboxylic acid	C_6H_5COOH
diphenylmethanone	$C_6H_5COC_6H_5$
butanone	$CH_3COCH_2CH_3$
ethanamide	CH_3CONH_2
ethylbenzenecarboxylate	$C_6H_5COOC_2H_5$
benzenecarbonitrile	C_6H_5CN

2. You are also provided with the apparatus and chemicals listed on the previous page.

PLANNING

1. Complete a written method detailing how you will identify these chemicals in the fewest possible steps. This method should include a diagram showing the sequence of tests and the outcomes for each sequence.

2. Complete a full risk assessment for this investigation.

TEACHER/TECHNICIAN NOTES

Apparatus per laboratory

- disposable gloves (or suitable long-term use gloves such as nitriles)

Chemicals per laboratory

- deionised water

Apparatus per student

- test tubes with stoppers
- boiling tubes
- small beakers
- test tube racks
- red and blue litmus or similar indicator papers

Specific numbers will vary depending on the method used by the students.

Chemicals per student

- 2 mol dm^{-3} NaOH solution
- 1 mol dm^{-3} H_2SO_4 solution
- $NaHCO_3$
- 2,4-DNPH
- 0.5 mol dm^{-3} $AgNO^3$ solution
- 1 mol dm^{-3} NaOH solution
- 0.5 mol dm^{-3} NH_3 solution
- I_2 solution
- $NaNO_2$
- $CH_3CH_2CH_2CH_2NH_2$
- C_6H_5CHO
- C_6H_5COOH
- $C_6H_5COC_6H_5$
- $CH_3COCH_2CH_3$
- CH_3CONH_2
- $C_6H_5COOC_2H_5$
- C_6H_5CN

Specific volumes will vary depending on the method used by the students.

PAPER CHROMATOGRAPHY

SPECIFICATION REFERENCE: OA 4

AIM

To analyse biro inks using paper (or thin layer) chromatography

APPARATUS AND CHEMICALS – PAPER CHROMATOGRAPHY

- Whatman Grade 1 chromatography paper
- scissors
- green, black, blue and red biro inks (or equivalent)
- 250 cm^3 bottle or conical flask with tight fitting bung
- Pasteur pipette

- developing solvent (20 cm^3 of water, 20 cm^3 of ethanol and 60 cm^3 butan-1-ol)

SAFETY CONSIDERATIONS – PAPER CHROMATOGRAPHY

developing solvent - flammable



METHOD – PAPER CHROMATOGRAPHY

1. Cut a piece of chromatography paper to fit the bottle or conical flask and draw a straight pencil line approximately 10-15 mm from the bottom edge of the paper.

2. Make sure the biro is working and then make a small dot (no more than 1-2 mm in diameter) on the paper on the pencil line. If more than one biro dot (e.g. different colours) is placed on the line, ensure that there is at least a 5 mm gap between the dots along the line. Do not put any dots close to the side edges of the chromatography paper.

3. Add the developing solvent to the bottle to a depth of no more than 10 mm. Use a fume cupboard when using the developing solution.

4. Carefully insert the paper into the solvent. Make sure that the pencil line with the biro dots stays above the solvent level. Hold the chromatography paper in place with a cork or bung.

5. Leave the flask/bottle where it will not be moved for about 30 minutes. Do not allow the solvent front to reach the bottom of the bung.

6. Remove the paper, mark the position of the solvent front and leave it to dry near an open window or in a fume cupboard.

7. The dry chromatograms can be stuck into the lab book.

Note: thin layer chromatography may be used in place of paper chromatography.

TEACHER/TECHNICIAN NOTES

Apparatus per student

- Whatman Grade 1 chromatography paper (or equivalent)
- scissors
- green, black, blue and red biro inks (or equivalent)
- 250 cm^3 bottle or conical flask with tight fitting bung
- Pasteur pipette

Chemistry per student

- ~ 50 cm^3 developing solvent

Notes

100 cm^3 of developing solvent is made by mixing 20 cm^3 of water, 20 cm^3 of ethanol and 60 cm^3 of butan-1-ol. Scale up the amounts as required.