

A user guide

**Edexcel Advanced Subsidiary GCE in
Chemistry (8080)**

Edexcel Advanced GCE in Chemistry (9080)

Externally assessed practical test

Unit tests 3A and 6A

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Introduction

This booklet has been written to help candidates preparing for, and taking, the externally assessed practical tests as part of their Advanced Subsidiary and Advanced GCE Chemistry qualification. Teachers may also find the information contained in this user guide helpful when preparing candidates. The material contained in the booklet does not extend the specification content, rather it seeks to help the candidate to succeed in the practical tests by:

- explaining in more depth what is required in carrying out the exercises, making observations and measurements with appropriate precision and recording these methodically.
- advising how to interpret, explain, evaluate and communicate the results of the exercises clearly and logically using the relevant chemical knowledge and understanding and using appropriate specialist vocabulary.

1 The Practical Test

1.1 Availability and summary of assessment

Practical tests for Advanced Subsidiary GCE (unit test 3A) and Advanced GCE (unit test 6A) are available as follows:

Unit Test	Paper reference	January session	May/June session	Duration	Marks	AS	Advanced
3A	6243/01A – Gp 1	✓	✓	1h 45 mins	50	20%	10%
	6243/01B – Gp 2	✗	✓	1h 45 mins	50	20%	10%
	6243/01C – Gp 3 ¹	✗	✓	1h 45 mins	50	20%	10%
6A	6246/01A – Gp 1	✗ ²	✓	1h 45 mins	50		10%
	6246/01B – Gp 2	✗ ²	✓	1h 45 mins	50		10%
	6246/01C – Gp 3 ¹	✗ ²	✓	1h 45 mins	50		10%

Notes: ¹ Available to **overseas** centres only

² Unit Test 6A will not be examined in the January session with effect from January 2005

In the May/June session, the three practical tests are offered as separate examinations to allow centres the maximum freedom of entry for their students. Group 1 and 2 practical tests must be taken on the dates specified on the main examination timetable. The Group 3 practical test is available to overseas centres ONLY. The date of the Group 3 practical test must be agreed in advance with Edexcel International Examination Services.

When making the entries for Units 3 and 6, centres must specify the correct entry option code for the practical test. Details of these entry codes are published in Edexcel's Information Manual.

1.2 Summary of assessment

Candidates should attempt all questions on the paper, writing their answers in the spaces provided in the question paper. Candidates may use any notes or textbooks during the assessment of practical skills, except those that give details of actual experiments set as planning exercises.

The practical test for unit test 3A is of a broadly quantitative nature and draws on the knowledge, understanding and skills developed during the first half of an advanced course of study. The practical test for unit test 6A is of a broadly qualitative nature and is based on the knowledge, understanding and skills taken from A2.

Assessment is made in relation to the following skills:

- Planning
- Manipulation
- Observation and recording
- Interpretation and evaluation

1.3 The content of the practical tests

Both the Advanced Subsidiary and Advanced GCE practical tests will include exercises on the ions, compounds and techniques listed in the specification for unit 3A and unit 6A. Candidates and teachers should refer to these sections of the specifications for a summary of content. Candidates should note that exercises will not be set which demand a knowledge of substances which are not part of the specification content. If the practical test makes use of a substance which candidates are not expected to be familiar with, no knowledge of that substance will be expected.

For example, if the addition of aqueous sodium hydroxide to a solution containing a d-block cation produces a white precipitate then the candidate would be expected to infer that the cation is zinc(II). The white precipitate would not be cadmium(II) hydroxide since the reaction between aqueous Cd^{2+} and aqueous NaOH is not included in the specification content.

1.4 Directions for the preparation of the practical test

Confidential Supervisor's Instructions for the preparation and conduct of the practical test are sent to centres before each examination session. This secure pack will contain one copy of the Supervisor's Instructions, one copy of the Question paper(s) and one copy of the Questionnaire (overseas centres only). The Supervisor's Instructions may be referred to at any time by the teacher responsible for the practical test, but must be kept under lock and key at all times when they are not in use. Under no circumstances may they be removed from the school/college premises or be left in any place where candidates may have access to them.

The Supervisor's Instructions contain a full list of the apparatus and chemicals required for the practical test. This list includes the bench reagents needed and the approximate volumes. However, further quantities can be issued to candidates without penalty. It is not intended that each candidate is supplied with his or her own set of reagents, but that these are shared at the discretion of the supervisor.

A set of chemicals is sent to HOME centres (only), making an entry for the practical test, by a firm of manufacturing chemists. On receipt of the chemicals, centres should first check that the carton is for the correct examination **before** opening it to check the contents.

The prompt despatch of the Supervisor's Instructions (and the chemicals) is dependent on the centre making its entry by the published deadline. Edexcel cannot guarantee to provide chemicals where very late entries or late amendments to entries are made.

Strict security concerning details of the practical test must be maintained. Any queries relating to the Supervisor's Instructions should be made directly to the Assessment Leader for GCE Chemistry. Telephone contact details are provided on the front cover of the Supervisor's Instructions or centres can email Chemistry.PracticalExam@edexcel.org.uk.

1.5 Directions for the conduct of the practical test

There are strict regulations concerning the conduct of all examinations including the practical tests.

Instructions and regulations are drawn up by the Joint Council for General Qualifications and are posted on its website (www.jcgq.org.uk). Two documents of particular importance are:

- *Instructions for the Conduct of Examinations*
- *Regulations and Guidance Relating to Candidates with Particular Requirements*

A Centre may find that it has a large number of candidates entered for the practical test and hence more than one session (of the same practical test) needs to be run on the day of the exam. If this situation occurs, the Centre must contact Edexcel's Special Requirements section in advance of the examination to receive approval for such a timetable variation.

On the day of the examination, candidates should be allowed 10 minutes to familiarise themselves with the layout of the laboratory and the materials available. They may NOT begin the practical work during this time nor read through the question paper. A plan showing the arrangement of candidates in the laboratory during the practical test must be submitted to the examiner, together with the candidates' scripts and the attendance register. In addition OVERSEAS centres MUST complete and submit the Questionnaire.

1.6 Health and safety

Candidates must follow the health and safety policy which normally operates in their centre laboratories when carrying out the practical test. This may include:

- eye protection must always be worn
- laboratory overalls are recommended
- all substances should be regarded as being potentially toxic and hazardous
- HazChem labels (eg flammable) should be read and appropriate precautions (eg keep liquid away from flame) taken
- all substances spilled on the skin should be rinsed off immediately
- chemicals must never be tasted
- gases and vapours should never be smelt unless the question instructs the candidates to do so and then only with great care.

2. Quantitative Exercises

A quantitative exercise will form a **major** part of the Advanced Subsidiary practical test 3A. A **short** quantitative exercise could be included in the Advanced GCE practical test 6A, in addition to the main qualitative exercise.

Quantitative exercises could involve:

- a volumetric analysis exercise
- a simple thermochemistry exercise
- an exercise to follow the rate of a reaction
- an exercise to find melting points or boiling points (unit test 6A only)

Further details can be found in the reference section.

2.1 Accuracy

Unless a question instructs candidates differently they should assume that readings from equipment and apparatus should be made with the following precision:

Apparatus	Precision of reading
pipette	one volume only of 25.0 cm ³
burette	each volume to the nearest 0.05 cm ³ . Mean titre to 0.05 cm ³ or to the second decimal place
measuring cylinder	a 100 cm ³ size to 5 cm ³ , a 10 cm ³ size to 1 cm ³
balance	readings should be made to 0.01 g or 0.001 g depending on the precision of the balance
timers	normally read to the nearest second.
thermometers	0 to 100°C thermometers should be read to the nearest 0.5°C and 0 to 50°C thermometers to the nearest 0.2°C

It is important that:

- when candidates record readings they include the appropriate number of decimal places.
eg a burette reading of exactly 24.7 cm³ should be recorded in a results table as 24.70 cm³.
A temperature reading of exactly 35°C should be recorded as 35.0°C.
- when titres have to be averaged, the mean is expressed to either the nearest 0.05 cm³ or to the second decimal point.
eg if a candidate records four titres as listed below the mean should be calculated as shown.

$$26.50 \text{ cm}^3 \quad 26.25 \text{ cm}^3 \quad 26.60 \text{ cm}^3 \quad 26.65 \text{ cm}^3$$

the candidate decides to ignore the second titre and to average the remaining three.

$$\frac{26.50 + 26.60 + 26.65}{3} = 26.583 \text{ which should be recorded as } 26.60 \text{ cm}^3 \text{ or as } 26.58 \text{ (to the second decimal place)}$$

- candidates should try to obtain **at least two** titres within 0.20 cm^3 , or better, of each other and average these to obtain a mean titre. Candidates should make clear which titres have been used to obtain a mean.
- a final result should only be given to the same number of significant figures as is suggested in the exercise.
- eg an exercise to find a ΔH value may involve a weighing of 1.56 g and a temperature rise of 7.5°C . A candidate who calculates a value of ΔH from these figures may obtain a value of 195.6843 on a calculator, but this should be finally recorded as 196 kJ mol^{-1} although $195.7 \text{ kJ mol}^{-1}$ would also be acceptable.
- units should always be included with a quantitative result.

A significant proportion of the marks for a quantitative exercise will be awarded for **accuracy**. These marks will be awarded by comparing the candidates results with either the examiner's (home centres) or the supervisor's (overseas centres) results.

2.2 Errors

Candidates should appreciate that any piece of equipment (burette, pipette, thermometer, balance) in a quantitative exercise has an uncertainty associated with its use. Even if the equipment is used carefully, the uncertainty leads to an error in the reading and in the final result.

eg A balance has an uncertainty of 0.01 g when read to the second decimal place. A reading of 2.64 g recorded in an experiment has an error of:

$$\frac{0.01}{2.64} \times 100\% = 0.38\%.$$

The following points apply:

1. the error in a reading = $\frac{\text{the uncertainty in the equipment}}{\text{the reading}} \times 100\%$
2. candidates should:
 - be able to calculate the error involved in using a particular piece of equipment and state what effect this has on the overall accuracy of the experiment.
 - understand that the percentage error would be affected by the magnitude of the quantity being measured. There will be a greater error in weighing a mass of 2.64 g than in weighing 8.64 g using the same balance.
- 3 no more than a simple treatment of errors is needed. Candidates will not be asked to combine errors.

2.3 Calculations

Usually calculations will be structured. Candidates will be taken/guided through a series of steps leading to a final value.

Since most of the marks for these steps will be for a correct method rather than for the numerical answer it is important that candidates include their working even if this seems to be trivial. No marks can be awarded for an incorrect answer without working but a correct method followed by an incorrect answer can often receive credit.

Units, if appropriate, should always be included with a quantitative result.

2.4 Graphs

Some exercises in the practical test will require candidates to treat their readings graphically. A grid will be printed in the examination booklet but axes will **not usually** be drawn. The question will, however, instruct the candidate which axis to use for each quantity being plotted.

The following are some useful points for candidates when constructing graphs:

- put the dependent variable, the quantity being measured (eg temperature) on the y-axis. And the pre-determined quantity (eg volume of solution) on the x-axis
- choose the scales so that the results are spread out as far apart as the size of the grid allows but this should not be at the expense of using a sensible scale.
eg use 1 cm on the axis to represent 3 or 4 units might spread the readings better than using 1 cm to represent 5 units but the scale would be hard to read
- the origin (0,0) need not necessarily be included on either scale if it is not relevant.
eg if temperature readings between 21.0°C and 39.0°C are to be plotted there is no need to begin the y-axis at 0. Rather it could be scaled from 20.0°C to 40.0°C
- clearly label the axes with the quantity being plotted (eg time) and its units (eg minutes)
- join the points plotted with a straight line or a smooth curve. Since the readings are all subject to experimental error the line drawn may not necessarily pass through every point. Points should never be joined by a series of short straight lines.

2.5 Assumed laboratory skills

The instructions for carrying out a quantitative exercise will include the essential points for the particular exercise. For example it will tell the candidate:

- which solutions to use
- the quantities needed
- the sequence of steps in the method
- the readings to be taken.

The practical test will assume that the candidate has developed a range of routine practical skills in the course leading up to the test. Instructions will not, therefore, include every step needed by the candidate to gain accurate readings.

Examples of normal laboratory procedures which may **not** be referred to in the instructions include the following:

- burettes, pipettes and measuring cylinders should be rinsed with the solution they are to contain
- a safety bulb filler should always be used with a pipette
- conical flasks and volumetric flasks should be rinsed out with distilled water
- a burette should be read at eye-level
- a thermometer bulb must be held in the centre of a solution when temperature readings are taken
- care should be taken not to lose drops of a solution when thermometers or stirring rods are removed from it
- containers may need to be labelled if this has not already been done for the candidate

- care should be taken as to where apparatus is placed on the bench. Temperature measurements should not be taken in an apparatus standing next to a Bunsen burner, in a patch of sunlight or in a strong draught.

3. Qualitative Exercises

The Advanced GCE practical test will include exercises which require the candidate to identify an unknown substance or mixture of substances and the Advanced Subsidiary practical test could include some simple qualitative tests in addition to the main quantitative exercise.

If the unknown is an inorganic compound or mixture of compounds then the exercise will usually be to identify the ions present. Candidates may be asked to identify an element. The unknown may be an organic compound or mixture of compounds. Candidates should understand that a single organic compound may contain two functional groups.

Candidates will be instructed to carry out a series of tests, usually on a test tube scale. The instructions for each test will clearly include details on quantities to be used, whether heat is required and whether excess reagent should be added. The reagents used for the tests will be limited to those included in the specification. Similarly, unknowns will be limited to compounds containing the ions, elements and organic compounds with the functional groups listed in the specification.

As part of an exercise to identify an organic unknown, spectroscopic data may be included for analysis by the candidate. Other physical data such as melting points may be given.

3.1 Observations

After a candidate has carried out each test as instructed in the paper, they must communicate the results of the test by giving a brief description of what has been observed. Possible changes occurring during tests are listed below along with **examples** of what the candidate should write in the Observation column on the paper.

Possible change	Example of observation
a colour change in solution	yellow solution turns orange
the formation of a precipitate	white precipitate (ppt is allowed) is formed
a precipitate dissolves in excess reagent	precipitate dissolves in excess to form a green solution
a gas is evolved	bubbles of gas or effervescence
tests on a gas	the gas turned damp red litmus paper blue
flame tests	yellow flame
a solid dissolves	dissolves to give a blue solution
a reaction is exothermic	the mixture becomes hot
a reaction is endothermic	the mixture feels cold
a coloured solution loses its colour	the yellow solution turns colourless

There are a number of common mistakes which candidates make when recording their observations. Candidates should avoid the following:

- referring to 'layers' in test tubes. Almost certainly the presence of layers is due to inadequate mixing of the reagents
- describing colours with elaborate adjectives such as brown–black or blue–green. The examiners will always award marks to candidates who describe the colours of solutions and precipitates with simple words such as black or blue even if the colour does contain a trace of a second colour
- stating that a gas is evolved without making an observation such as bubbles or effervescence

3.2 Inferences

The purpose of asking candidates to complete the Inference column is to test knowledge, understanding and evaluation of the chemistry which leads to the observations. Candidates can consult books, notes as well as this booklet when making their inferences. Caution is required when using books since they will include substances which are not in the specification.

Although the examiners could ask candidates to carry out tests and make observations on substances which are not in the specification they would **not** expect them to identify the substances.

A candidate should normally attempt to identify any precipitate or gas formed in a test and which is recorded as an observation. In some cases the test may be enough to enable the candidate to suggest the identity of a particular ion or functional group but, in others a number of possibilities may exist as a result of a single test.

Example 1

The addition of aqueous ammonia to an inorganic compound produces a green precipitate.

The inference should be iron(II) hydroxide, chromium(III) hydroxide or nickel(II) hydroxide. Further tests would be set to identify the unknown.

Example 2

In an organic analysis, a compound produces an orange precipitate with 2,4–dinitrophenylhydrazine.

The expected inference would be that the unknown compound is an aldehyde or a ketone. If a following test shows that the compound is an aldehyde then the inference still stands since this was valid on the basis of the 2,4–dinitrophenylhydrazine test. If more is known about the compound, for example its molecular formula and the fact that it cannot be oxidised, then it may be that the compound can be exactly identified as a result of the 2,4–dinitrophenylhydrazine test.

4. Planning Exercises

In both practical test unit 3A and 6A, candidates will be asked to design an experiment similar to one which they have carried out on their chemistry course. Possibilities for planning exercises are many and varied and could be to investigate an enthalpy change or to identify organic compounds or to carry out an investigation based on a titration.

Outline guidance when answering **some** of the possibilities are given below:

Planning exercise	Outline guidance
Identify the ions in an unknown compound	<ul style="list-style-type: none">describe tests (eg flame test, AgNO_3, BaCl_2 etc) and likely results in a logical sequence.
Identify an organic compound	<ul style="list-style-type: none">describe tests eg PCl_5, $\text{K}_2\text{Cr}_2\text{O}_7/\text{H}_2\text{SO}_4$ for alcohols and likely results in a logical sequence
Plan a procedure to purify a compound	<ul style="list-style-type: none">describe techniques from organic chemistry: distillation, recrystallisation; fractional distillation
Find an enthalpy change	<ul style="list-style-type: none">measure quantities of reagentsdescribe temperature measurements as reaction takes place in plastic cupoutline method of calculation
Carry out titration or make up solution to be titrated	<ul style="list-style-type: none">describe steps in titration including end-point.weigh, dissolve and make up solution in volumetric flask
Make use of the stoichiometry of a reaction to find relative molecular mass	<ul style="list-style-type: none">carry out reaction with measured quantities collecting product eg gas in gas syringeconvert quantities to moles

Reference Section

A: Inorganic compounds and elements

1 Appearance

- Coloured crystalline substances are usually hydrated salts of d-block elements. The colour of the solid substance may provide evidence for its identity.

Colour	Possible ions in salt
blue	copper (II)
pale green	iron (II)
green	copper (II), chromium (III), nickel (II)
purple	manganate (VII), chromium (III)
violet	iron (III)
brown	iron (III)
deep pink/red	cobalt (II)
pink	manganese (II)
yellow	chromate (VI)
orange	dichromate (VI)

- Other solids which may be encountered in the qualitative tests include those listed in the table below.

colour	Possible identity
yellow	PbO, S
brown	PbO ₂ , Fe ₂ O ₃
black	C, CuO, MnO ₂
green	Cr ₂ O ₃ , NiCO ₃
red	Pb ₃ O ₄ , Cu ₂ O
off-white	MnCO ₃

2 Flame tests

- To carry out a flame test, a clean nichrome wire is used to mix a sample of a solid with one drop of concentrated hydrochloric acid. The wire is held in progressively hotter parts of a non-luminous Bunsen flame.

Flame colour	Inference
intense, lasting yellow or orange	sodium ion
lilac	potassium ion
orange red*	calcium ion
red*	lithium or strontium ion
pale green	barium ion
blue green	copper (II) ion

* Further tests would be needed to distinguish these ions.

3 Ignition

- Candidates may be asked to heat an unknown alone in an ignition tube or a test tube. The colour changes or the identity of any gases evolved may provide evidence as to the identity of the unknown.

Colour change on heating		Possible hydrated salt
<i>From</i>	<i>To</i>	
blue	white	copper (II) sulphate
blue	black	copper (II) nitrate
red	blue	cobalt(II) salts
green	purple	chromium (III) salts
pale green	brown	iron (II) salts
violet	brown	iron (III) salts

- The residue remaining **after** ignition may have a different colour when hot and cold.

Appearance of residue		Possible identity
<i>Hot</i>	<i>Cold</i>	
white	yellow	zinc oxide
yellow	red	lead (II) oxide
white	white	not a d-block element
green	green	chromium (III) oxide

black	black	oxide of copper or nickel
brown	black	iron (III) oxide
no residue (sublimes)		ammonium salt

- Gases or vapours may be evolved **on heating** the solid.

Gas or vapour	Possible source
carbon dioxide	carbonates of metals other than Group 1; hydrogencarbonates of Group 1
ammonia	ammonium salts
oxygen alone	group 1 nitrates (other than Li)
oxygen and nitrogen dioxide	nitrates (other than Na or K)
hydrogen chloride	hydrated chlorides or ammonium chloride
water	hydrated salts, hydrogencarbonates and other acid salts

4 Recognition and identification of common gases

Gas	Observations
oxygen	colourless gas which relights a glowing splint
carbon dioxide	colourless gas which gives a white precipitate with limewater
sulphur dioxide	colourless gas which is acidic and decolourises acidified potassium manganate(VII)
ammonia	colourless gas which turns moist red litmus paper blue
nitrogen dioxide	brown gas
hydrogen	colourless gas which ignites with a 'pop'
hydrogen chloride	steamy fumes on exposure to moist air, acidic
chlorine	pale green gas which bleaches moist litmus paper
bromine	brown gas
iodine	purple vapour
water vapour	turns blue cobalt chloride paper pink

5 Action of dilute acids

When dilute sulphuric or hydrochloric acid is added to a substance a gas may be evolved or there may be a colour change in solution.

Action of acid	Possible Source
carbon dioxide evolved	carbonate or hydrogencarbonate*
sulphur dioxide evolved on warming	sulphite

hydrogen evolved	a metal
yellow solution turns orange	chromate(VI) to dichromate(VI)
sulphur dioxide evolved and pale yellow precipitate formed	thiosulphate

* To distinguish between the carbonates and hydrogencarbonates of Group 1 prepare an aqueous solution of the salt and proceed as follows.

Test	Observations	
	carbonate	hydrogencarbonate
on aqueous solution		
pH	12–14	7–8
boil	no change	CO ₂ evolved and pH increases to 12–14
add an aqueous Ca or Mg salt and boil the mixture	immediate white precipitate at room temperature	a white precipitate only forms on boiling

6 Tests for oxidising and reducing agents

<p>Reducing agents usually:</p> <ul style="list-style-type: none"> • decolourise aqueous acidified potassium manganate(VII) and • may also turn aqueous, acidified potassium dichromate(VI) from orange to green.
<p>Reducing agents likely to be used in the practical test include:</p> <ul style="list-style-type: none"> • iron (II) ions • sulphite ions, • iodide ions • hydrogen peroxide

<p>Oxidising agents usually:</p> <ul style="list-style-type: none"> • liberate iodine as a brown solution or black solid from aqueous potassium iodide. Iodine solution gives a black colouration with starch.
<p>Oxidising agents likely to be used in the practical test include:</p> <ul style="list-style-type: none"> • manganate(VII) ions • chromate (VI) ions • hydrogen peroxide • copper(II)ions • aqueous chlorine • aqueous bromine

7 Hydrogen peroxide solution

Aqueous hydrogen peroxide (H_2O_2), can act as both an oxidising and a reducing agent often with the evolution of oxygen although this may be unreliable.

Observation on adding H_2O_2	Inference
brown precipitate	manganate(VII); brown precipitate is MnO_2
purple solution is decolourised	manganate(VII) in acid solution
pale green solution turns yellow	iron(II) to iron(III) in acid solution
green precipitate turns brown	iron(II) hydroxide to iron(III) hydroxide
brown precipitate in alkaline solution	lead (II); brown precipitate is PbO_2
green alkaline solution goes yellow	chromium(III) to chromate(VI)
brown solution or black precipitate	iodine from iodide in acid solution

B: Precipitates

When two aqueous solutions are mixed and an insoluble compound is formed this is known as a **precipitate** not a suspension. The observation that a precipitate is formed should always be accompanied with the colour of the precipitate even if this is white. Some reagents should be added until they are in excess. This may result in a precipitate forming then dissolving in excess reagent.

1. Sodium hydroxide solution

- When aqueous sodium hydroxide (NaOH) is added to a solution containing a metal ion a precipitate of the insoluble hydroxide eg $\text{Mn}(\text{OH})_2$ is usually formed. Those precipitates which are amphoteric hydroxides will dissolve in excess sodium hydroxide to give a solution containing a complex ion eg $[\text{Al}(\text{OH})_6]^{3-}$.
- Candidates should assume that aqueous sodium hydroxide should be added until it is **in excess** even if this is not explicitly stated in the instructions.

Observation on adding aqueous NaOH	Observation on adding excess NaOH	Likely ion
off white precipitate which darkens on exposure to air	precipitate is insoluble	manganese(II)
green precipitate which turns brown on exposure to air	precipitate is insoluble	iron(II)
brown precipitate	precipitate is insoluble	iron(III)
green precipitate	precipitate is insoluble	nickel(II)
blue precipitate (which turns pink on standing)	precipitate is insoluble	cobalt(II)
blue precipitate	precipitate is insoluble	copper(II)
white precipitate	precipitate dissolves to a colourless solution	zinc, lead(II), aluminium
green precipitate	precipitate dissolves to a green solution	chromium(III)
white precipitate	precipitate is insoluble	magnesium, lithium, barium, strontium, calcium
no precipitate		sodium, potassium
no precipitate but ammonia evolved.		ammonium
orange to yellow solution		dichromate(VI) to chromate(VI)

2. Ammonia solution

- Aqueous ammonia (NH_3), when added to a solution containing a cation, will form the same hydroxide precipitate as does aqueous sodium hydroxide eg $\text{Mn}(\text{OH})_2$. Excess aqueous ammonia may dissolve the precipitate to form a complex ion eg $[\text{Cu}(\text{NH}_3)_4(\text{H}_2\text{O})_2]^{2+}$.
- Candidates should assume that aqueous ammonia must be added until it is **in excess**.

Observation on adding aqueous NH_3	Observation on adding excess NH_3	Likely ion
Green precipitate	precipitate is slightly soluble	chromium(III)
green precipitate turning brown	precipitate insoluble	iron(II)
brown precipitate	precipitate insoluble	iron(III)
blue precipitate	precipitate dissolves to give brown solution	cobalt(II)
green precipitate	precipitate dissolves to give blue solution	nickel(II)
blue precipitate	precipitate dissolves to give deep blue solution	copper(II)
white precipitate	precipitate dissolves to give colourless solution	zinc
white precipitate	precipitate is insoluble	aluminium, magnesium, lead(II)
off-white precipitate	precipitate is insoluble	manganese(II)

3 Sodium carbonate

- Aqueous sodium carbonate (Na_2CO_3), will form precipitates with all of the d-block cations. Those with a 3+ charge will form a hydroxide with effervescence (CO_2). Others will form an insoluble carbonate.
- None of the precipitates will dissolve in excess aqueous sodium carbonate.

Observation on adding aqueous Na_2CO_3	Likely ion	Formula of precipitate
white precipitate	calcium, strontium, barium, lithium, magnesium, lead	CaCO_3 , SrCO_3 , BaCO_3 , Li_2CO_3 , MgCO_3 , PbCO_3
white precipitate	silver	Ag_2CO_3
green precipitate	iron(II), nickel	FeCO_3 , NiCO_3
off-white precipitate	manganese	MnCO_3
pink precipitate	cobalt (II)	CoCO_3
blue precipitate	copper(II)	CuCO_3
white precipitate with effervescence	aluminium	$\text{Al}(\text{OH})_3$
brown precipitate with effervescence	iron(III)	$\text{Fe}(\text{OH})_3$
green precipitate with effervescence	chromium(III)	$\text{Cr}(\text{OH})_3$

4. Lead nitrate or lead ethanoate solution

- Most lead(II) compounds are insoluble. When aqueous lead ions (either lead nitrate or lead ethanoate) are added to a solution containing the appropriate anion a precipitate will form.

Anion	Precipitate	
	<i>colour</i>	<i>formula</i>
carbonate	white	PbCO ₃
sulphate	white	PbSO ₄
chloride	white	PbCl ₂
bromide	white	PbBr ₂
iodide	yellow	PbI ₂

5. Barium chloride solution

- Aqueous barium chloride forms precipitates of insoluble barium salts with a number of anions but is usually used as the test for the sulphate, SO₄²⁻, ion. Aqueous barium chloride is usually used with dilute hydrochloric acid.

Anion	Precipitate		Addition of dilute HCl
	<i>colour</i>	<i>formula</i>	
sulphate	white	BaSO ₄	precipitate is insoluble
sulphite	white	BaSO ₃	precipitate dissolves
carbonate	white	BaCO ₃	precipitate dissolves with effervescence
chromate(VI)	yellow	BaCrO ₄	precipitate dissolves to give orange solution

- If dilute hydrochloric acid is added to the anion solution **before** aqueous barium chloride then only the sulphate will form as a precipitate.

6 Silver nitrate solution

- Aqueous silver nitrate is commonly used to test for the presence of halide ions in solution. Anions which would interfere with the test (eg carbonate) are first removed by adding dilute nitric acid before the aqueous silver nitrate.
- The identity of a halide may be confirmed by the addition of aqueous ammonia, (NH₃) both dilute and concentrated.
- The silver halides which dissolve in ammonia do so to form a colourless solution of the complex ion, [Ag(NH₃)₂]⁺

Anion	Precipitate		Addition of aqueous NH ₃	
	colour	formula	dilute	concentrated
chloride	white	AgCl	soluble	
bromide	cream	AgBr	slightly soluble	soluble
iodide	yellow	AgI	insoluble	insoluble

7 Concentrated sulphuric acid

- When a few drops of concentrated acid (H₂SO₄) are added to a solid halide the observed reaction products may be used to identify the particular halide ion present. **This is a potentially hazardous reaction.**
- **It must be carried out on a small scale and in a fume cupboard.**
- The products in brackets will not be observed since they are colourless gases. The halide ion may be identified without the need to test for these gases. **No attempt should ever be made to detect these gases by smell.**

Halide	Observations on adding conc H ₂ SO ₄	Observed reaction products
chloride	steamy vapour	HCl
bromide	steamy vapour, brown vapour	HBr, Br ₂ (SO ₂)
iodide	steamy fumes, black solid, purple vapour, yellow solid, vigorous reaction	HI, I ₂ , S, (H ₂ S)

8 Test for the nitrate ion

Since all nitrates are soluble no precipitation test is possible for the nitrate ion. A solid which is suspected of being a nitrate then the following tests should be carried out:

- warmed with aqueous sodium hydroxide and aluminium or zinc powder
- if the solid is a nitrate then ammonia gas will be evolved. This will turn damp red litmus blue.

C: Organic compounds

Candidates will always be told that a compound or mixture of compounds to be identified is organic. Often the molecular formula, or the number of carbon atoms in a molecule, of a compound will be given. Chemical tests may be followed by spectroscopic information.

1 Appearance

Simple organic compounds used in the practical test are usually colourless liquids or white solids. It is unlikely that appearance will provide candidates with firm evidence for identification.

2 Solubility

Solubility of compound	Possible identity	pH of solution	Possible identity
dissolves in water	simple alcohols, simple carboxylic acids, propanone, simple aldehydes, simple amines and their salts	above 7	amines
		below 7	carboxylic acids
dissolve in dilute acid but may not dissolve in water	amines		
dissolve in aqueous alkali but may not dissolve in water	carboxylic acids, phenols		

3 Ignition

Heating an organic unknown on a crucible lid may be of value in identifying it.

Observation	Possible inferences
burns with a smoky flame	aromatic, unsaturated
burns with a clean flame	saturated low molar mass compound
no residue	most lower molar mass compounds

4 Chemical tests

The details of how these tests are to be carried out will be included in the instructions to candidates in the practical tests

Test	Observation	Inference
warm with acidified potassium dichromate(vi)	orange to green solution	primary or secondary alcohol; aldehyde
shake with bromine solution	yellow solution is decolourised	alkene
	if white precipitate also formed	phenol
warm with aqueous sodium hydroxide, acidify with dilute nitric acid then add aqueous silver nitrate	precipitate: white cream yellow	Halogenoalkanes C-Cl C-Br C-I
phosphorus pentachloride	effervescence, steamy fumes of HCl	OH group in alcohols and carboxylic acids
2,4-dinitrophenylhydrazine (Brady's Reagent)	orange precipitate	C=O group in aldehydes and ketones
boil with Fehling's solution	blue solution gives red precipitate	aldehyde
warm with ammoniacal silver nitrate	silver mirror	aldehyde
sodium or potassium hydrogencarbonate	effervescence, CO ₂ gas evolved	carboxylic acid
iodine in alkaline solution or aqueous potassium iodide and sodium(i) chlorate – the iodoform test	pale yellow precipitate	-C-CH ₃ O (methyl ketone or ethanal), CH ₃ CH(OH) – (methyl secondary alcohol or ethanol)
warm with carboxylic acid and a few drops of concentrated sulphuric acid	ester smell eg glue-like	alcohol
sodium nitrite and dilute hydrochloric acid followed by an alkaline solution of phenol in ice-cold conditions	orange precipitate	aromatic amine

D: Spectroscopy

Modern instrumentation plays a vital role in the characterisation and identification of molecules and ions. The sections which follow will enable interpretation of the data which are likely to be presented to candidates in the practical test 6A only.

1. Mass spectrometry

Simplified mass spectra will be given which should be interpreted **without** reference to fragmentation tables. However, some important points are summarised here:

- the value of the compound's relative molecular mass may be obtained from the m/z value of M^+ , the molecular ion. This will have the highest value of m/z . This need not necessarily be the 'base' peak – which is simply the most abundant ion
- if the compound contains one or more chlorine or bromine atoms, the presence of isotopes will give two or more molecular ions.

2. Infra-red spectroscopy

- This is a very powerful non-destructive technique which provides information regarding the nature of covalent bonds within the molecule.
- Candidates should look at the most intense absorptions to gain structural clues quickly. Table 1 provides sufficient details to enable the principal bands to be assigned. Candidates should remember that the absorption frequency is affected by the chemical environment and that absorption may take place outside the range given.
- The connection between structures should be recognised, for instance, an alcohol [O-H] stretch will be accompanied by a [C-O] stretch.

Table 1


Characteristic infra-red absorbencies in organic molecules

m =medium

s =strong

v =variable


br =broad

Bond	Assignment	Wavenumber/cm ⁻¹
C-H	alkanes	2850 – 2950 m/s
	alkenes, arenes	3000 – 3100 m/s
C=C	alkenes	1610 – 1680 m
	arenes	1450 – 1650 v (several bands)
C=O	aldehydes ketones, amides, esters, carboxylic acids	1680 – 1750 s
C-O	alcohols, esters	1000 – 1300 s
	aromatic esters	1300 – 1400 s
C≡N	nitriles	2215 – 2260 m
C-Cl		700 – 800s
O-H	'free'	3580 – 3670 s
	hydrogen bonded alcohols/phenols	3230 – 3550 s (br)
	hydrogen bonded carboxylic acids	2500 – 3300 s (br)
N-H	primary amines	3200 – 3500 s
N-H	amide	3140 – 3500 s

3. Proton magnetic resonance

- Proton magnetic resonance spectra may be included as part of an investigation into structure. Spectra will be low resolution with no spin – spin coupling displayed. Chemical shifts are given in Table 2, relative to TMS as internal standard

Table 2


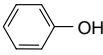
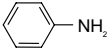
Type of Proton	Chemical shift/ppm
R —CH ₃	0.9
R —CH ₂ —R	1.3
$\begin{array}{c} \text{R} - \text{CH} - \text{R} \\ \\ \text{R} \end{array}$	2.0
$\begin{array}{c} - \text{C} - \text{CH}_3 \\ \\ \text{O} \end{array}$	2.3
$\begin{array}{c} > \text{C} = \text{C} < \\ & \text{H} \end{array}$	4.7
$\begin{array}{c} \text{H} \\ \diagdown \\ - \text{O} \end{array}$	5.0
$\begin{array}{c} > \text{C} = \text{C} < \\ & \text{H} \end{array}$	5.3
$\begin{array}{c} \text{H} & & \text{H} \\ & \diagdown & / \\ & \text{C} = \text{C} & \\ & / & \diagdown \\ \text{H} & & \text{C} - \\ & & \\ & & \text{O} \end{array}$	6.1
	7.5
$\begin{array}{c} \text{O} \\ // \\ - \text{C} \\ \backslash \\ \text{H} \end{array}$	9.5
$\begin{array}{c} \text{O} \\ // \\ - \text{C} \\ \backslash \\ \text{O} - \text{H} \end{array}$	11.0
—CH ₂ —Cl	3.6
—CH ₂ —Br	3.5
—CH ₂ — I	3.2
—CH ₂ —C≡N	2.3

Type of Proton	Chemical shift/ppm
$\begin{array}{c} \text{--- CH --- Br} \\ \\ \text{R} \end{array}$	4.3
$\text{C--- CH}_2\text{--- OH}$	3.6
$\begin{array}{c} \text{C--- CH --- OH} \\ \\ \text{R} \end{array}$	3.9
$\begin{array}{c} \text{CH}_3\text{--- C} \begin{array}{l} \nearrow \text{O} \\ \searrow \text{O---R} \end{array} \end{array}$	2.0
$\begin{array}{c} \text{O} \\ \\ \text{CH}_3\text{--- O--- C--- R} \end{array}$	3.7

4. UV/visible spectra

- The visible and UV spectra of organic molecules and of transition metal ions are associated with transitions between electronic energy levels. The ultra-violet region of the electromagnetic spectrum extends from approximately 160 – 400 nm, and the visible from 400 nm (violet) to 700 nm (red). Absorbance in the blue end gives the compound a red colour (ie the complementary colour).
- Some typical absorption maxima for groups of atoms in organic compounds are given in Table 3.

Table 3

Chromophore	Maximum absorption wavelength/nm
$\text{—C}\equiv\text{N}$	160
R—Cl	170
R—OH	180
>C=C<	190
	190 and 260
>C=O	190 and 280
R-NH_2	195
$\begin{array}{c} \text{—C—O—} \\ \\ \text{O} \end{array}$	200
R-Br	205
$\begin{array}{c} \text{O} \\ // \\ \text{—C} \\ \backslash \\ \text{NH}_2 \end{array}$	210
	210 and 270
$\text{CH}_2=\text{CH—C—}$ $\quad\quad\quad $ $\quad\quad\quad \text{O}$	220 and 325
	230 and 280
R—I	260
$\overset{+}{\text{N}}\equiv\text{N}$	350

Textbooks and other resources

Textbooks available from publishers

<i>Advanced Practical Chemistry</i> , Alec Thompson et, John Murray	ISBN 0 7195 4188 3
<i>Chemistry Data Book</i> , Stark and Wallace, John Murray	ISBN 0 7195 3951 X
<i>A-Level Practical Chemistry</i> , Ratcliff, Cambridge	ISBN 0 521 37899 0
<i>A-Level Practical Chemistry (teacher's guide)</i> , Ratcliff, Cambridge	ISBN 0 521 38696 9

Additional useful resource material

Basic Laboratory Skills: A Training Pack for Laboratory Techniques, Peter Houlgate and Elizabeth Pritchard, LGC (Teddington) Ltd. The pack consists of a booklet: *Guide to Improving Analytical Quality in Chemistry*, and a CD Rom: *Basic Laboratory Skills*.

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