6092 Practical Revision Guide

Chemistry Practical Test

Candidates may be asked to carry out exercises based on:

1. Titration, e.g. acid-base titration (with suitable indicators such as methyl orange, screened methyl orange, and thymolphthalein). Other types of titrations may also be required, and where appropriate, sufficient working details will be given.

2. Speeds of reaction that may involve measuring of quantities, e.g. temperature, volume, length, mass or time measurements

3. Experiments involving separation techniques such as paper chromatography, filtration and distillation

4. Salt preparation

5. Gas collection Candidates would not be required to carry out drying of gases during practical examination.

6. Qualitative inorganic analysis involving an element, a compound or a mixture, including displacement reactions and tests for oxidising and reducing agents. Candidates should be familiar with the reactions of cations, reactions of anions and tests for gases as detailed in the Notes for Qualitative Analysis. Candidates would not be required to carry out tests involving sulfur dioxide gas. Reactions involving ions not included in the Notes for Qualitative Analysis may be tested: in such cases, candidates will not be expected to identify the ions but only to draw conclusions of a general nature. Candidates should not attempt tests, other than those specified, on substances, except when it is appropriate to test for a gas.

7. Qualitative organic analysis requiring a knowledge of simple organic reactions as outlined in Topic 11 Organic Chemistry, e.g. test-tube reactions indicating the presence of unsaturation (C=C) may be set, but this would be for the testing of observation skills and drawing general conclusions only.

Practical Techniques

a) Candidates should normally record burette readings to the nearest 0.05 cm^3 and they should ensure that they have carried out a sufficient number of titrations, e.g. in an experiment with a good end-point, two titres within 0.20 cm^3.

b) In qualitative analysis, candidates should use approximately 1 cm depth of a solution (1–2 cm^3) for each test and add reagents slowly, ensuring good mixing, until no further change is seen. Candidates should indicate at what stage a change occurs. Answers should include details of colour changes and precipitates formed, and the name and test for any gases evolved.

Tips on Qualitative Analysis

A. Procedures for Heating

1. Keep the **air hole closed** when lighting a Bunsen burner.
2. When the **Bunsen burner is lighted**, **open the air hole a little**.
3. Use a test tube holder when heating anything in a test tube.
4. **Slant** the test tube.
5. Point the mouth of the test tube **away** from yourself and anyone nearby.
6. While heating, remove the test tube from the flame every now and then and shake the tube gently. (to prevent the solution from splurting out)
7. Ensure that the test tube is **dry** if you are heating a solid and observe if any water droplets formed at the side of test tube during heating.

B. Recording Observations

1. Record your observations (**in pen**) as you are conducting the tests. This will ensure that important observations are not left out.
2. Negative results must be recorded as correct inferences can be drawn from these. Example of negative results: **No precipitate is formed** or **solution remains colourless.**
3. If a precipitate forms slowly, say so in your record of observations.
4. There is an observation for every test. IF you do not observe any change, then you should record '**no visible change**' or '**no visible reaction**'. **Do not leave a blank space in your record.**
5. **Record your observations directly next to the instructions.** This is important when the test instructions consist of two or more separate steps.
6. **You must only carry out the tests in the practical examinations.** You must NOT do extra tests of your own (unless asked to do so).
7. Remember that the practical exam tests your **practical skills**, **observation powers** and ability to **make correct conclusions**.

Understanding Practical Instructions

warm gently	adjust gas tap so that flame is small (CANNONT HEAR SOUNDS) look out for: • colour change of solid / solution • test for gas evolved
heat strongly	adjust gas tap so that flame is big (CAN HEAR SOME SOUNDS) look out for: • colour change of solid / solution • test for gas evolved
to a portion	about 2 cm^3 ≈ a section of your thumb
until a change is seen	add a few drops at a time, stop and observe
until no further change is seen	add a few drops at a time, stop and observe, and continue adding a little at a time with shaking until ¾ full, observe
add excess	adding a little at a time with shaking until ¾ full, observe
leave to stand	leave aside and check occasionally and record observations look out for: • colour change of solid / solution
filter the mixture	leave aside and check occasionally and record observations for BOTH filtrate & residue!

Time Saving Tips

warm gently	prepare to test for gas immediately (DON'T LET THE GAS ESCAPE)
heat strongly	prepare to test for gas immediately (DON'T LET THE GAS ESCAPE)
leave to stand	leave aside and check occasionally (GO & DO OTHER THINGS)
filter the mixture	leave aside and check occasionally (GO & DO OTHER THINGS)
test for SO_2	leave the filter paper strip on the test tube and check occasionally (GO & DO OTHER THINGS)
if test for gas negative	pour away the reagents and re-use test tube, repeat experiment (THERE IS NO NEED TO WASH BECAUSE YOU ARE INTRODUCING THE SAME REAGENTS)

(a) Tests for Cations

You are expected to test for and identify cations and use these test results to answer questions in the conclusion. Cations can be identified by using sodium hydroxide solution and aqueous ammonia. **All cations (except Na^+, K^+ and NH_4^+) give precipitate with these alkalis.**

Objectives

1. To note the colour of the precipitate produced.

2. To see whether the precipitate is soluble or insoluble in an excess of the reagent.

cation	*effect of **aqueous sodium hydroxide***	*effect of **aqueous ammonia (ammonium hydroxide, NH_4OH)***	*type of reaction*
ammonium (NH_4^+)	• on warming, gas produced turns damp red litmus paper blue • **ammonia** gas produced	-	alkali + ammonium salt → salt + ammonia gas + water
calcium (Ca^{2+})	• white ppt., insoluble in excess • **calcium hydroxide** formed	no ppt.	precipitation
copper(II) (Cu^{2+})	• light blue ppt., insoluble in excess • **copper(II) hydroxide** formed	• light blue ppt., soluble in excess giving a dark blue solution • **copper(II) hydroxide** formed	precipitation
iron(II) (Fe^{2+})	• green ppt., insoluble in excess • **iron(II) hydroxide** formed	• green ppt., insoluble in excess • **iron(II) hydroxide** formed	precipitation
iron(III) (Fe^{3+})	• red-brown ppt., insoluble in excess • **iron(III) hydroxide** formed	• red-brown ppt., insoluble in excess • **iron(III) hydroxide** formed	precipitation

zinc (Zn^{2+})	• white ppt., soluble in excess giving a colourless solution • **zinc hydroxide** formed	• white ppt., soluble in excess giving a colourless solution • **zinc hydroxide** formed	precipitation
aluminium	• white ppt., soluble in excess giving a colourless solution • **aluminum hydroxide** formed	• white ppt., insoluble in excess • **aluminum hydroxide** formed	precipitation

Procedures

1. Use about 2 cm^3 (approximately 1 cm depth) of solution containing the cation.
2. Get ready the materials (damp red litmus paper) if ammonia is expected to be formed.
3. Add **2 to 3 drops** of aqueous sodium hydroxide or aqueous ammonia **slowly**.
4. **Mix the solutions by shaking it thoroughly after each addition of reagent**.
5. Look out for the formation of any precipitate. Note the colour carefully.
6. **When carrying out an experiment, always leave the tube to stand for one or two minutes as some changes take some time.**
7. Add more (excess) aqueous sodium hydroxide or aqueous ammonia.
8. Observe if the precipitate formed dissolves in an excess of the reagent. **You may need to shake the test tube slightly.**

Explanation

Compounds that are insoluble in water appear in the form of a precipitate.
When aqueous sodium hydroxide (contains OH^- ions) is added to a cation, a hydroxide is formed. Most metal hydroxides are insoluble in water.

Examples:

$Cu^{2+}(aq) + 2OH^-(aq) \rightarrow Cu(OH)_2(s)$

$Fe^{3+}(aq) + 3OH^-(aq) \rightarrow Fe(OH)_3(s)$

Writing Observations

test no.	test	observations
1	**(a)** To a portion of **solution X**, add dilute sodium hydroxide until a change is seen. **(b)** Add an excess of dilute sodium hydroxide to the mixture from **(a)**.	**A white precipitate is formed.** **The white precipitate is soluble in excess dilute sodium hydroxide, forming a colourless solution.**
2	**(a)** To a portion of **solution X**, add dilute ammonia solution until a change is seen. **(b)** Add an excess of dilute ammonia to the mixture from **(a)**.	**A white precipitate is formed.** **The white precipitate is insoluble in excess dilute sodium hydroxide.**

(b) Tests for Anions

You are expected to test for and identify anions and use these test results to answer questions in the conclusion. Some anions (Cl^- or SO_4^{2-}) can be identified by the precipitates which their solutions give with different reagents.

Objectives

1. To note the colour of any precipitate formed.
2. To record and identify any gases that may be produced.
3. To confirm the anion(s) present in a salt or a mixture.

anion	***test***	***test result***	*type of reaction*
carbonate (CO_3^{2-})	add dilute acid	• effervescence is seen • gas gives white ppt. with limewater (calcium hydroxide) • **carbon dioxide** gas produced	acid + carbonate → salt + carbon dioxide + water
chloride (Cl^-) [in solution]	acidify with dilute nitric acid, then add aqueous silver nitrate	• white ppt. • **silver chloride** formed	precipitation
nitrate (NO_3^-) [in solution]	add aqueous sodium hydroxide, then **aluminium foil**; warm carefully	• gas turns damp red litmus paper blue • **ammonia** gas produced	not in syllabus
sulfate (SO_4^{2-}) [in solution]	acidify with dilute nitric acid, then add aqueous barium nitrate	• white ppt. • **barium sulfate** formed	precipitation

Procedures

1. Use about 2 cm^3 (approximately 1 cm depth) of solution containing the anion.
2. Get ready the materials (e.g. limewater and damp red litmus paper) if a gas is expected to be formed.
3. Add reagents slowly in small amounts.
4. Mix the solutions by shaking it thoroughly after each addition of reagent.
5. Observe any changes that occur.

   When carrying out an experiment, always leave the tube to stand for one or two minutes as some changes take some time.

Explanation

(a) Chlorides: react with silver ions from aqueous silver nitrate to give a white precipitate of silver chloride.

$$Ag^+(aq) + Cl^-(aq) \rightarrow AgCl(s)$$
white precipitate

(b) Sulfates: react with barium ions from aqueous barium nitrate to give a white precipitate of barium sulfate.

$$Ba^{2+}(aq) + SO_4^{2-}(aq) \rightarrow BaSO_4(s)$$
white precipitate

In all the above tests (a) and (b), dilute acid is usually added to remove other anions, such as carbonate, which might also give precipitates with the reagents.

(c) Carbonates: identified by adding dilute hydrochloric acid or dilute nitric acid. Carbonates react with acids to produce carbon dioxide gas which forms a white precipitate with limewater. Sulfuric acid can also be used, but some carbonates react very slowly with this acid and produce too little gas for testing with limewater.

(d) Nitrates: identified by reacting with aluminium metal and dilute sodium hydroxide. Ammonia gas is produced in the reaction and this is identified with damp red litmus paper. Hydrogen gas is also produced in this test, by the reaction of aluminium with sodium hydroxide, but this is insignificant. (Hydrogen is produced whether nitrate is present or not.)

Writing Observations

test	observations	identity of anion
Dissolve about three-quarters of substance **A** in 20 cm^3 of water. Use about 2 cm^3 portions for each of the following tests: **(i)** Add aqueous barium chloride, followed by dilute hydrochloric acid.	**White ppt formed.**	**Sulfate ions present.**

(c) Tests for Gases

Only test for gases when you see that a gas is being produced. When you:

- can see bubbles in a liquid or fumes, a gas is being produced.
- heating a solid gently or strongly, a gas is produced.

Objectives

1. To identify a number of gases that may be produced in chemical tests.

2. To record the names of gases and tests that you did to identify them. This is done as part of your observations.

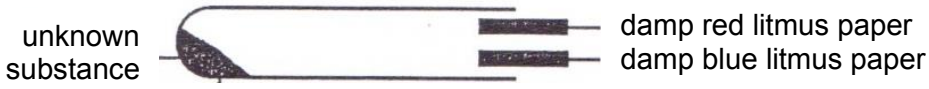
Procedures

1. Note the colour of the gas.

2. Note the smell of the gas.

3. Test the gas using damp litmus paper.

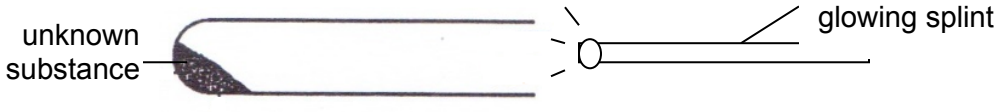
   (a) Make sure the damp litmus paper does not touch the sides of the test tube.
   (b) Do NOT drop the damp litmus paper into the test tube.



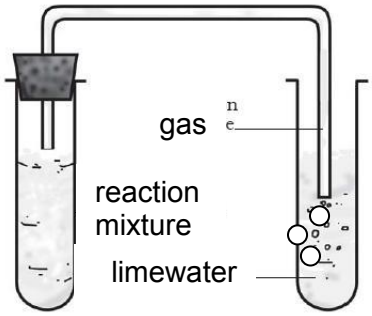
4. **Note: Steps 1 to 3 help you narrow down the number of gases to test for.**

5. Carry out the appropriate test to identify the gas.

6. Test the gas with a glowing (**orange glow**) or lighted (**with flame**) wooden splint.
   (a) Make sure the wooden splint does not touch the sides of the test tube.
   (b) Do NOT insert the wooden splint into the test tube such that it touches the sample.

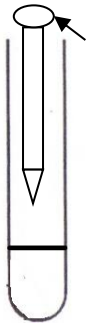


7. Test the gas with limewater or acidified potassium manganate(VII) solution using delivery tube.
   (a) If the gas is to be tested while the reaction mixture is heated, remove the test tube of limewater or acidified potassium manganate(VII) solution before the Bunsen burner is turned off. (to prevent suck-back of limewater that will contaminate the reaction mixture)

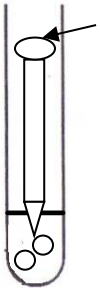


8. For tests involving passing a gas into another reagent via a delivery tube, use as little of the reagent as possible. This will give quicker and more effective results.

9. Test the gas with limewater using teat pipette.
   (a) Collect the gas using a teat pipette. Make sure that the teat pipette does NOT touch the reaction mixture.
   (b) Insert the teat pipette into limewater and release the gas. Bubbles will be seen.

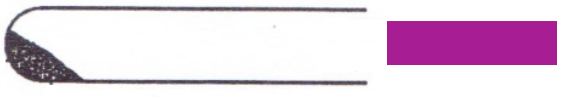


squeeze the bulb and release it while the teat pipette is not submerged in reaction mixture to withdraw the gas



squeeze the bulb while the teat pipette is submerged in reaction mixture to release the gas

10. Test the gas with acidified potassium manganate(VII) using filter paper .
    (a) Add sulfuric acid to potassium manganate(VII) solution just before the test is to be conducted.
    (b) Add a few drops of acidified potassium manganate(VII) solution to a piece of filter paper.



filter paper soaked with acidifed potassium manganate(VII)

test result	possible gas(es)	Is litmus test confirmatory?	follow-up test
The gas produced **turns damp blue litmus paper red**.	carbon dioxide or sulfur dioxide	no	• limewater (for CO_2) • acidified potassium manganate(VII) (for SO_2)
The gas produced **bleaches** damp red or blue litmus paper.	chlorine	yes	Nil
The gas produced **turns damp red litmus paper blue**.	ammonia	yes	Nil
The gas produced causes damp red litmus paper to **remain red** and damp blue litmus paper to **remain blue.**	hydrogen or oxygen	no	• lighted splint (for H_2) • glowing splint (for O_2)

Writing Observations

test	observation	identity of gas
Place a little **solid X** in a test tube. Add dilute hydrochloric acid and warm the test tube.	**Colourless and odourless gas is produced.** **Gas forms a white ppt in limewater.**	**Carbon dioxide**
Dissolve a little of **solid F** in water in a test tube. Then add a little dilute hydrochloric acid. Leave the mixture to stand for a few minutes.	**Colourless and pungent gas is produced.** **Gas turns aqueous acidified potassium manganate(VII) purple to colourless.**	**Sulfur dioxide**

Appearance of Solids

Observation	Possible substance present
colourless crystals	salts of Group I, II & III elements, NH_4^+ or Zn^{2+} or Pb^{2+}
white powder	salts of Group I, II & III elements, NH_4^+ or Zn^{2+} or Pb^{2+}
yellow (hot) white (cold)	ZnO
silver grey powder	metals
black powder	CuO, C, MnO_2
green powder	$CuCO_3$
blue crystals	Cu^{2+} salt
pale green crystals	Fe^{2+} salt
yellow or brown crystals	Fe^{3+} salt

Action of Heat on Solids

Observation	Possible substance present
colour change: green to black	$CuCO_3$
colour change: blue to white	$CuSO_4.5H_2O$
colour change: yellow (hot) white (cold)	ZnO
gas evolved: CO_2	carbonate, C
gas evolved: O_2	nitrates, oxide
gas evolved: NO_2 (acidic, brown)	nitrates
gas evolved: NH_3	NH_4^+ salt
gas evolved: NH_3 & HCl (leaving no residue)	NH_4Cl

C. Test for Oxidising Agents and Reducing Agents

- To test for a reducing agent, an oxidising agent is added to it.

oxidising agent	observation	inference
acidified potassium manganate(VII) solution (acidify with sulfuric acid)	Acidified potassium manganate(VII) solution turns from purple to colourless.	presence of reducing agent

- To test for an oxidising agent, a reducing agent is added to it.

reducing agent	observation	inference
potassium iodide solution	Potassium iodide solution turns from colourless to brown. (presence of I_2 formed is confirmed by adding starch which turns dark blue)	presence of oxidising agent

D. Flame Test

The flame test is used to visually determine the identity of an unknown metal ion based on the characteristic color the salt turns the flame of a Bunsen burner.

You will **NOT** be asked to identify the metal present in the unknown based on the flame colour.

Procedure:
a. Adjust Bunsen burner to blue flame.
b. **Moisten** the end of a wooden splint with water.
c. Dip the moist end of the splint into the sample.
d. Place the end of the splint in the Bunsen burner flame.
Note: Use a new splint for each test.

metal	flame colour
calcium	red
copper	green
iron	gold
lead	blue
potassium	lilac
sodium	yellow
zinc	blue-green

For the BLUR KINGS & QUEENS

ppt ≠ solution	colour of ppt may not necessarily be the same as the colour of the solution it is in! check colour of ppt against a white background
don't use the term: "milky"	white ppt is formed
don't use the term: "cloudy"	white ppt is formed
don't use the term: "chalky"	white ppt is formed
don't use the phrase: "white solution"	white ppt is formed
don't use the phrase: "white mixture"	white ppt is formed
don't use the phrase: "white suspension"	white ppt is formed
don't use the phrase: "clear solution"	state colour of solution (ALL SOLUTIONS ARE CLEAR...)
don't use the phrase: "transparent solution"	state colour of solution (ALL SOLUTIONS ARE TRANSPARENT...)
don't use the phrase: "solution turns into a white ppt"	white ppt is formed
if ppt refuse to mix (cos u forgot to shake)	use stirrer provided
if aq NH_3 is added	don't test for NH_3 (YOU JUST ADDED IT!!)
thorough mixing is important	or you will miss some observations

don't use the phrase: "ppt settles at the bottom and solution is on top"	Why tell the marker that you did not mix thoroughly??
Bubbles from boiling is not effervescence	You just boiled the solution!! It may not be a gas being formed.
Always read the first paragraph for clues to the identity of the unknown	If not why would it be there???
Always read the question carefully	formula or name? (WRITE PROPERLY!!!)

Tips on use of different experimental apparatus

Quantity	S.I. Unit	Apparatus for measurement	Record in
time	second (s) [1h = 60 min] [1 min = 60 s]	Electronic stopwatch	**nearest second** eg 58 s , 60 s
temperature	Kelvin (K) more commonly used - degree Celsius (°C)	Laboratory thermometer	**nearest 0.5°C** eg 30.5 °C , 30.0 °C
mass	Kilogram (kg) Other units – gram (g) [1 kg = 1000 g]	Electronic balance	**nearest 0.01 g** eg 60.15 g
volume	Cubic metre (m^3) Other units - litre (l), cubic centimetre (cm^3)	<u>liquid</u> Pipette – fixed volumes (eg. 20.0 cm^3, 25.0 cm^3) Burette – accuracy to 2 d.p. (eg. 24.85 cm^3)	pipette – 25.0 cm^3 burette – **nearest 0.05 cm^3**

	[1 m^3 = 1000dm^3] [1 dm^3 = 1000cm^3]	Measuring cylinder – accuracy to 1 d.p (eg. 20.5 cm^3)	measuring cylinder – **nearest 0.5 cm^3**
		<u>gas</u> gas syringe	

Samples of Sources of Error

NOTE: **These are not the only sources of error. You cannot just memorise the errors as it may not apply to the experiment you are doing. Different experiments will have different errors. You must study the experiment properly.**

You must state specifically how the error causes an inaccuracy. For example, *heat lost to environment during stirring may lower the temperature of the solution.*

Type of experiment	Source of Error	How does it cause inaccuracy
Retrieving salts	• Insufficient time for crystals to form	• Decrease amount of product (crystals) formed. • Affects percentage yield.
	• Some crystals are stuck on the filter paper during filtration.	
	• Drying the crystals between pieces of filter paper causes some crystals to be stuck in the filter paper.	
Determining mp/bp/ temperature of a substance	• Human reaction time in reading the temperature at the specific time. Slight time lapse in recording the temperature.	• Temperature recorded at that time would be higher/ lower.
	• Inconsistent stirring when dissolving the substance.	• There will be uneven heating/ cooling. Hence temperature will rise/fall at an inconsistent rate.
	• Heat lost to environment due to... (Depending on the experiment)	• Lower the temperature recorded.
	• Temperature in the laboratory not constant	• Fluctuations may increase/ decrease the temperature reading.
Quantitative Analysis (Titration)	• Oxidising/ reducing impurities in air	• The volume of solution added from the burette may be more/less (depending on the situation)
	• Acidic/ alkaline impurities (reacts with alkali/ acid)	• (inaccurate burette reading – either more or less depending on the situation)

in air.

→ No more inconsistent swirling.

→ Avoid dark colour of solution.

- Alkali in air react with acid
- Con of acid will drop
- More volume of acid need to react with Alkali in exp

25.1 25.3 25.4

Speed affected by swirling

Swirling of the mixuture due to pouring

If place conical flask place on cross

↳ Allow liquid to settle than pour other soln in

↳ Due to pouring of other soln, the swirling of the liquid may speed up rxt

→ Best to flush out all air bubbles. from burette

→ Pipette.
Bottom of flask or surface of liquid (a few times)

→ Fill Burette above "0" line then flush out

→ Table must have correct heading & units.