

# Practical Booklet 1

## Gravimetric analysis

# Cambridge International AS & A Level

## Chemistry 9701

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## Introduction

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Practical work is an essential part of science. Scientists use evidence gained from prior observations and experiments to build models and theories. Their predictions are tested with practical work to check that they are consistent with the behaviour of the real world. Learners who are well trained and experienced in practical skills will be more confident in their own abilities. The skills developed through practical work provide a good foundation for those wishing to pursue science further, as well as for those entering employment or a non-science career.

The science syllabuses address practical skills that contribute to the overall understanding of scientific methodology. Learners should be able to:

- plan experiments and investigations
- collect, record and present observations, measurements and estimates
- analyse and interpret data to reach conclusions
- evaluate methods and quality of data, and suggest improvements.

The practical skills established at AS Level are extended further in the full A Level. Learners will need to have practised basic skills from the AS Level experiments before using these skills to tackle the more demanding A Level exercises. Although A Level practical skills are assessed by a timetabled written paper, the best preparation for this paper is through extensive hands-on experience in the laboratory.

The example experiments suggested here can form the basis of a well-structured scheme of practical work for the teaching of AS and A Level science. The experiments have been carefully selected to reinforce theory and to develop learners' practical skills. The syllabus, scheme of work and past papers also provide a useful guide to the type of practical skills that learners might be expected to develop further. About 20% of teaching time should be allocated to practical work (not including the time spent observing teacher demonstrations), so this set of experiments provides only the starting point for a much more extensive scheme of practical work.

## Guidance for teachers

### Aim

To determine the formula of magnesium oxide by measuring the increase in mass of a piece of magnesium ribbon when heated in air.

### Outcomes

Syllabus section 1.4(b) and 1.5(a) and experimental skills 2, 3 and 4.

The further work includes the possibility of supporting experimental skill 1.

### Skills included in the practical

AS Level skills		How learners develop the skills
<b>Manipulation, measurement and observation</b>	<i>Successful collection of data and observations</i>	use the apparatus successfully to obtain an increase in mass
	<i>Decisions relating to measurements or observations</i>	allow air into the crucible or loss of product as smoke
<b>Presentation of data and observations</b>	<i>Recording data and observations</i>	record all balance readings (to same level of precision) and calculated masses with suitable headings and units
	<i>Display of calculation and reasoning</i>	show working in the calculation and use suitable significant figures for the precision of their balance
	<i>Data layout</i>	single table for results and calculated masses
<b>Analysis, conclusions and evaluation</b>	<i>Interpretation of data or observations and identifying sources of error</i>	calculate the mole ratio of Mg : O
	<i>Drawing conclusions</i>	suggest the formula of magnesium oxide from their data
	<i>Suggesting improvements</i>	identify significant sources of error in the experiment, suggest a modification to increase accuracy of the results, and suggest how to extend the investigation to answer a new question

### Method

#### Safety

- Learners must wear safety glasses for this investigation.
- The experiment can be very dangerous if magnesium turnings are used, due to the intense heat produced. Ribbon is much safer in this experiment.
- Good practical techniques when crucibles are used for gravimetric experiments should be discussed. Heat is gentle to start with, to avoid loss of some MgO as smoke. Stronger heat, using a 'cone' flame, is used later to ensure that the reaction is as complete as possible. Cooling should always be carried out with the crucible lid on, to ensure that the residue does not absorb moisture as it cools.

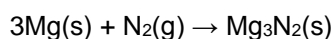
- Learners should be reminded of the hazards of hot apparatus and that the white smoke, MgO, formed is an irritant. Carrying hot crucibles to the balance is unsafe. Also, hot weighing may introduce balance reading errors owing to buoyancy effects, depending on the precision of the balance.
- It is important that as much of the magnesium as possible is oxidised to magnesium oxide, though it is unavoidable that the oxidation occurs more easily on the surface of the metal. Heating to constant mass, as carried out in this experiment, is one way of trying to increase the accuracy of the results obtained.
- Results in this experiment always show a lower uptake of oxygen than expected. This is mainly due to incomplete oxidation (learners should suggest why this is) and also because some magnesium reacts with nitrogen in the air to give magnesium nitride, Mg<sub>3</sub>N<sub>2</sub>.
- Learners should be taught how to calculate the empirical formula of magnesium oxide from their data. In this calculation it is easier to consider oxygen as 'O' atoms rather than as 'O<sub>2</sub>'.
- Learners could use a range of lengths of magnesium to find out whether the percentage error increases or decreases with length. Possible relationships can be discussed before collecting data. (Longer reduces % error in mass but makes it less likely that all the Mg will have reacted on heating.)

## Results

Learners should draw a table for their balance readings and masses of magnesium and oxygen reacting, which includes unambiguous headings and units shown as / g or (g) (as specified in the syllabus).

## Interpretation and evaluation

- The equation  $n = m/A_r$  can be introduced or revised.
- The appropriate number of significant figures can be discussed. Most balance readings would be to 2 decimal places so 3 sf is appropriate when dealing with a mass of Mg < 1 g.
- The need for a whole number ratio in a formula can be discussed. Theoretically this is 1:1 for MgO but often it is not (for reasons considered below) and in this case a sensible ratio should be deduced. An example could be if Mg : O = 1 : 0.66 = 3 : 2, formula is Mg<sub>3</sub>O<sub>2</sub>.
- The reasons for Mg : O being 1 : <1 and how to improve this result can be brainstormed. They include (i) not all the magnesium has reacted, (ii) some of the magnesium oxide escaped as smoke.
- It is easy to suggest a modification to minimise the error due to not all the magnesium reacting: steps 4 to 6 of the method should be repeated until no further increase in mass occurs. The modification must be to the *procedure* and so suggestions involving changes in method of measurement are not appropriate. (Extra apparatus may be selected.)
- As well as reacting with oxygen, magnesium also reacts with nitrogen in the air.



This could lead to a discussion of:

- (1) how you know magnesium nitride does not contain oxygen (the ending 'ide' shows that only the specified elements are present);
- (2) how you know it is a solid (ionic bonding);
- (3) what effect its formation would have on the experimentally derived formula of magnesium oxide (it would lower the ratio of oxygen in the formula);
- (4) whether the magnesium may have reacted with other gases in air and whether any such reaction would be significant in determining the formula.

The presence of magnesium nitride can be detected by adding a few drops of water to the residue in the crucible while it is warm (but not hot) and testing any gas with moist red litmus paper. If any nitride is present, some ammonia gas will be produced, so the litmus will turn blue.

- If the individual results are collected in a table, learners can see whether there is a trend between the length of magnesium ribbon and error in the formula calculated.
- Learners could also plan an experiment to determine the moles of water of crystallisation in one mole of a hydrated salt. ( $\text{MgSO}_4 \cdot x\text{H}_2\text{O}$  or  $\text{BaCl}_2 \cdot x\text{H}_2\text{O}$  [T] or  $\text{CuSO}_4 \cdot x\text{H}_2\text{O}$  [H] [N] could be used.)

They should be encouraged to think about:

- (1) the quantity of hydrated solid used (small amount in crucible – large % errors in mass; large amount in crucible – loss of solid by spitting on initial heating as heated without lid);
- (2) how to minimise spitting of solid (heat gently at first);
- (3) how to make sure all the water is driven off (heat to constant mass);
- (4) how to make sure no moisture from the air is absorbed on cooling (cool with lid on, cool in a desiccator, or cool in a closed container with a drying agent such as calcium chloride).

Care must be taken if hydrated copper(II) sulfate is used, as the anhydrous salt will start to decompose on strong heating to form copper(II) oxide [H] [N] and sulfur trioxide [C]. However, if water is added to the cold anhydrous copper(II) sulfate, the resulting exothermic reaction and colour change shows that the initial reaction is reversible.

### Typical results for the experiment to determine the formula of magnesium oxide:

Mass crucible + lid / g = 22.46

Mass crucible + lid + magnesium / g = 22.66

Final mass crucible + lid + magnesium oxide / g = 22.78

### Calculation

Moles of Mg =  $0.20 / 24.3$  =  $8.23 \times 10^{-3}$  mol

Moles of O =  $0.12 / 16.0$  =  $7.50 \times 10^{-3}$  mol

Mg : O = 1.1 : 1.0

Formula = MgO

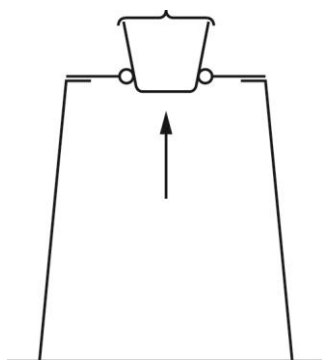
length of Mg ribbon / cm	mole ratio Mg : O
13.5	1.10 : 1.00
etc.	

## Information for technicians








Each learner will require:

- (a) Eye protection
- (b) 1 × porcelain crucible, approximately 15 cm<sup>3</sup>, with lid
- (c) 1 × pipe-clay triangle
- (d) 1 × tongs (suitable to remove crucible lid)
- (e) 1 × Bunsen burner (spirit burners are not suitable)
- (f) 1 × tripod
- (g) 1 × heatproof mat
- [F]** (h) 10–30 cm length of magnesium ribbon (cleaned with sandpaper to remove any surface corrosion)
- (i) 1 × crucible tongs – optional
- (j) Access to a balance reading to 2 dp.  
(If a balance reading to 1 dp is used, the errors in mass will be significantly greater for this procedure.)
- (k) Ruler and scissors

## Diagram of apparatus



## Hazard symbols

	GHS02 (flammable <b>F</b> )		GHS03 (oxidising <b>O</b> )
	GHS05 (corrosive <b>C</b> )		GHS06 (acutely toxic <b>T</b> )
	GHS07 (moderate hazard <b>MH</b> )		GHS08 (health hazard <b>HH</b> )
	GHS09 (hazardous to the aquatic environment <b>N</b> )		

# Worksheet

## Aim

To determine the formula of magnesium oxide by measuring the increase in mass of a piece of magnesium ribbon when heated in air.

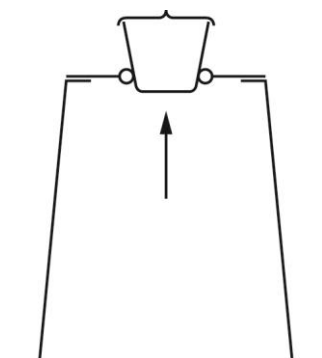
## Method

### Safety

- Wear eye protection.
- The tripod, pipe-clay triangle and gauze remain hot for a considerable time. Use tongs for the hot crucible and take care that all apparatus is cool before you touch it.
- Do not inhale the white smoke that can be given off when magnesium burns.

- (1) Weigh a crucible with its lid. Record your balance reading in a table of results.
- (2) Curl the length of magnesium ribbon loosely so that it fits into the crucible and weigh the crucible + lid + magnesium. Record this mass reading in the table.
- (3) Set up the apparatus for heating the crucible + lid and contents.
- (4) Heat the crucible + lid and contents, gently at first and then more strongly until eventually you are heating it very strongly with the air hole of the Bunsen burner fully open.
- (5) Periodically use tongs to lift the lid to allow air into the crucible. You will see the magnesium burning, with a white flame or glowing red, as it reacts to give magnesium oxide. Stop the oxide escaping as a white smoke by putting the lid back on the crucible.
- (6) When the magnesium no longer burns when the lid is lifted, allow the crucible to cool. (You may wish to lift the crucible off the pipe-clay triangle with crucible tongs and place it on the heat proof mat to cool.)
- (7) When the crucible is cool weigh crucible + lid + magnesium oxide. Record your balance reading in the table.
- (8) If there is time, repeat steps 4 to 7 until there is no further increase in the mass of residue. This is called 'heating to constant mass'.
- (9) Record the new balance reading and calculate the mass of magnesium used and the mass of oxygen that reacted in the table.

## Diagram of apparatus



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## Worksheet, *continued*

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### Results

Record **all** your observations.

Draw a table for your balance readings and masses of magnesium and oxygen reacting, which includes unambiguous headings and units shown as / g or (g) (as specified in the syllabus).

### Interpretation and evaluation

#### Calculation

Use the Periodic Table for any data that you may require.

- (1) Calculate the number of moles of magnesium used.
- (2) Calculate the number of moles of oxygen atoms that reacted with the magnesium.
- (3) Use your answers to 1 and 2 to calculate the formula of magnesium oxide.

#### Points to consider

- (1) To how many significant figures should you calculate the number of moles of each reactant?
- (2) Explain how you used the ratio of the number of moles of Mg and O to deduce the formula of magnesium oxide.
- (3) Explain the advantage of re-heating the residue to constant mass. (Step 8)
- (4) The ratio of Mg:O obtained in this practical nearly always has less oxygen than would be expected.
  - a. Suggest **two** sources of error that could account for this fact.
  - b. Suggest a modification to the experimental procedure that would lower the error due to **one** of the causes you gave in (i). (You may use additional apparatus or increase the number of steps in the method.)
- (5) Magnesium can also react with nitrogen in the air, to give magnesium nitride.
  - a. Write the equation for this reaction.
  - b. Explain why formation of magnesium nitride (instead of magnesium oxide) reduces the expected increase in mass of the product measured in this experiment.
- (6) Others have used different lengths of magnesium ribbon (different masses) from you. Is there any relationship between the length of the magnesium ribbon used and the mole ratio of Mg:O?

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