

CANDIDATE
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CHEMISTRY

9701/31

Paper 3 Advanced Practical Skills 1

May/June 2014

2 hours

Candidates answer on the Question Paper.

Additional Materials: As listed in the Confidential Instructions

READ THESE INSTRUCTIONS FIRST

Write your Centre number, candidate number and name on all the work you hand in.
Give details of the practical session and laboratory where appropriate, in the boxes provided.
Write in dark blue or black pen.
You may use an HB pencil for any diagrams or graphs.
Do not use staples, paper clips, glue or correction fluid.
DO NOT WRITE IN ANY BARCODES.

Answer **all** questions.
Electronic calculators may be used.
You may lose marks if you do not show your working or if you do not use appropriate units.
Use of a Data Booklet is unnecessary.

Qualitative Analysis Notes are printed on pages 10 and 11.

At the end of the examination, fasten all your work securely together.
The number of marks is given in brackets [] at the end of each question or part question.

Session
Laboratory

For Examiner's Use	
1	
2	
3	
Total	

This document consists of **11** printed pages and **1** blank page.



- 1 Hydrogen peroxide, H_2O_2 , is unstable and decomposes to give water and oxygen. In addition to the usual units of concentration, mol dm^{-3} , the concentration of a solution of hydrogen peroxide can also be given in 'volume strength' or 'vol'. For example, in **Question 3** you will use a solution of '20 vol' H_2O_2 . This term means that when 1 dm^3 of this solution is completely decomposed it generates 20 dm^3 of oxygen at room temperature and pressure.

The aim of the following titration is to determine the volume strength of a solution of hydrogen peroxide. To do this you will titrate an acidified solution of hydrogen peroxide with potassium manganate(VII) solution.

FA 1 is $0.0200 \text{ mol dm}^{-3}$ potassium manganate(VII), KMnO_4 .

FA 2 is aqueous hydrogen peroxide, H_2O_2 .

FA 3 is 1.0 mol dm^{-3} sulfuric acid, H_2SO_4 .

(a) Method

- Fill the burette with **FA 1**.
- Pipette 25.0 cm^3 of **FA 2** into the conical flask.
- Use the measuring cylinder to add 25 cm^3 of **FA 3** to the conical flask.
- Run **FA 1** from the burette into the conical flask until the pink colour remains.
- Perform a **rough titration** and record your burette readings in the space below.

The rough titre is cm^3 .

- Carry out as many accurate titrations as you think necessary to obtain consistent results.
- Make certain any recorded results show the precision of your practical work.
- Record, in a suitable form below, all of your burette readings and the volume of **FA 1** added in each accurate titration.

I	
II	
III	
IV	
V	
VI	

[6]

- (b) From your accurate titration results obtain a suitable value to be used in your calculations. Show clearly how you obtained this value.

25.0 cm³ of **FA 2** required cm³ of **FA 1**. [1]

(c) Calculations

Show your working and appropriate significant figures in the final answer to **each** step of your calculations.

- (i) Calculate the number of moles of potassium manganate(VII) present in the volume of **FA 1** calculated in (b).

moles of KMnO_4 = ... mol

- (ii) Use the following equation and your answer to (i) to calculate the number of moles of hydrogen peroxide used in each titration.



moles of H_2O_2 = ... mol

- (iii) Calculate the concentration, in mol dm^{-3} , of H_2O_2 in **FA 2**.

concentration of H_2O_2 = ... mol dm^{-3}

- (iv) Write an equation for the decomposition of hydrogen peroxide to produce oxygen and water.

.....

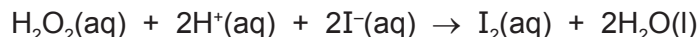
- (v) Calculate the concentration in 'volume strength' of H_2O_2 in **FA 2**. You must assume that the molar volume of oxygen is $24.0 \text{ dm}^3 \text{ mol}^{-1}$ at room temperature and pressure.

I	
II	
III	
IV	
V	

concentration of **FA 2** = ... 'vol'
[5]

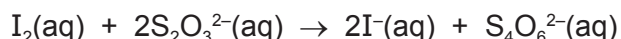
[Total: 12]

- 2 An acidified solution of hydrogen peroxide is able to oxidise iodide ions, $\text{I}^-(\text{aq})$, to iodine, $\text{I}_2(\text{aq})$.



In this experiment, you will investigate how the rate of this reaction depends on the concentration of the hydrogen peroxide and on the concentration of the iodide ions.

The rate of this reaction can be measured by adding thiosulfate ions, $\text{S}_2\text{O}_3^{2-}$, and starch indicator to the mixture. As the iodine is produced, it reacts immediately with the thiosulfate ions and is reduced back to iodide ions.



When all the thiosulfate has reacted, the iodine then turns the starch indicator blue-black. The rate of reaction may be determined by timing how long it takes the reaction mixture to turn blue-black.

FA 2 is aqueous hydrogen peroxide, H_2O_2 .

FA 3 is 1.0 mol dm^{-3} sulfuric acid, H_2SO_4 .

FA 4 is 0.50 mol dm^{-3} potassium iodide, KI .

FA 5 is $0.025 \text{ mol dm}^{-3}$ sodium thiosulfate, $\text{Na}_2\text{S}_2\text{O}_3$.

starch indicator

Read through the instructions carefully and prepare a table for your results before starting any practical work.

(a) Method

Experiment 1

- Use the measuring cylinder to transfer the following volumes into the same 100 cm^3 beaker.
 - 10 cm^3 of **FA 3**
 - 20 cm^3 of **FA 4**
 - 10 cm^3 of **FA 5**
 - 10 cm^3 of distilled water
- Add 10 drops of starch indicator to the beaker.
- Rinse the measuring cylinder with water and shake dry.
- Use the measuring cylinder to measure 10 cm^3 of **FA 2**.
- Add the contents of the measuring cylinder to the beaker and start timing immediately.
- Stir the mixture once and place the beaker on a white tile.
- Stop timing as soon as the solution turns blue-black.
- Record this reaction time to the nearest second.
- Wash out the beaker and the measuring cylinder with water and shake dry.

Experiment 2

- Use the measuring cylinder to transfer the following volumes into the same 100 cm^3 beaker.
 - 10 cm^3 of **FA 3**
 - 20 cm^3 of **FA 4**
 - 10 cm^3 of **FA 5**
- Add 10 drops of starch indicator to the beaker.
- Rinse the measuring cylinder with water and shake dry.
- Use the measuring cylinder to measure 20 cm^3 of **FA 2**.

- Add the contents of the measuring cylinder to the beaker and start timing immediately.
- Stir the mixture once and place the beaker on a white tile.
- Stop timing as soon as the solution turns blue-black.
- Record this reaction time to the nearest second.
- Wash out the beaker and the measuring cylinder with water and shake dry.

Experiment 3

- Use the measuring cylinder to transfer the following volumes into the same 100 cm³ beaker.
 - 10 cm³ of **FA 3**
 - 10 cm³ of **FA 4**
 - 10 cm³ of **FA 5**
 - 10 cm³ of distilled water
- Add 10 drops of starch indicator to the beaker.
- Rinse the measuring cylinder with water and shake dry.
- Use the measuring cylinder to measure 20 cm³ of **FA 2**.
- Add the contents of the measuring cylinder to the beaker and start timing immediately.
- Stir the mixture once and place the beaker on a white tile.
- Stop timing as soon as the solution turns blue-black.
- Record this reaction time to the nearest second.

Record all your results in a single table. You should include the volume of hydrogen peroxide, the volume of potassium iodide, the volume of distilled water and the reaction time. You should also include the 'rate of reaction' which is given by the following expression.

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

I	
II	
III	
IV	
V	
VI	

[6]

(b) From your results, what can you conclude about how the 'rate of reaction' is affected by,

(i) the concentration of hydrogen peroxide,

.....

(ii) the concentration of potassium iodide?

.....

[1]

- (c) It is not possible to draw a reliable conclusion about the effect of changing the concentration of a reagent on the 'rate of reaction' from only two experiments.

Suggest **three** experiments that could be carried out in addition to **Experiments 2** and **3** to investigate the effect of changing the concentration of **FA 4**, potassium iodide. In each case give the volumes of each solution/liquid that would be used.

Do not carry out these experiments.

[2]

- (d) A student was unable to complete all three experiments on the same day but had to return to the investigation the following morning. The student noticed that the results were significantly different.

Give **two** possible reasons for these differences and explain how the student could have overcome these problems.

.....

 [2]

- (e) There are many sources of error in these experiments. In the following, assume that the only error involves measuring the reaction time.

- (i) If the error in each recorded time is ± 1 s, calculate the percentage error in the time recorded in **Experiment 1**.

percentage error = ... %

- (ii) Another student decided to repeat **Experiment 1** but used $0.050 \text{ mol dm}^{-3}$ sodium thiosulfate in place of **FA 5**.

How would this change the percentage error in the recorded time? Explain your answer.

.....

 [2]

[Total: 13]

3 Qualitative Analysis

At each stage of any test you are to record details of the following.

- colour changes seen
- the formation of any precipitate
- the solubility of such precipitates in an excess of the reagent added

Where gases are released they should be identified by a test, **described in the appropriate place in your observations.**

You should indicate clearly at what stage in a test a change occurs.

Marks are **not** given for chemical equations.

No additional tests for ions present should be attempted.

If any solution is warmed, a boiling tube MUST be used.

Rinse and reuse test-tubes and boiling tubes where possible.

Where reagents are selected for use in a test, the name or correct formula of the element or compound must be given.

(a) **FA 6** is a solution of two different salts. It contains two different cations, one of which is listed in the Qualitative Analysis Notes on page 10. It contains two anions, both of which are listed in the Qualitative Analysis Notes on page 11.

- (i) Choose reagents that will allow you to identify one of the cations. Carry out suitable tests using these reagents and record your results in the space below.

I	
II	
III	
IV	
V	

One of the cations in **FA 6** is

(ii) Carry out the following tests to identify the two anions present in **FA 6**.

<i>test</i>	<i>observations</i>
To a 1 cm depth of FA 6 in a test-tube add a 1 cm depth of aqueous silver nitrate, then	
add aqueous ammonia.	
To a 1 cm depth of FA 6 in a test-tube add a 1 cm depth of aqueous barium chloride (or aqueous barium nitrate), then	
add dilute nitric acid.	

The anions in **FA 6** are and

[9]

VI	
VII	
VIII	
IX	

(b) **FA 7** is an acidified solution of iron(II) sulfate, $\text{FeSO}_4(\text{aq})$.

Carry out the following tests and record your observations.

<i>test</i>	<i>observations</i>
(i) To a 1 cm depth of FA 7 in a test-tube add aqueous sodium hydroxide and leave for a few minutes.	
(ii) To a 1 cm depth of FA 7 in a boiling tube add a 1 cm depth of dilute sulfuric acid followed by a 1 cm depth of '20 vol' hydrogen peroxide. Stir the mixture, then	
(iii) pour a 1 cm depth of the mixture into a clean boiling tube and add a 3 cm depth of aqueous sodium hydroxide.	

I	
II	
III	
IV	
V	
VI	

(iv) What type of reaction takes place in (ii)?

.....

.....

(v) Explain your observations in (iii).

.....

.....

[6]

[Total: 15]

Qualitative Analysis Notes

Key: [ppt. = precipitate]

1 Reactions of aqueous cations

ion	reaction with	
	NaOH(aq)	NH ₃ (aq)
aluminium, Al ³⁺ (aq)	white ppt. soluble in excess	white ppt. insoluble in excess
ammonium, NH ₄ ⁺ (aq)	no ppt. ammonia produced on heating	—
barium, Ba ²⁺ (aq)	no ppt. (if reagents are pure)	no ppt.
calcium, Ca ²⁺ (aq)	white ppt. with high [Ca ²⁺ (aq)]	no ppt.
chromium(III), Cr ³⁺ (aq)	grey-green ppt. soluble in excess giving dark green solution	grey-green ppt. insoluble in excess
copper(II), Cu ²⁺ (aq)	pale blue ppt. insoluble in excess	blue ppt. soluble in excess giving dark blue solution
iron(II), Fe ²⁺ (aq)	green ppt. turning brown on contact with air insoluble in excess	green ppt. turning brown on contact with air insoluble in excess
iron(III), Fe ³⁺ (aq)	red-brown ppt. insoluble in excess	red-brown ppt. insoluble in excess
magnesium, Mg ²⁺ (aq)	white ppt. insoluble in excess	white ppt. insoluble in excess
manganese(II), Mn ²⁺ (aq)	off-white ppt. rapidly turning brown on contact with air insoluble in excess	off-white ppt. rapidly turning brown on contact with air insoluble in excess
zinc, Zn ²⁺ (aq)	white ppt. soluble in excess	white ppt. soluble in excess

2 Reactions of anions

<i>ion</i>	<i>reaction</i>
carbonate, CO_3^{2-}	CO_2 liberated by dilute acids
chloride, $\text{Cl}^-(\text{aq})$	gives white ppt. with $\text{Ag}^+(\text{aq})$ (soluble in $\text{NH}_3(\text{aq})$);
bromide, $\text{Br}^-(\text{aq})$	gives cream ppt. with $\text{Ag}^+(\text{aq})$ (partially soluble in $\text{NH}_3(\text{aq})$);
iodide, $\text{I}^-(\text{aq})$	gives yellow ppt. with $\text{Ag}^+(\text{aq})$ (insoluble in $\text{NH}_3(\text{aq})$);
nitrate, $\text{NO}_3^-(\text{aq})$	NH_3 liberated on heating with $\text{OH}^-(\text{aq})$ and Al foil
nitrite, $\text{NO}_2^-(\text{aq})$	NH_3 liberated on heating with $\text{OH}^-(\text{aq})$ and Al foil; NO liberated by dilute acids (colourless $\text{NO} \rightarrow$ (pale) brown NO_2 in air)
sulfate, $\text{SO}_4^{2-}(\text{aq})$	gives white ppt. with $\text{Ba}^{2+}(\text{aq})$ (insoluble in excess dilute strong acids)
sulfite, $\text{SO}_3^{2-}(\text{aq})$	SO_2 liberated with dilute acids; gives white ppt. with $\text{Ba}^{2+}(\text{aq})$ (soluble in excess dilute strong acids)

3 Tests for gases

<i>gas</i>	<i>test and test result</i>
ammonia, NH_3	turns damp red litmus paper blue
carbon dioxide, CO_2	gives a white ppt. with limewater (ppt. dissolves with excess CO_2)
chlorine, Cl_2	bleaches damp litmus paper
hydrogen, H_2	“pops” with a lighted splint
oxygen, O_2	relights a glowing splint
sulfur dioxide, SO_2	turns acidified aqueous potassium manganate(VII) from purple to colourless

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CHEMISTRY

9701/32

Paper 3 Advanced Practical Skills 2

May/June 2014

2 hours

Candidates answer on the Question Paper.

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Session
Laboratory

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Total	

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- 1 Carbon dioxide in the atmosphere dissolves in rain water making it acidic. When this water passes through limestone containing calcium carbonate, it forms solutions of calcium hydrogen carbonate.

In this experiment, you will determine the concentration of hydrogen carbonate ions in a solution by titration with hydrochloric acid.

FB 1 is $0.100 \text{ mol dm}^{-3}$ hydrochloric acid, HCl .

FB 2 contains an unknown concentration of hydrogen carbonate ions, HCO_3^- .
methyl orange indicator

(a) Method

- Fill the burette with **FB 1**.
- Pipette 25.0 cm^3 of **FB 2** into a conical flask.
- Add a few drops of methyl orange indicator to the conical flask.
- Perform a **rough titration** and record your burette readings in the space below.

The rough titre is cm^3 .

- Carry out as many accurate titrations as you think necessary to obtain consistent results.
- Make certain any recorded results show the precision of your practical work.
- Record, in a suitable form below, all of your burette readings and the volume of **FB 1** added in each accurate titration.

I	
II	
III	
IV	
V	
VI	
VII	

[7]

- (b) From your accurate titration results obtain a suitable value to be used in your calculations. Show clearly how you obtained this value.

25.0 cm³ of **FB 2** required cm³ of **FB 1**. [1]

(c) Calculations

Show your working and appropriate significant figures in the final answer to **each** step of your calculations.

- (i) Calculate the number of moles of hydrochloric acid present in the volume of **FB 1** calculated in (b).

moles of HCl = ... mol

- (ii) Write the **ionic** equation for the reaction of **FB 1** with **FB 2**.

.....

- (iii) Calculate the concentration, in mol dm⁻³, of hydrogen carbonate ions in **FB 2**.

I	
II	

concentration of HCO₃⁻ in **FB 2** = ... mol dm⁻³
[2]

[Total: 10]

- 2 As well as hydrogen carbonate ions, water that has passed through rocks also contains a range of metal ions such as Ca^{2+} . The presence of Ca^{2+} ions leads to the water forming scum with certain types of soap. Soluble metal carbonates can be added to remove these ions. One such soluble carbonate is $\text{M}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$.

In this experiment you will heat a sample of the carbonate salt to remove the water of crystallisation and leave the anhydrous salt, M_2CO_3 . By working out how much water was lost you will be able to calculate the relative formula mass of $\text{M}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$ and hence identify **M**.

FB 3 is the hydrated salt, $\text{M}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$.

(a) Method

- Weigh a clean, dry crucible and its lid.
- Transfer all the **FB 3** into the crucible and put the crucible lid on.
- Reweigh the crucible, the lid and the contents.
- Place the crucible on the pipe-clay triangle.
- Heat the crucible very gently for 5 minutes.
- Remove the lid and heat strongly for a further 5 minutes.
- Allow the crucible to cool.
You should start **Question 3** while cooling is taking place.
- When the crucible is cool enough to handle, reweigh the crucible, the lid and the contents.

Record, in an appropriate form in the space below, all your weighings and calculations including the mass of **FB 3** used and the mass of water lost.

I	
II	
III	
IV	

[4]

(b) Calculations

Show your working and appropriate significant figures in the final answer to **each** step of your calculations.

- (i) Use the mass of water lost on heating to calculate the number of moles of hydrated salt, $\text{M}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$, in the initial sample.
[A_r: H, 1.0; O, 16.0]

moles of $\text{M}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$ = ... mol

(ii) Calculate the relative formula mass of $M_2CO_3 \cdot 10H_2O$, **FB 3**.

relative formula mass of $M_2CO_3 \cdot 10H_2O$ = ...

(iii) Identify **M** in the salt, **FB 3**.

[A_r: H, 1.0; Li, 6.9; C, 12.0; O, 16.0; Na, 23.0; K, 39.1; Rb, 85.5; Cs, 133]

I	
II	
III	
IV	
V	

M is
[5]

(c) (i) State the maximum error in the mass of **FB 3** you recorded in (a).

maximum error in the mass of **FB 3** = ... g

(ii) Calculate the maximum percentage error in the mass of water lost in (a).

percentage error = ...%
[1]

(d) Suggest a change to the method that would improve the accuracy of this experiment. Explain your answer.

.....

 [2]

[Total: 12]

I	
II	
III	

3 Qualitative Analysis

At each stage of any test you are to record details of the following.

- colour changes seen
- the formation of any precipitate
- the solubility of such precipitates in an excess of the reagent added

Where gases are released they should be identified by a test, **described in the appropriate place in your observations.**

You should indicate clearly at what stage in a test a change occurs.

Marks are **not** given for chemical equations.

No additional tests for ions present should be attempted.

If any solution is warmed, a boiling tube MUST be used.

Rinse and reuse test-tubes and boiling tubes where possible.

Where reagents are selected for use in a test, the name or correct formula of the element or compound must be given.

- (a) Water in rivers and streams contains a range of different cations and anions. A particular salt occurs in some samples of spring water. You will carry out tests to identify this salt.

FB 4 is a solution of the salt. It contains one cation, which is listed in the Qualitative Analysis Notes on page 10. It also contains one anion, which is either sulfate, SO_4^{2-} , or sulfite, SO_3^{2-} .

- (i) Choose reagents that will allow you to identify the cation in **FB 4**. Carry out suitable tests using these reagents and record your observations in the space below.

The cation in **FB 4** is

- (ii) Choose reagents to identify the anion in **FB 4**. Carry out suitable tests using these reagents and record your results in the space below.

I	
II	
III	
IV	
V	
VI	
VII	
VIII	

The anion in **FB 4** is

[8]

Before starting parts (b) and (c), half-fill a 250 cm³ beaker with water and heat with a Bunsen burner to approximately 60 °C. You will use this as a hot water bath.
Turn off the Bunsen burner.

- (b) **FB 5, FB 6** and **FB 7** are solutions each containing a single compound which could be ethanol, ethanal or propanone. To identify each compound you will react the samples with Tollens' reagent and with acidified potassium manganate(VII).

(i) **Preparation of Tollens' reagent**

- To approximately 2 cm depth of aqueous silver nitrate in a boiling tube, add approximately 0.5 cm depth of aqueous sodium hydroxide.
- Add aqueous ammonia a little at a time with continuous shaking until the brown precipitate **just** dissolves. Do not add an excess of ammonia.

test	observations		
	FB 5	FB 6	FB 7
To a 1 cm depth of each solution in a clean, dry test-tube add a few drops of the Tollens' reagent that you have prepared. Do not shake the tube. If no reaction is seen, warm the tube in the hot water bath.			
When you have made your observations, rinse out all the test-tubes and the boiling tube which were used with Tollens' reagent.			
To a 1 cm depth of each solution in a test-tube add a 1 cm depth of dilute sulfuric acid. Then add a few drops of aqueous potassium manganate(VII). If no reaction is seen, warm the tube in the hot water bath.	Do not carry out this test		

- (ii) Identify each compound.

FB contains ethanol.

FB contains ethanal.

FB contains propanone.

I	
II	
III	
IV	
V	

- (iii) Another reagent can be used with these samples to identify which compounds have a carbonyl group, C=O. This reagent has not been supplied so you cannot carry out this test. State the reagent and the expected observations of the three tests.

reagent:

expected observations

FB 5

FB 6

FB 7

[7]

- (c) **FB 8** is an aqueous solution of an organic compound. Carry out the following tests. You do not need to identify **FB 8**.

<i>test</i>	<i>observations</i>
To a 1 cm depth of FB 8 in a test-tube add a 1 cm depth of dilute sulfuric acid. Then add a few drops of aqueous potassium manganate(VII). If no reaction is seen, place the test-tube in the hot water bath and leave to stand.	
To a 1 cm depth of FB 8 in a test-tube carefully add a small spatula measure of sodium hydrogen carbonate.	

[3]

[Total: 18]

Qualitative Analysis Notes

Key: [ppt. = precipitate]

1 Reactions of aqueous cations

ion	reaction with	
	NaOH(aq)	NH ₃ (aq)
aluminium, Al ³⁺ (aq)	white ppt. soluble in excess	white ppt. insoluble in excess
ammonium, NH ₄ ⁺ (aq)	no ppt. ammonia produced on heating	—
barium, Ba ²⁺ (aq)	no ppt. (if reagents are pure)	no ppt.
calcium, Ca ²⁺ (aq)	white ppt. with high [Ca ²⁺ (aq)]	no ppt.
chromium(III), Cr ³⁺ (aq)	grey-green ppt. soluble in excess giving dark green solution	grey-green ppt. insoluble in excess
copper(II), Cu ²⁺ (aq)	pale blue ppt. insoluble in excess	blue ppt. soluble in excess giving dark blue solution
iron(II), Fe ²⁺ (aq)	green ppt. turning brown on contact with air insoluble in excess	green ppt. turning brown on contact with air insoluble in excess
iron(III), Fe ³⁺ (aq)	red-brown ppt. insoluble in excess	red-brown ppt. insoluble in excess
magnesium, Mg ²⁺ (aq)	white ppt. insoluble in excess	white ppt. insoluble in excess
manganese(II), Mn ²⁺ (aq)	off-white ppt. rapidly turning brown on contact with air insoluble in excess	off-white ppt. rapidly turning brown on contact with air insoluble in excess
zinc, Zn ²⁺ (aq)	white ppt. soluble in excess	white ppt. soluble in excess

2 Reactions of anions

<i>ion</i>	<i>reaction</i>
carbonate, CO_3^{2-}	CO_2 liberated by dilute acids
chloride, $\text{Cl}^-(\text{aq})$	gives white ppt. with $\text{Ag}^+(\text{aq})$ (soluble in $\text{NH}_3(\text{aq})$);
bromide, $\text{Br}^-(\text{aq})$	gives cream ppt. with $\text{Ag}^+(\text{aq})$ (partially soluble in $\text{NH}_3(\text{aq})$);
iodide, $\text{I}^-(\text{aq})$	gives yellow ppt. with $\text{Ag}^+(\text{aq})$ (insoluble in $\text{NH}_3(\text{aq})$);
nitrate, $\text{NO}_3^-(\text{aq})$	NH_3 liberated on heating with $\text{OH}^-(\text{aq})$ and Al foil
nitrite, $\text{NO}_2^-(\text{aq})$	NH_3 liberated on heating with $\text{OH}^-(\text{aq})$ and Al foil; NO liberated by dilute acids (colourless $\text{NO} \rightarrow$ (pale) brown NO_2 in air)
sulfate, $\text{SO}_4^{2-}(\text{aq})$	gives white ppt. with $\text{Ba}^{2+}(\text{aq})$ (insoluble in excess dilute strong acids)
sulfite, $\text{SO}_3^{2-}(\text{aq})$	SO_2 liberated with dilute acids; gives white ppt. with $\text{Ba}^{2+}(\text{aq})$ (soluble in excess dilute strong acids)

3 Tests for gases

<i>gas</i>	<i>test and test result</i>
ammonia, NH_3	turns damp red litmus paper blue
carbon dioxide, CO_2	gives a white ppt. with limewater (ppt. dissolves with excess CO_2)
chlorine, Cl_2	bleaches damp litmus paper
hydrogen, H_2	“pops” with a lighted splint
oxygen, O_2	relights a glowing splint
sulfur dioxide, SO_2	turns acidified aqueous potassium manganate(VII) from purple to colourless

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CANDIDATE
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CHEMISTRY

9701/33

Paper 3 Advanced Practical Skills 1

May/June 2014

2 hours

Candidates answer on the Question Paper.

Additional Materials: As listed in the Confidential Instructions

READ THESE INSTRUCTIONS FIRST

Write your Centre number, candidate number and name on all the work you hand in.
Give details of the practical session and laboratory where appropriate, in the boxes provided.
Write in dark blue or black pen.
You may use an HB pencil for any diagrams or graphs.
Do not use staples, paper clips, glue or correction fluid.
DO NOT WRITE IN ANY BARCODES.

Answer **all** questions.
Electronic calculators may be used.
You may lose marks if you do not show your working or if you do not use appropriate units.
Use of a Data Booklet is unnecessary.

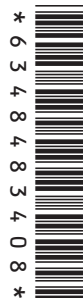
Qualitative Analysis Notes are printed on pages 10 and 11.

At the end of the examination, fasten all your work securely together.
The number of marks is given in brackets [] at the end of each question or part question.

Session
Laboratory

For Examiner's Use	
1	
2	
3	
Total	

This document consists of **11** printed pages and **1** blank page.



- 1 You are to determine, by titration, the change in oxidation number of a transition metal ion, M^{2+} , when reacted with acidified potassium manganate(VII).

FA 1 is $0.0200 \text{ mol dm}^{-3}$ potassium manganate(VII), KMnO_4 .

FA 2 is $0.0530 \text{ mol dm}^{-3}$ transition metal salt, MSO_4 .

FA 3 is 1.0 mol dm^{-3} sulfuric acid, H_2SO_4 .

(a) Method

- Fill the burette with **FA 1**.
- Pipette 25.0 cm^3 of **FA 2** into the conical flask.
- Use the measuring cylinder to add 25 cm^3 of **FA 3** into the conical flask.
- Carry out a **rough titration** and record your burette readings in the space below. Add **FA 1** until the contents of the flask turn a permanent pale pink colour.

The rough titre is cm^3 .

- Carry out as many accurate titrations as you think necessary to obtain consistent results.
- Make certain any recorded results show the precision of your practical work.
- Record below, in a suitable form, all of your burette readings and the volume of **FA 1** added in each accurate titration.

I	
II	
III	
IV	
V	
VI	
VII	

[7]

- (b)** From your accurate titration results, obtain a suitable value to be used in your calculations. Show clearly how you have obtained this value.

25.0 cm^3 of **FA 2** required cm^3 of **FA 1**. [1]

(c) Calculations

Show your working and appropriate significant figures in the final answer to **each** step of your calculations.

- (i) Calculate the number of moles of potassium manganate(VII) present in the volume of **FA 1** calculated in (b).

moles of $\text{KMnO}_4 = \dots\dots\dots$ mol

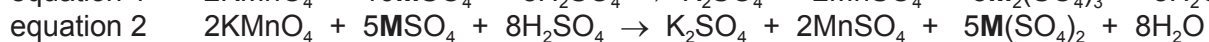
- (ii) Calculate the number of moles of MSO_4 in 25.0 cm^3 of **FA 2**.

moles of MSO_4 in $25.0\text{ cm}^3 = \dots\dots\dots$ mol

- (iii) Use your answers to (i) and (ii) to calculate the number of moles of MSO_4 that react with 1 mole of KMnO_4 .

moles of $\text{MSO}_4 = \dots\dots\dots$ mol

- (iv) Two possible equations for the reaction of acidified KMnO_4 with MSO_4 are below.



State and explain which of these two equations is consistent with your answer to (iii).

.....

- (v) Use your answer to (iv) to state the oxidation number of the transition metal **M** in the product of the reaction.

.....

[5]

[Total: 13]

I	
II	
III	
IV	
V	

- 2 You will determine the enthalpy change, ΔH , for the reaction between magnesium and dilute sulfuric acid. The equation for the reaction is given below.



FA 3 is 1.00 mol dm⁻³ sulfuric acid, H₂SO₄.

two different coiled lengths of magnesium ribbon, Mg.

(a) Method

Read through the method **before** starting any practical work and prepare a table for your results in the space below.

- Weigh the shorter piece of magnesium ribbon and record its mass.
- Support the plastic cup in the 250 cm³ beaker.
- Use the measuring cylinder to transfer 50 cm³ of **FA 3** into the plastic cup.
- Place the thermometer in the **FA 3** in the plastic cup and record the initial temperature.
- Add the shorter piece of magnesium ribbon into the plastic cup. Ensure that all of the magnesium is in contact with the acid. (**Care**: acid spray may occur.)
- Stir the mixture and record the maximum temperature.
- Empty and rinse the plastic cup. Shake out any excess water.
- Repeat the experiment using the longer piece of magnesium ribbon and record all your data.

Results

[4]

(b) Calculations

Show your working and appropriate significant figures in the final answer to **each** step of your calculations.

- (i) Show by calculation that the sulfuric acid, **FA 3**, was used in excess in both experiments.
(*A_r*: Mg, 24.3)

- (ii) State an observation which confirms that the sulfuric acid, **FA 3**, was in excess.

.....

- (iii) Calculate the heat energy produced when the **shorter** piece of magnesium was added to **FA 3**.

(Assume that 4.3 J of heat energy changes the temperature of 1.0 cm³ of solution by 1.0 °C.)

heat energy produced = ... J

- (iv) Calculate the enthalpy change, in kJ mol⁻¹, for the reaction between the **shorter** piece of magnesium and the sulfuric acid.

enthalpy change = ... kJ mol⁻¹
(sign) (value)

- (v) Calculate the heat energy produced when the **longer** piece of magnesium was added to **FA 3**.

(Assume that 4.3 J of heat energy changes the temperature of 1.0 cm³ of solution by 1.0 °C.)

heat energy produced = ... J

- (vi) Calculate the enthalpy change, in kJ mol⁻¹, for the reaction between the **longer** piece of magnesium and the sulfuric acid.

enthalpy change = ... kJ mol⁻¹
(sign) (value)

[5]

- (c) (i) What is the maximum error in a reading of the thermometer used in this experiment?

maximum error = ... °C.

- (ii) Which of your temperature changes has the higher percentage error?

.....

- (iii) Calculate this maximum percentage error.

maximum percentage error in the temperature change = ... %
[1]

- (d) Apart from errors due to heat loss and thermometer readings, suggest another significant source of error in this experiment. State what improvement could be made to the procedure to reduce this error.

.....

.....

..... [2]

[Total: 12]

3 Qualitative Analysis

At each stage of any test you are to record details of the following.

- colour changes seen
- the formation of any precipitate
- the solubility of such precipitates in an excess of the reagent added

Where gases are released they should be identified by a test, **described in the appropriate place in your observations.**

You should indicate clearly at what stage in a test a change occurs.

Marks are **not** given for chemical equations.

No additional tests for ions present should be attempted.

If any solution is warmed, a boiling tube MUST be used.

Rinse and reuse test-tubes and boiling tubes where possible.

Where reagents are selected for use in a test, the name or correct formula of the element or compound must be given.

(a) **FA 4, FA 5** and **FA 6** are solutions, each containing one transition metal ion. One of the solutions also contains the ammonium ion. All the cations present are listed in the Qualitative Analysis Notes on page 10.

(i) Carry out the following tests on the three solutions.

		<i>test</i>	<i>observations</i>
I		To a 1 cm depth of FA 4 in a test-tube, add FA 1 , aqueous potassium manganate(VII), dropwise.	
II			
III			
IV		To a 1 cm depth of FA 5 in a test-tube, add FA 1 , aqueous potassium manganate(VII), dropwise.	
		To a 1 cm depth of FA 6 in a test-tube, add FA 1 , aqueous potassium manganate(VII), dropwise.	

(ii) State which solution(s) contain ions which have been oxidised.

..... [4]

- (b) (i) Select a reagent or reagents to identify **all** the cations present in the three solutions.

reagent(s)

Carry out experiments using your reagent(s) on each of **FA 4**, **FA 5** and **FA 6** and record your observations in a suitable form in the space below.

I	
II	
III	
IV	
V	
VI	
VII	
VIII	

- (ii) Use your observations to identify the cations present in the three solutions.

FA 4 contains

FA 5 contains

FA 6 contains

[8]

(c) Each of the solutions **FA 4**, **FA 5** and **FA 6** contains either a chloride or a sulfate ion.

(i) Choose a reagent or reagents to identify which solution(s) contain **chloride** ions.

reagent(s)

Use your reagent(s) to carry out a test on each of **FA 4**, **FA 5** and **FA 6** and record your results in the space below.

(ii) State which solution(s) contain a chloride ion.

..... [3]

[Total: 15]

Qualitative Analysis Notes

Key: [ppt. = precipitate]

1 Reactions of aqueous cations

ion	reaction with	
	NaOH(aq)	NH ₃ (aq)
aluminium, Al ³⁺ (aq)	white ppt. soluble in excess	white ppt. insoluble in excess
ammonium, NH ₄ ⁺ (aq)	no ppt. ammonia produced on heating	—
barium, Ba ²⁺ (aq)	no ppt. (if reagents are pure)	no ppt.
calcium, Ca ²⁺ (aq)	white ppt. with high [Ca ²⁺ (aq)]	no ppt.
chromium(III), Cr ³⁺ (aq)	grey-green ppt. soluble in excess giving dark green solution	grey-green ppt. insoluble in excess
copper(II), Cu ²⁺ (aq)	pale blue ppt. insoluble in excess	blue ppt. soluble in excess giving dark blue solution
iron(II), Fe ²⁺ (aq)	green ppt. turning brown on contact with air insoluble in excess	green ppt. turning brown on contact with air insoluble in excess
iron(III), Fe ³⁺ (aq)	red-brown ppt. insoluble in excess	red-brown ppt. insoluble in excess
magnesium, Mg ²⁺ (aq)	white ppt. insoluble in excess	white ppt. insoluble in excess
manganese(II), Mn ²⁺ (aq)	off-white ppt. rapidly turning brown on contact with air insoluble in excess	off-white ppt. rapidly turning brown on contact with air insoluble in excess
zinc, Zn ²⁺ (aq)	white ppt. soluble in excess	white ppt. soluble in excess

2 Reactions of anions

<i>ion</i>	<i>reaction</i>
carbonate, CO_3^{2-}	CO_2 liberated by dilute acids
chloride, $\text{Cl}^-(\text{aq})$	gives white ppt. with $\text{Ag}^+(\text{aq})$ (soluble in $\text{NH}_3(\text{aq})$);
bromide, $\text{Br}^-(\text{aq})$	gives cream ppt. with $\text{Ag}^+(\text{aq})$ (partially soluble in $\text{NH}_3(\text{aq})$);
iodide, $\text{I}^-(\text{aq})$	gives yellow ppt. with $\text{Ag}^+(\text{aq})$ (insoluble in $\text{NH}_3(\text{aq})$);
nitrate, $\text{NO}_3^-(\text{aq})$	NH_3 liberated on heating with $\text{OH}^-(\text{aq})$ and Al foil
nitrite, $\text{NO}_2^-(\text{aq})$	NH_3 liberated on heating with $\text{OH}^-(\text{aq})$ and Al foil; NO liberated by dilute acids (colourless $\text{NO} \rightarrow$ (pale) brown NO_2 in air)
sulfate, $\text{SO}_4^{2-}(\text{aq})$	gives white ppt. with $\text{Ba}^{2+}(\text{aq})$ (insoluble in excess dilute strong acids)
sulfite, $\text{SO}_3^{2-}(\text{aq})$	SO_2 liberated with dilute acids; gives white ppt. with $\text{Ba}^{2+}(\text{aq})$ (soluble in excess dilute strong acids)

3 Tests for gases

<i>gas</i>	<i>test and test result</i>
ammonia, NH_3	turns damp red litmus paper blue
carbon dioxide, CO_2	gives a white ppt. with limewater (ppt. dissolves with excess CO_2)
chlorine, Cl_2	bleaches damp litmus paper
hydrogen, H_2	“pops” with a lighted splint
oxygen, O_2	relights a glowing splint
sulfur dioxide, SO_2	turns acidified aqueous potassium manganate(VII) from purple to colourless

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CHEMISTRY

9701/34

Paper 3 Advanced Practical Skills 2

May/June 2014

2 hours

Candidates answer on the Question Paper.

Additional Materials: As listed in the Confidential Instructions

READ THESE INSTRUCTIONS FIRST

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Give details of the practical session and laboratory where appropriate, in the boxes provided.
Write in dark blue or black pen.
You may use an HB pencil for any diagrams or graphs.
Do not use staples, paper clips, glue or correction fluid.
DO NOT WRITE IN ANY BARCODES.

Answer **all** questions.
Electronic calculators may be used.
You may lose marks if you do not show your working or if you do not use appropriate units.
Use of a Data Booklet is unnecessary.

Qualitative Analysis Notes are printed on pages 11 and 12.

At the end of the examination, fasten all your work securely together.
The number of marks is given in brackets [] at the end of each question or part question.

Session
Laboratory

For Examiner's Use	
1	
2	
3	
Total	

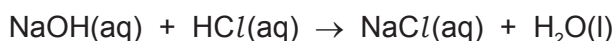
This document consists of **12** printed pages.



- 1 You are to determine the percentage of calcium carbonate in a sample of crushed limestone. You will first react a known mass of the crushed limestone in a known amount of hydrochloric acid, HCl(aq) , to make a solution. You may assume that only the calcium carbonate present in the sample will react with the acid.



The amount of acid that did not react with the carbonate is then found by a titration using sodium hydroxide. You may assume that no compounds present in the limestone will react with the sodium hydroxide.



FB 1 is crushed limestone, impure calcium carbonate.

FB 2 is 2.0 mol dm^{-3} hydrochloric acid, HCl .

FB 4 is 0.20 mol dm^{-3} sodium hydroxide, NaOH .
methyl orange indicator

(a) Method

Read through the method before starting any practical work.

Making the solution

- Weigh the container with the limestone, **FB 1**, and record the mass below.
- Tip all the solid **FB 1** into a 250 cm^3 beaker.
- Reweigh the container and record the mass.
- Fill the burette with **FB 2**.
- Slowly run between 47.5 and 48.5 cm^3 of **FB 2** into the beaker containing **FB 1**.
- Record, in the space below, both your burette readings and the volume of **FB 2** added.
- Stir the mixture carefully until all the solid has reacted.
- Transfer the contents of the beaker into the volumetric flask.
- Rinse the beaker with distilled water and add it to the volumetric flask. Make the solution up to 250 cm^3 with distilled water and mix thoroughly. This is solution **FB 3**.

Titration

- Empty and rinse the burette with distilled water.
- Fill the burette with **FB 3** from the volumetric flask.
- Pipette 25.0 cm³ of **FB 4** into a conical flask.
- Add a few drops of methyl orange indicator.
- Perform a **rough titration** and record your burette readings in the space below.

The rough titre is cm³.

- Carry out as many accurate titrations as you think necessary to obtain consistent results.
- Make certain any recorded results show the precision of your practical work.
- Record, in a suitable form below, all of your burette readings and the volume of **FB 3** added in each accurate titration.

I	
II	
III	
IV	
V	
VI	
VII	
VIII	
IX	

[9]

- (b) From your accurate titration results, obtain a suitable value to be used in your calculations. Show clearly how you have obtained this value.

25.0 cm³ of **FB 4** required cm³ of **FB 3**. [1]

(c) Calculations

Show your working and appropriate significant figures in the final answer to **each** step of your calculations.

- (i) Calculate the number of moles of sodium hydroxide present in 25.0 cm³ of **FB 4**.

moles of NaOH = ... mol

- (ii) Hence state the number of moles of hydrochloric acid present in the volume of **FB 3** calculated in (b).

moles of HCl = ... mol

- (iii) Use your answer to (ii) to calculate the number of moles of hydrochloric acid present in 250 cm³ of **FB 3**.

moles of HCl in 250 cm³ of **FB 3** = ... mol

- (iv) Calculate the number of moles of hydrochloric acid, **FB 2**, added to **FB 1** in (a).

moles of HCl added to **FB 1** = ... mol

- (v) Use your answers to (iii) and (iv) to calculate the number of moles of hydrochloric acid that reacted with the calcium carbonate in **FB 1**.

I	
II	
III	
IV	
V	
VI	
VII	

moles of HCl reacted with CaCO₃ = ... mol

- (vi) Calculate the number of moles of calcium carbonate present in your sample of **FB 1**.

moles of CaCO₃ = ... mol

- (vii) From your answer to (vi) and the mass of **FB 1** used in (a), calculate the percentage by mass of calcium carbonate in the limestone.
[A_r: C, 12.0; O, 16.0; Ca, 40.1]

percentage of calcium carbonate = ... %
[7]

- (d) (i) The maximum error in a single burette reading is $\pm 0.05 \text{ cm}^3$.
Student X, carrying out this experiment, recorded that 48.50 cm^3 of **FB 2** was added to **FB 1**.
What are the smallest and largest possible volumes of **FB 2** that were added?

smallest volume used = ... cm^3

largest volume used = ... cm^3

- (ii) Student Y used an identical mass of **FB 1** but added 47.70 cm^3 of **FB 2**. How would the value obtained in (b) by student X compare with the value obtained by student Y?
Explain your answer.

.....
.....
.....

[3]

[Total: 20]

- 2 You are to determine the percentage by mass of calcium carbonate in another sample of the limestone by thermal decomposition. You may assume that none of the other compounds in the limestone sample is affected by heating. The equation for the reaction that occurs is given below.



FB 5 is crushed limestone, impure calcium carbonate.

(a) Method

Read through the method **before** starting any practical work and prepare a table for your results in the space below.

- Weigh the empty crucible and record the mass in your table.
- Transfer **all** the **FB 5** into the crucible.
- Weigh the crucible with **FB 5** and record the mass.
- Place the crucible on the pipe-clay triangle.
- Heat the crucible gently for about one minute and then strongly for three minutes.
- Remove the Bunsen burner and allow the crucible to cool.
- While the crucible is cooling start working on another question.
- Reweigh the cooled crucible with contents and record the mass.
- Reheat the crucible strongly for three minutes, cool and reweigh. Record the mass.
- Record the mass of **FB 5** used and the mass of solid remaining after the second heating.

[2]

(b) Calculations

Show your working and appropriate significant figures in the final answer to **each** step of your calculations.

- (i) From your results in **(a)**, calculate the total mass of carbon dioxide lost on heating **FB 5**.

mass of CO_2 lost = ... g

- (ii) Use your answer to **(i)** to calculate the mass of calcium carbonate present in the sample of **FB 5** heated.

[A_r : C, 12.0; O, 16.0; Ca, 40.1]

mass of CaCO_3 = ... g

- (iii) Calculate the percentage by mass of calcium carbonate in the limestone.

percentage of calcium carbonate = ... %
[3]

- (c) **FB 5** and **FB 1** are samples of the same limestone. You have determined the percentage of calcium carbonate in both **Questions 1** and **2** using two different procedures.

- (i) Which procedure is the less accurate? Explain your answer.

.....
.....

- (ii) Suggest a change to the less accurate practical procedure that would improve the accuracy and explain your answer.

.....
.....
.....

[2]

[Total: 7]

3 Qualitative Analysis

At each stage of any test you are to record details of the following.

- colour changes seen
- the formation of any precipitate
- the solubility of such precipitates in an excess of the reagent added

Where gases are released they should be identified by a test, **described in the appropriate place in your observations.**

You should indicate clearly at what stage in a test a change occurs.

Marks are **not** given for chemical equations.

No additional tests for ions present should be attempted.

If any solution is warmed, a boiling tube MUST be used.

Rinse and reuse test-tubes and boiling tubes where possible.

Where reagents are selected for use in a test, the name or correct formula of the element or compound must be given.

- (a) A different sample of limestone was reacted with dilute nitric acid to give solution **FB 6**. This sample of limestone contained calcium carbonate, CaCO_3 , and one other salt. This additional salt contains a single cation and a single anion from those listed on pages 11 and 12. By carrying out the following tests you will be able to suggest identities of the additional ions.

<i>test</i>	<i>observations</i>
(i) To a 1 cm depth of FB 6 in a test-tube add aqueous ammonia.	
(ii) To a 1 cm depth of FB 6 in a test-tube add a 1 cm depth of aqueous silver nitrate.	
(iii) To a 1 cm depth of FB 6 in a test-tube add a 1 cm depth of aqueous barium chloride or barium nitrate.	

- (iv) Suggest all possible identities for the ions present in **FB 6**, apart from Ca^{2+} and NO_3^- .

.....

- (v) Select a reagent to use in a further test on **FB 6** to confirm that one of the cations you have listed in (iv) is **not** present in **FB 6**. Carry out your test and complete the table.

<i>test</i>	<i>observations</i>	<i>conclusion</i>
To a 1 cm depth of FB 6 in a test-tube add 		

I	
II	
III	
IV	
V	
VI	

[6]

- (b) You are provided with a solid, **FB 7**. By carrying out the following tests you will be able to identify three of the ions present in **FB 7**.

<i>test</i>	<i>observations</i>
(i) Place a spatula measure of FB 7 in a hard-glass test-tube. Heat gently at first, then	
heat more strongly until no further change is seen, then	
allow the tube to cool.	

I	
II	
III	
IV	
V	
VI	
VII	

<i>test</i>	<i>observations</i>
(ii) Place a spatula measure of FB 7 in a test-tube. Add about a 5 cm depth of dilute nitric acid. You will use the solution formed for tests (iii) to (v).	
(iii) To a 1 cm depth of the solution in a test-tube add a few drops of aqueous potassium manganate(VII), then	
add a few drops of starch solution.	
(iv) To a 1 cm depth of the solution in a test-tube add a few drops of aqueous silver nitrate.	
(v) To a 1 cm depth of the solution in a test-tube add aqueous ammonia.	

Use the Qualitative Analysis Notes on pages 11 and 12 to identify **three** of the ions present.

.....

[7]

[Total: 13]

Qualitative Analysis Notes

Key: [ppt. = precipitate]

1 Reactions of aqueous cations

ion	reaction with	
	NaOH(aq)	NH ₃ (aq)
aluminium, Al ³⁺ (aq)	white ppt. soluble in excess	white ppt. insoluble in excess
ammonium, NH ₄ ⁺ (aq)	no ppt. ammonia produced on heating	—
barium, Ba ²⁺ (aq)	no ppt. (if reagents are pure)	no ppt.
calcium, Ca ²⁺ (aq)	white ppt. with high [Ca ²⁺ (aq)]	no ppt.
chromium(III), Cr ³⁺ (aq)	grey-green ppt. soluble in excess giving dark green solution	grey-green ppt. insoluble in excess
copper(II), Cu ²⁺ (aq)	pale blue ppt. insoluble in excess	blue ppt. soluble in excess giving dark blue solution
iron(II), Fe ²⁺ (aq)	green ppt. turning brown on contact with air insoluble in excess	green ppt. turning brown on contact with air insoluble in excess
iron(III), Fe ³⁺ (aq)	red-brown ppt. insoluble in excess	red-brown ppt. insoluble in excess
magnesium, Mg ²⁺ (aq)	white ppt. insoluble in excess	white ppt. insoluble in excess
manganese(II), Mn ²⁺ (aq)	off-white ppt. rapidly turning brown on contact with air insoluble in excess	off-white ppt. rapidly turning brown on contact with air insoluble in excess
zinc, Zn ²⁺ (aq)	white ppt. soluble in excess	white ppt. soluble in excess

2 Reactions of anions

<i>ion</i>	<i>reaction</i>
carbonate, CO_3^{2-}	CO_2 liberated by dilute acids
chloride, $\text{Cl}^-(\text{aq})$	gives white ppt. with $\text{Ag}^+(\text{aq})$ (soluble in $\text{NH}_3(\text{aq})$);
bromide, $\text{Br}^-(\text{aq})$	gives cream ppt. with $\text{Ag}^+(\text{aq})$ (partially soluble in $\text{NH}_3(\text{aq})$);
iodide, $\text{I}^-(\text{aq})$	gives yellow ppt. with $\text{Ag}^+(\text{aq})$ (insoluble in $\text{NH}_3(\text{aq})$);
nitrate, $\text{NO}_3^-(\text{aq})$	NH_3 liberated on heating with $\text{OH}^-(\text{aq})$ and Al foil
nitrite, $\text{NO}_2^-(\text{aq})$	NH_3 liberated on heating with $\text{OH}^-(\text{aq})$ and Al foil; NO liberated by dilute acids (colourless $\text{NO} \rightarrow$ (pale) brown NO_2 in air)
sulfate, $\text{SO}_4^{2-}(\text{aq})$	gives white ppt. with $\text{Ba}^{2+}(\text{aq})$ (insoluble in excess dilute strong acids)
sulfite, $\text{SO}_3^{2-}(\text{aq})$	SO_2 liberated with dilute acids; gives white ppt. with $\text{Ba}^{2+}(\text{aq})$ (soluble in excess dilute strong acids)

3 Tests for gases

<i>gas</i>	<i>test and test result</i>
ammonia, NH_3	turns damp red litmus paper blue
carbon dioxide, CO_2	gives a white ppt. with limewater (ppt. dissolves with excess CO_2)
chlorine, Cl_2	bleaches damp litmus paper
hydrogen, H_2	“pops” with a lighted splint
oxygen, O_2	relights a glowing splint
sulfur dioxide, SO_2	turns acidified aqueous potassium manganate(VII) from purple to colourless

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CANDIDATE
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CHEMISTRY

9701/35

Paper 3 Advanced Practical Skills 1

May/June 2014

2 hours

Candidates answer on the Question Paper.

Additional Materials: As listed in the Confidential Instructions

READ THESE INSTRUCTIONS FIRST

Write your Centre number, candidate number and name on all the work you hand in.
Give details of the practical session and laboratory where appropriate, in the boxes provided.
Write in dark blue or black pen.
You may use an HB pencil for any diagrams or graphs.
Do not use staples, paper clips, glue or correction fluid.
DO NOT WRITE IN ANY BARCODES.

Answer **all** questions.
Electronic calculators may be used.
You may lose marks if you do not show your working or if you do not use appropriate units.
Use of a Data Booklet is unnecessary.

Qualitative Analysis Notes are printed on pages 11 and 12.

At the end of the examination, fasten all your work securely together.
The number of marks is given in brackets [] at the end of each question or part question.

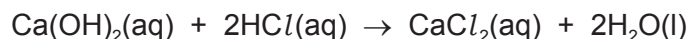
Session
Laboratory

For Examiner's Use	
1	
2	
3	
Total	

This document consists of **12** printed pages.



- 1 Limewater is a saturated solution of calcium hydroxide. A sample of limewater was diluted to produce solution **FA 1**. You are to determine the concentration of calcium hydroxide, Ca(OH)_2 , in **FA 1** by titration with hydrochloric acid, HCl , which you will first dilute. The equation for the reaction between calcium hydroxide and hydrochloric acid is given below.



FA 1 is aqueous calcium hydroxide, Ca(OH)_2 .

FA 2 is $0.500 \text{ mol dm}^{-3}$ hydrochloric acid, HCl .

methyl orange indicator

(a) Method

Read through the method before starting any practical work.

(i) Dilution

- Fill the burette with **FA 2**.
- Run between 24.00 and 26.00 cm^3 of **FA 2** into the 250 cm^3 volumetric flask.
- Record your burette readings and the volume used in the space below.
- Make up the solution to 250 cm^3 with distilled water and shake the flask to ensure thorough mixing. This solution is **FA 3**.

volume of $0.500 \text{ mol dm}^{-3}$ HCl used = ... cm^3

(ii) Titration

- Empty and rinse the burette.
- Fill the burette with **FA 3**.
- Pipette 25.0 cm^3 of **FA 1** into a conical flask.
- Add a few drops of methyl orange indicator.
- Perform a **rough titration** and record your burette readings in the space below.

The rough titre is cm^3 .

- Carry out as many accurate titrations as you think necessary to obtain consistent results.
- Make certain any recorded results show the precision of your practical work.
- Record, in a suitable form below, all of your burette readings and the volume of **FA 3** added in each accurate titration.

I	
II	
III	
IV	
V	
VI	
VII	
VIII	

[8]

- (b) From your accurate titration results, obtain a suitable value to be used in your calculations. Show clearly how you have obtained this value.

25.0 cm³ of **FA 1** required cm³ of **FA 3**. [1]

(c) Calculations

Show your working and appropriate significant figures in the final answer to **each** step of your calculations.

- (i) Use your volume of **FA 2** from **(a)(i)** to calculate the concentration of **FA 3**.

concentration of **FA 3** = ... mol dm⁻³

- (ii) Use your answer to **(c)(i)** and the value calculated in **(b)** to calculate the number of moles of hydrochloric acid used to neutralise 25.0 cm³ of **FA 1**.

moles of HCl = ... mol

- (iii) Use your answer to **(c)(ii)** to calculate the number of moles of calcium hydroxide in 25.0 cm³ of **FA 1**.

moles of Ca(OH)₂ = ... mol

- (iv) Use your answer to **(c)(iii)** to calculate the concentration, in mol dm⁻³, of calcium hydroxide in **FA 1**.

I	
II	
III	
IV	
V	

concentration of Ca(OH)₂ = ... mol dm⁻³
[5]

- (d) (i) The maximum error in a single burette reading is $\pm 0.05 \text{ cm}^3$.
When making up the diluted acid, **FA 3**, a student recorded that 24.80 cm^3 of **FA 2** was used.
What are the smallest and largest possible volumes of acid that were run into the volumetric flask?

smallest volume used = ... cm^3 largest volume used = ... cm^3

- (ii) If the actual volume of acid added had been less than 24.80 cm^3 , how would the value the student calculated for the concentration of Ca(OH)_2 in (c)(iv) compare to the true value? Explain your answer.

.....
.....
.....

[2]

- (e) If a sample of limewater is left open to the air, the concentration of calcium hydroxide decreases. Explain why.

.....
..... [1]

[Total: 17]

- 2 You are to determine the percentage by mass of zinc carbonate in a sample of powdered zinc carbonate ore by means of thermal decomposition. You may assume that none of the other components of the ore is affected by heating. The equation for the reaction occurring is given below.



FA 4 is zinc carbonate ore.

(a) Method

Read through the method **before** starting any practical work and prepare a table for your results in the space below.

- Weigh the empty crucible and record the mass in your table.
- Transfer **all** the **FA 4** into the crucible.
- Weigh the crucible with **FA 4** and record the mass.
- Place the crucible on the pipe-clay triangle.
- Heat the crucible gently for about one minute and then strongly for four minutes.
- Remove the Bunsen burner and allow the crucible to cool.
- While the crucible is cooling start working on another question.
- Reweigh the cooled crucible with contents and record the mass.
- Record the mass of **FA 4** used and the mass of solid remaining after heating.
- Beneath your table, record any observations you have made while the solid was heated and cooled.

I	
II	
III	
IV	

[4]

(b) Calculations

Show your working and appropriate significant figures in the final answer to **each** step of your calculations.

- (i) From your results in (a), calculate the mass of carbon dioxide lost on heating **FA 4**.

mass of CO_2 lost = ... g

- (ii) Use your answer to (i) to calculate the mass of zinc carbonate present in the sample of **FA 4** that was heated.

[A_r : C, 12.0; O, 16.0; Zn, 65.4]

mass of ZnCO_3 = ... g

- (iii) Calculate the percentage by mass of zinc carbonate in the zinc carbonate ore.

I	
II	
III	
IV	

percentage of ZnCO_3 = ... %
[4]

- (c) Suggest how a student, using all the same apparatus, could alter the method to be more confident that the percentage of zinc carbonate is correct. Explain your answer.

.....

 [2]

[Total: 10]

3 Qualitative Analysis

At each stage of any test you are to record details of the following.

- colour changes seen
- the formation of any precipitate
- the solubility of such precipitates in an excess of the reagent added

Where gases are released they should be identified by a test, **described in the appropriate place in your observations.**

You should indicate clearly at what stage in a test a change occurs.

Marks are **not** given for chemical equations.

No additional tests for ions present should be attempted.

If any solution is warmed, a boiling tube MUST be used.

Rinse and reuse test-tubes and boiling tubes where possible.

Where reagents are selected for use in a test, the name or correct formula of the element or compound must be given.

- (a) You are provided with a different sample of zinc carbonate ore, labelled **FA 5**. This sample contains zinc carbonate, ZnCO_3 , and one other salt. This additional salt contains a single cation and a single anion from those listed on pages 11 and 12. By carrying out the following tests you will be able to suggest the identities of the ions in the additional salt.

<i>test</i>	<i>observations</i>
(i) Transfer the solid FA 5 into a boiling tube and add a 3 cm depth of dilute nitric acid to make solution FA 6. Use this solution in tests (ii) to (v).	
(ii) To a 1 cm depth of FA 6 in a test-tube, add aqueous sodium hydroxide.	
(iii) To a 1 cm depth of FA 6 in a test-tube, add aqueous ammonia.	
(iv) To a 1 cm depth of FA 6 in a test-tube, add a 1 cm depth of aqueous silver nitrate.	
(v) To a 1 cm depth of FA 6 in a test-tube, add a 1 cm depth of aqueous barium chloride or barium nitrate.	

- (vi) Suggest the identity of the cation and anion (apart from Zn^{2+} and CO_3^{2-}) present in **FA 5**. Explain your choice.

cation

reason

anion

reason

[7]

I		II		III		IV		V		VI		VII	
---	--	----	--	-----	--	----	--	---	--	----	--	-----	--

- (b) You are provided with solid **FA 7**, which is a mixture of two salts.
Use separate portions of **FA 7** to perform the experiments below.

<i>test</i>		<i>observations</i>
(i)	Heat a spatula measure of FA 7 in a hard-glass test-tube gently at first, then	
	heat more strongly and test the gas evolved with litmus paper.	
Dissolve a spatula measure of FA 7 in a test-tube with a 5 cm depth of distilled water for use in tests (ii) and (iii).		
(ii)	To a 1 cm depth of FA 7 (aq) in a test-tube, add aqueous silver nitrate,	
	followed by aqueous ammonia.	
(iii)	To a 1 cm depth of FA 7 (aq) add barium chloride or barium nitrate,	
	followed by dilute nitric acid.	

I	
II	
III	
IV	
V	
VI	

- (iv) Use your observations and the Qualitative Analysis Notes from pages 11 and 12 to identify **three** of the ions present. Give evidence for your choice of ions.

ion evidence

.....

ion evidence

.....

ion evidence

.....

[6]

[Total: 13]

Qualitative Analysis Notes

Key: [ppt. = precipitate]

1 Reactions of aqueous cations

ion	reaction with	
	NaOH(aq)	NH ₃ (aq)
aluminium, Al ³⁺ (aq)	white ppt. soluble in excess	white ppt. insoluble in excess
ammonium, NH ₄ ⁺ (aq)	no ppt. ammonia produced on heating	—
barium, Ba ²⁺ (aq)	no ppt. (if reagents are pure)	no ppt.
calcium, Ca ²⁺ (aq)	white ppt. with high [Ca ²⁺ (aq)]	no ppt.
chromium(III), Cr ³⁺ (aq)	grey-green ppt. soluble in excess giving dark green solution	grey-green ppt. insoluble in excess
copper(II), Cu ²⁺ (aq)	pale blue ppt. insoluble in excess	blue ppt. soluble in excess giving dark blue solution
iron(II), Fe ²⁺ (aq)	green ppt. turning brown on contact with air insoluble in excess	green ppt. turning brown on contact with air insoluble in excess
iron(III), Fe ³⁺ (aq)	red-brown ppt. insoluble in excess	red-brown ppt. insoluble in excess
magnesium, Mg ²⁺ (aq)	white ppt. insoluble in excess	white ppt. insoluble in excess
manganese(II), Mn ²⁺ (aq)	off-white ppt. rapidly turning brown on contact with air insoluble in excess	off-white ppt. rapidly turning brown on contact with air insoluble in excess
zinc, Zn ²⁺ (aq)	white ppt. soluble in excess	white ppt. soluble in excess

2 Reactions of anions

<i>ion</i>	<i>reaction</i>
carbonate, CO_3^{2-}	CO_2 liberated by dilute acids
chloride, $\text{Cl}^-(\text{aq})$	gives white ppt. with $\text{Ag}^+(\text{aq})$ (soluble in $\text{NH}_3(\text{aq})$);
bromide, $\text{Br}^-(\text{aq})$	gives cream ppt. with $\text{Ag}^+(\text{aq})$ (partially soluble in $\text{NH}_3(\text{aq})$);
iodide, $\text{I}^-(\text{aq})$	gives yellow ppt. with $\text{Ag}^+(\text{aq})$ (insoluble in $\text{NH}_3(\text{aq})$);
nitrate, $\text{NO}_3^-(\text{aq})$	NH_3 liberated on heating with $\text{OH}^-(\text{aq})$ and Al foil
nitrite, $\text{NO}_2^-(\text{aq})$	NH_3 liberated on heating with $\text{OH}^-(\text{aq})$ and Al foil; NO liberated by dilute acids (colourless $\text{NO} \rightarrow$ (pale) brown NO_2 in air)
sulfate, $\text{SO}_4^{2-}(\text{aq})$	gives white ppt. with $\text{Ba}^{2+}(\text{aq})$ (insoluble in excess dilute strong acids)
sulfite, $\text{SO}_3^{2-}(\text{aq})$	SO_2 liberated with dilute acids; gives white ppt. with $\text{Ba}^{2+}(\text{aq})$ (soluble in excess dilute strong acids)

3 Tests for gases

<i>gas</i>	<i>test and test result</i>
ammonia, NH_3	turns damp red litmus paper blue
carbon dioxide, CO_2	gives a white ppt. with limewater (ppt. dissolves with excess CO_2)
chlorine, Cl_2	bleaches damp litmus paper
hydrogen, H_2	“pops” with a lighted splint
oxygen, O_2	relights a glowing splint
sulfur dioxide, SO_2	turns acidified aqueous potassium manganate(VII) from purple to colourless

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