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9701/31

May/June 2007

2 hours

Additional Materials: As listed in the Instructions to Supervisors

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 a soft pencil for any diagrams, graphs or rough working.
Do not use staples, paper clips, highlighters, glue or correction fluid.
DO NOT WRITE ON ANY BARCODES.

Answer **all** questions.
You are advised to show all working in calculations.
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 required to find the percentage purity of a sample of sodium carbonate, Na_2CO_3 .

FA 1 contains 4.50 g dm^{-3} of the impure sodium carbonate.

FA 2 is 0.50 mol dm^{-3} hydrochloric acid, HCl .

(a) Dilution of FA 2

By using a burette, measure between 33.00 cm^3 and 34.00 cm^3 of **FA 2** into the 250 cm^3 graduated flask labelled **FA 3**.

Record your burette readings and the volume of **FA 2** added to the flask in the space below.

Make up the contents of the flask to the 250 cm^3 mark with distilled water. Place the stopper in the flask and mix the contents thoroughly by slowly inverting the flask a number of times.

Titration

Fill a second burette with **FA 3**, the diluted solution of hydrochloric acid.

Pipette 25.0 cm^3 of **FA 1** into a conical flask. Add a few drops of methyl orange indicator and titrate with **FA 3**.

Perform a rough (trial) titration and sufficient further titrations to obtain accurate results.

Record your titration results in the space below. Make certain that your recorded results show the precision of your working.

i	
ii	
iii	
iv	
v	
vi	

[6]

- (b)** From your titration results obtain a suitable volume of **FA 3** to be used in your calculations.
Show clearly how you obtained this volume.

[1]

Calculations

Show your working and appropriate significant figures in all of your calculations.

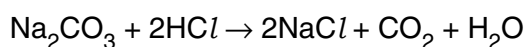
- (c) Calculate how many moles of HCl are contained in the **FA 2** run into the graduated flask.

.....mol of HCl were run into the graduated flask.

Calculate how many moles of HCl are contained in the volume of **FA 3** which reacted with 25.0 cm^3 of **FA 1**.

.....mol of HCl reacted with 25.0 cm^3 of **FA 1**.

Use this answer to calculate how many moles of sodium carbonate, Na_2CO_3 , are present in 1.00 dm^3 of **FA 1**.



.....mol of Na_2CO_3 are present in 1.00 dm^3 of **FA 1**.

Calculate the mass of sodium carbonate, Na_2CO_3 , in 1.00 dm^3 of **FA 1**.
[A_r : C, 12.0; O, 16.0; Na, 23.0]

FA 1 is g dm^{-3} of Na_2CO_3 .

Calculate, to **3 significant figures**, the percentage purity of the sodium carbonate, Na_2CO_3 , dissolved in **FA 1**.

The percentage purity of the sodium carbonate dissolved in **FA 1** is%.

[5]

i	
ii	
iii	
iv	
v	

- (d) Look at the scale on the 25 cm³ measuring cylinder provided. Record the smallest scale division on the measuring cylinder and estimate the error in reading the scale.

smallest division = ... cm³

estimated error = $\pm$ cm³

If 25 cm³ of **FA 1** is measured with a measuring cylinder, calculate the estimated percentage error.

The estimated error is %.

Your pipette is calibrated with an error of ± 0.06 cm³.

Calculate the percentage error when measuring 25.0 cm³ of solution with this pipette.

The error is %.
[2]

- (e) Use the measuring cylinder to place 25 cm^3 of **FA 1** into a conical flask.
Add methyl orange indicator as before and titrate with **FA 3**.
Repeat the titration **once** using the measuring cylinder. Record your results below.

Would you expect to be able to obtain consistent titres using a measuring cylinder?
Explain your answer.

.....

.....

.....

.....

.....[1]

- (f) A student suspects that the presence of dissolved carbon dioxide affects the end-point of the titration.
Suggest a simple modification to the experimental technique to eliminate the dissolved carbon dioxide as the titration is performed.

.....

.....

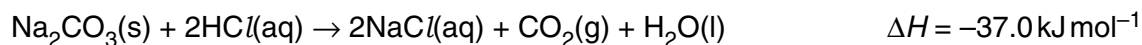
.....

.....[1]

[Total: 16]

- 2 Read through the question before starting any practical work.

The percentage purity of the sodium carbonate can also be determined by measuring the temperature change when a weighed sample of the solid carbonate reacts with an excess of hydrochloric acid and the following information is used.



You are provided with the following.

FA 4, impure solid sodium carbonate

FA 5, 2.0 mol dm^{-3} hydrochloric acid

Measurement of temperature change

- (a) Follow the instructions below to determine the percentage purity of the sodium carbonate.

You will carry out the experiment twice.

- Weigh the empty weighing bottle.
- Reweigh the bottle with between 4.00 g and 4.50 g of **FA 4**.
- Support the plastic cup in the 250 cm^3 beaker and add to it, from a measuring cylinder, 50 cm^3 of **FA 5**.
- Measure and record the steady temperature of the **FA 5** in the plastic cup.
- Add the **FA 4** from the weighing bottle to the plastic cup, a little at a time to prevent acid spray. Stir and record the highest temperature reached.
- Reweigh the empty weighing bottle.

In an appropriate format in the space below, record

- all measurements of mass and temperature,
- the mass of **FA 4** used, **m**,
- the temperature rise, ΔT .

Empty and rinse the plastic cup.

Repeat the experiment.

Results

Calculations

- (b) For **each** experiment calculate $\frac{\Delta T}{m}$, the temperature rise per gram of **FA 4** used.

first experiment $\frac{\Delta T}{m} = \dots\dots\dots ^\circ\text{C g}^{-1}$

second experiment $\frac{\Delta T}{m} = \dots\dots\dots ^\circ\text{C g}^{-1}$

Calculate the mean value of $\frac{\Delta T}{m}$.

The mean value of $\frac{\Delta T}{m} = \dots\dots\dots ^\circ\text{C g}^{-1}$
[2]

- (c) Is one repeat of the experiment sufficient or should it be repeated again? Explain your answer.

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

- (d) Calculate the percentage purity of the sodium carbonate using the following expression.
Note, this value is likely to be different from the one you obtained in question 1.

$$\text{purity} = \frac{\Delta T}{m} \times \frac{1}{37.0} \times 2279\%$$

purity = ...% [1]

[Total: 8]

- 3 **FA 6** and **FA 7** are solids **each** containing one cation and one anion from those listed on page 10 and page 11.

You will dissolve each solid in dilute nitric acid, HNO_3 , and use the solutions formed in reactions with aqueous sodium hydroxide, NaOH , and aqueous ammonia, NH_3 .

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

- colour changes seen
- the formation of any precipitate
- the solubility of any precipitate 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.

(a) Preparation of the solutions

Dissolve each solid separately in dilute HNO_3 in a 100 cm^3 beaker. Use the **minimum** quantity of acid, added a little at a time and warm to dissolve the solid if necessary. Then add distilled water to each solution to give a total volume of about 60 cm^3 .

Record your observations in the space below.

[4]

(b) Reactions of the solutions formed in (a) with NaOH(aq) and $\text{NH}_3(\text{aq})$

Use separate portions of each of the solutions formed in **(a)** in reactions with aqueous NaOH and with aqueous NH_3 , added until in excess.

For each test use 1 cm depth of solution in a boiling-tube.

Record details of the tests performed and the observations made.

[6]

- (c) Both cations and one anion can now be definitely identified.

cation in **FA 6**

evidence

cation in **FA 7**

evidence

The anion in **FA** is

evidence

[3]

- (d) Tests to identify the remaining anion. Do not carry out these tests.

From the Quantitative Analysis Notes for anions select **two** reagents which could be used, in **one** test, to indicate the presence of chloride ions in one of the solutions.

Describe how you would use these reagents and the expected observations if the chloride ion were present.

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

Select another reagent that would also indicate the presence of a chloride ion in the solution. Describe the expected observation if the chloride ion were present.

.....
.....
.....[3]

[Total: 16]

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)	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. insoluble in excess	green ppt. insoluble in excess
iron(III), Fe ³⁺ (aq)	red-brown ppt. insoluble in excess	red-brown ppt. insoluble in excess
lead(II), Pb ²⁺ (aq)	white ppt. soluble in excess	white 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. insoluble in excess	off-white ppt. insoluble in excess
zinc, Zn ²⁺ (aq)	white ppt. soluble in excess	white ppt. soluble in excess

[Lead(II) ions can be distinguished from aluminium ions by the insolubility of lead(II) chloride.]

2 Reactions of anions

<i>ion</i>	<i>reaction</i>
carbonate, CO_3^{2-}	CO_2 liberated by dilute acids
chromate(VI), $\text{CrO}_4^{2-}(\text{aq})$	yellow solution turns orange with $\text{H}^+(\text{aq})$; gives yellow ppt. with $\text{Ba}^{2+}(\text{aq})$; gives bright yellow ppt. with $\text{Pb}^{2+}(\text{aq})$
chloride, $\text{Cl}^-(\text{aq})$	gives white ppt. with $\text{Ag}^+(\text{aq})$ (soluble in $\text{NH}_3(\text{aq})$); gives white ppt. with $\text{Pb}^{2+}(\text{aq})$
bromide, $\text{Br}^-(\text{aq})$	gives pale cream ppt. with $\text{Ag}^+(\text{aq})$ (partially soluble in $\text{NH}_3(\text{aq})$); gives white ppt. with $\text{Pb}^{2+}(\text{aq})$
iodide, $\text{I}^-(\text{aq})$	gives yellow ppt. with $\text{Ag}^+(\text{aq})$ (insoluble in $\text{NH}_3(\text{aq})$); gives yellow ppt. with $\text{Pb}^{2+}(\text{aq})$
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)
sulphate, $\text{SO}_4^{2-}(\text{aq})$	gives white ppt. with $\text{Ba}^{2+}(\text{aq})$ or with $\text{Pb}^{2+}(\text{aq})$ (insoluble in excess dilute strong acid)
sulphite, $\text{SO}_3^{2-}(\text{aq})$	SO_2 liberated with dilute acids; gives white ppt. with $\text{Ba}^{2+}(\text{aq})$ (soluble in excess dilute strong acid)

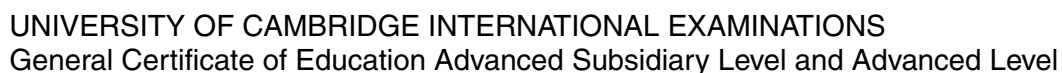
3 Tests for gases

<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
sulphur dioxide, SO_2	turns potassium dichromate(VI) (aq) from orange to green

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Answer **all** questions.
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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	
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Total	

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

- 1 You are required to determine the concentration in g dm^{-3} of hydrated ammonium iron(II) sulphate, $(\text{NH}_4)_2\text{SO}_4 \cdot \text{FeSO}_4 \cdot 6\text{H}_2\text{O}$, in the solution **FB 1**.

FB 1 contains hydrated ammonium iron(II) sulphate.

FB 2 is $0.0120 \text{ mol dm}^{-3}$ potassium manganate(VII), KMnO_4 .

(a) Dilution of FB 1

By using a burette measure between 36.00 cm^3 and 37.00 cm^3 of **FB 1** into the 250 cm^3 graduated flask labelled **FB 3**.

Record your burette readings and the volume of **FB 1** added to the flask in the space below.

Make up the contents of the flask to the 250 cm^3 mark with distilled water. Place the stopper in the flask and mix the contents thoroughly by slowly inverting the flask a number of times.

Titration

Fill a second burette with **FB 2**.

Pipette 25.0 cm^3 of **FB 3** into a conical flask. Use a measuring cylinder to add approximately 10 cm^3 of 1.0 mol dm^{-3} sulphuric acid, H_2SO_4 , and titrate with **FB 2** until the first permanent pink colour remains in the solution.

Perform one rough (trial) titration and sufficient further titrations to obtain accurate results.

Record your titration results in the space below. Make certain that your recorded results show the precision of your working.

i	
ii	
iii	
iv	
v	
vi	

[6]

- (b) From your titration results obtain a suitable volume of **FB 2** to be used in your calculations.
Show clearly how you obtained this volume.

[1]

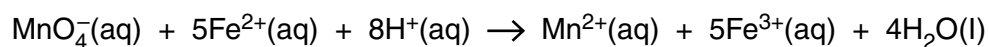
Calculations

Show your working and appropriate significant figures in all of your calculations.

- (c) Calculate how many moles of KMnO_4 were run from the burette during the titration.

..... mol of KMnO_4 were run from the burette.

Calculate how many moles of Fe^{2+} ions reacted with the KMnO_4 run from the burette.



..... mol of Fe^{2+} reacted with the KMnO_4 run from the burette.

Calculate the concentration, in mol dm^{-3} , of Fe^{2+} in **FB 3**.

Concentration of Fe^{2+} in **FB 3** = ... mol dm^{-3} .

Calculate the concentration, in mol dm^{-3} , of Fe^{2+} in **FB 1**.

Concentration of Fe^{2+} in **FB 1** = ... mol dm^{-3} .

Calculate, to **4 significant figures**, the concentration of $(\text{NH}_4)_2\text{SO}_4 \cdot \text{FeSO}_4 \cdot 6\text{H}_2\text{O}$ in **FB 1** in g dm^{-3} .

[A_r : Fe, 55.8; H, 1.0; N, 14.0; O, 16.0; S, 32.1]

i	
ii	
iii	
iv	
v	

FB 1 contains g dm^{-3} of $(\text{NH}_4)_2\text{SO}_4 \cdot \text{FeSO}_4 \cdot 6\text{H}_2\text{O}$.
[5]

- (d) A student learns that the solution of the iron(II) salt has been prepared by dissolving the solid in distilled water that has absorbed air from the laboratory.
Suggest a way in which the distilled water can be prepared and stored in the laboratory to ensure that it contains a minimum of dissolved air.

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

- (e) Estimate the error in reading a volume from a burette.

smallest division on burette scale = ... cm^3

estimated error in reading a volume = $\pm$ cm^3 [1]

- (f) A titre value is obtained by the difference between final and initial burette readings.

What is the **maximum** possible error in obtaining a titre reading?

estimated **maximum** error in the titre = $\pm$ cm^3 [1]

- (g) During one titration a student reads the burette twice.

Each reading has an error but the titre has no error. Explain how this can happen.

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

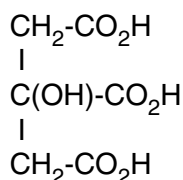
[Total: 16]

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2 Read through the question before starting any practical work.

You are required to determine the enthalpy change when citric acid reacts with an excess of sodium hydrogencarbonate.

Citric acid, found in citrus fruit such as lemons and limes, is 2-hydroxypropane-1,2,3-tricarboxylic acid.



FB 4 is 0.8 mol dm⁻³ citric acid.

FB 5 is solid sodium hydrogencarbonate, NaHCO₃.

- (a) Citric acid is a triprotic (tribasic) acid – one mole of the acid reacts with three moles of sodium hydrogencarbonate.

Calculate the minimum mass of sodium hydrogencarbonate that will react with all of the acid in 50.0 cm³ of **FB 4**.

[A_r: Na, 23.0; H, 1.0; C, 12.0; O, 16.0]

mass of NaHCO₃ = ... g [1]

(b) Method

Follow the instructions below to determine the enthalpy change for the reaction. You will carry out the experiment twice.

- Weigh the empty weighing bottle.
- Weigh the bottle with between 11.5 g and 12.0 g, an excess, of **FB 5**.
- Support the plastic cup in the 250 cm³ beaker and pipette into it 50.0 cm³ of **FB 4**.
- Measure and record the steady temperature of the **FB 4** in the plastic cup.
- Add the **FB 5** from the weighing bottle, a little at a time, to the plastic cup.
- Stir and record the lowest temperature reached.
- Reweigh the empty weighing bottle.

In an appropriate form at the top of the next page record

- all measurements of mass and temperature,
- the temperature fall, ΔT .

Empty and rinse the plastic cup.

Repeat the experiment and calculate the mean value of ΔT .

Results

The mean value of ΔT is °C.

[6]

- (c) Calculate the enthalpy change of reaction using the following expression.

$$\Delta H_{\text{reaction}} = \text{mean } \Delta T \times 4.3 \text{ kJ mol}^{-1}$$

Your answer should include the appropriate sign.

$$\Delta H_{\text{reaction}} = \text{..... kJ mol}^{-1} \text{ [1]}$$

[Total: 8]

- 3** You are provided with three solutions, **FB 6**, **FB 7** and **FB 8**, each containing one cation and one anion.

One or more of the solutions contains a halide ion. One or more of the solutions contains a sulphate or sulphite ion.

Identification of the anions in FB 6, FB 7 and FB 8

(a) By reference to the Qualitative Analysis Notes on page 12 you are to select and use

- (i)** one reagent to precipitate any halide ion that is present,
- (ii)** a second reagent to confirm the identity of any halide ion present.

Because the solutions are coloured you will need to remove traces of solution from the precipitates.

Record the tests performed, the practical procedures used and the observations made for each of the solutions.

Present this information as clearly as possible in a suitable format in the space below.

i	
ii	
iii	
iv	
v	
vi	
vii	

Use your observations to identify any halide ions present in the solutions **FB 6**, **FB 7** and **FB 8** and state which ion is present in which solution.

.....

.....

.....

.....

.....

(b) Select reagents and carry out tests

- (i)** to show which of the solutions contains a sulphate ion or a sulphite ion, and
- (ii)** to establish which of these ions is present.

Record your tests and observations below.

State which of the ions, sulphate or sulphite, is present in which of the solutions **FB 6**, **FB 7** and **FB 8** and explain how you reached this conclusion from your tests above.

.....

.....

.....

[3]

Identification of the cations in FB 6, FB 7 and FB 8

- (c)** Using aqueous sodium hydroxide and aqueous ammonia it is possible to identify two of the cations present and to draw some conclusions about the nature of the remaining cation.

Carry out tests with these reagents, recording details of what you did and observed in a suitable format in the space below.

[4]

- (d) Explain how your observations in (c) identify **two** of the cations present and which of the solutions contain those cations.

The cation contained in solution **FB** is

explanation

.....

.....

.....

The cation contained in solution **FB** is

explanation

.....

.....

.....

What conclusion of a general nature about the third cation can you draw from your observations in (c)?

.....

.....

.....

.....

.....

.....

[2]

[Total: 16]

Key: [ppt. = precipitate.]

1 Reactions of aqueous cations

	<i>reaction with</i>	
	NaOH(aq)	NH ₃ (aq)
aluminium, Al ³⁺ (aq)	white ppt. soluble in excess	white ppt. insoluble in excess
ammonium, NH ₄ ⁺ (aq)	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. insoluble in excess	green ppt. insoluble in excess
iron(III), Fe ³⁺ (aq)	red-brown ppt. insoluble in excess	red-brown ppt. insoluble in excess
lead(II), Pb ²⁺ (aq)	white ppt. soluble in excess	white 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. insoluble in excess	off-white ppt. insoluble in excess
zinc, Zn ²⁺ (aq)	white ppt. soluble in excess	white ppt. soluble in excess

[Lead(II) ions can be distinguished from aluminium ions by the insolubility of lead(II) chloride.]

2 Reactions of anions

<i>ion</i>	<i>reaction</i>
carbonate, CO_3^{2-}	CO_2 liberated by dilute acids
chromate(VI), CrO_4^{2-} (aq)	yellow solution turns orange with H^+ (aq); gives yellow ppt. with Ba^{2+} (aq); gives bright yellow ppt. with Pb^{2+} (aq)
chloride, Cl^- (aq)	gives white ppt. with Ag^+ (aq) (soluble in NH_3 (aq)); gives white ppt. with Pb^{2+} (aq)
bromide, Br^- (aq)	gives pale cream ppt. with Ag^+ (aq) (partially soluble in NH_3 (aq)); gives white ppt. with Pb^{2+} (aq)
iodide, I^- (aq)	gives yellow ppt. with Ag^+ (aq) (insoluble in NH_3 (aq)); gives yellow ppt. with Pb^{2+} (aq)
nitrate, NO_3^- (aq)	NH_3 liberated on heating with OH^- (aq) and Al foil
nitrite, NO_2^- (aq)	NH_3 liberated on heating with OH^- (aq) and Al foil, NO liberated by dilute acids (colourless $\text{NO} \rightarrow$ (pale) brown NO_2 in air)
sulphate, SO_4^{2-} (aq)	gives white ppt. with Ba^{2+} (aq) or with Pb^{2+} (aq) (insoluble in excess dilute strong acid)
sulphite, SO_3^{2-} (aq)	SO_2 liberated with dilute acids; gives white ppt. with Ba^{2+} (aq) (soluble in excess dilute strong acid)

3 Tests for gases

<i>gas</i>	<i>test and test results</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)
chloride, Cl_2	bleaches damp litmus paper
hydrogen, H_2	“pops” with a lighted splint
oxygen, O_2	relights a glowing splint
sulphur dioxide, SO_2	turns potassium dichromate(VI) (aq) from orange to green

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