HWA CHONG INSTITUTION
C2 Preliminary Examination
Higher 2

NAME		**CT GROUP**	**18S**

CHEMISTRY

9729/04

Paper 4 Practical

28 August 2019

2 hours 30 minutes

Candidates answer on the Question Paper

READ THESE INSTRUCTIONS FIRST

Write your name and class on all the work you hand in.
Give details of the practical shift 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.

Answer **all** questions in the spaces provided on the Question Paper.

The use of an approved scientific calculator is expected, where appropriate.
You may lose marks if you do not show your working or if you do not use appropriate units.
Qualitative Analysis Notes are printed on pages 17 and 18.

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.

Shift
Laboratory

For Examiner's Use	
1	
2	
3	
4	
Total	

Answer **all** the questions in the spaces provided.

1 Determination of the enthalpy change of neutralisation, ΔH_{neut}, of a strong acid by a strong base

The enthalpy change of neutralisation, ΔH_{neut}, is the enthalpy change when one mole of water is formed during a neutralisation reaction as shown in equation 1.

equation 1 $\quad H^+(aq) + OH^-(aq) \rightarrow H_2O(l)$

FA 1 is a solution of sulfuric acid, H_2SO_4
FA 2 is 1.50 mol dm^{-3} sodium hydroxide, NaOH

You will perform a series of experiments using different volumes of **FA 1** and **FA 2** which together give a total volume of 50 cm^3. The change in temperature, ΔT, for each experiment will be determined and used to plot a graph of ΔT against volume of **FA 1** used.

You will then use data from the graph to determine the concentration of sulfuric acid in **FA 1**, and a value for the enthalpy change of neutralisation, ΔH_{neut}.

(a) (i) Determining the change in temperature for a series of reactions between FA 1 and FA 2

1. Place the Styrofoam cup in a 250 cm^3 beaker to prevent it from tipping over. Use a measuring cylinder to transfer 10.0 cm^3 of **FA 1** into the cup.

2 Use a measuring cylinder to measure 40.0 cm^3 of **FA 2.**

3. Measure the temperature of the **FA 1** solution using the thermometer. Record the initial temperature of **FA 1** as T_{FA1}.

4. Add **FA 2** to **FA 1** in the Styrofoam cup. Stir the mixture using the thermometer and record the maximum temperature, T_{max}, reached.

5. Wash and dry the Styrofoam cup.

6. Repeat steps 1 to 5 using 20.0 cm^3, 25.0 cm^3, 30.0 cm^3, 35.0 cm^3 and 40.0 cm^3 of **FA 1** and appropriate volumes of **FA 2** each time such that the total volume of the reacting mixture is 50.0 cm^3.

Keep the remaining FA 1 and FA 2 solutions for use in questions 2 and 4.

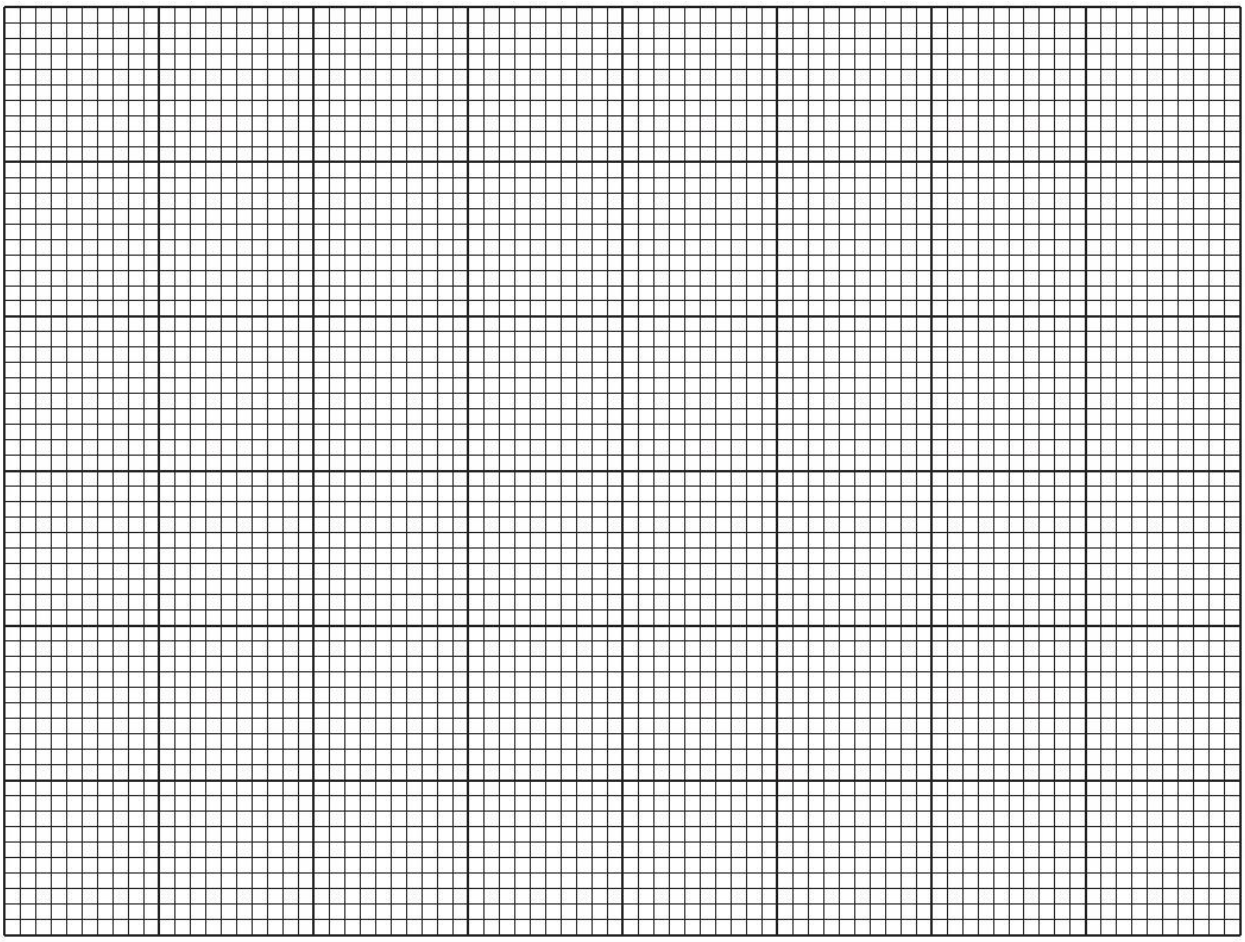
In an appropriate format in the space provided on **page 3**, record:

- all measurements of volumes used,
- all temperatures measured and the change in temperature, ΔT.

Results

[4]

(ii) On the grid provided, plot a graph of ΔT (y-axis) against volume of **FA 1** (x-axis) using the data you obtained in **1(a)(i)**.



Draw two lines of best fit.

- The first best-fit line should be drawn using the plotted points before the maximum change in temperature.
- The second best-fit line should be drawn using the plotted points after the maximum change in temperature.

Extrapolate these lines until they cross.

[3]

(iii) Determine from your graph, the maximum change in temperature, ΔT_{max}, and the volume, V_{max}, of **FA 1** required to obtain this value.

ΔT_{max} = ... V_{max} = ...

[1]

(b) Using your answers in **1(a)(iii),** calculate

(i) the concentration, in mol dm^{-3}, of H_2SO_4 in **FA 1**.

concentration of H_2SO_4 in **FA 1** = ...[1]

(ii) the heat change for the neutralisation reaction at ΔT_{max}.

You should assume that the specific heat capacity of the final solution is 4.18 J g^{-1} K^{-1}, and the density of the final solution is 1.00 g cm^{-3}.

heat change = ...[1]

(c) Using your answers from **1(b)(i)** and **1(b)(ii),** calculate a value for the enthalpy change of neutralisation, ΔH_{neut}.

ΔH_{neut} =..... [1]

(d) Predict the effect on ΔH_{neut} if the experiment was repeated with malonic acid, $HO_2CCH_2CO_2H$, of the same concentration instead of sulfuric acid. Explain your answer.

……

……

……[1]

(e) State one significant source of error in the experiment and suggest an improvement that can be made to reduce this error.

……

……

……[1]

[Total: 13]

2 Determination of water of crystallisation in a hydrated iron(III) salt

FA 3 is hydrated iron(III) sulfate with formula $Fe_2(SO_4)_3.nH_2O$. The addition of excess zinc to a solution of **FA 3** reduces the Fe^{3+} ions to Fe^{2+} ions.

The amount of Fe^{2+} ions can be determined quantitatively by titration against a standard solution of potassium manganate(VII), $KMnO_4$. The reaction is shown in equation 2.

equation 2 $$5Fe^{2+} + MnO_4^- + 8H^+ \rightarrow 5Fe^{3+} + Mn^{2+} + 4H_2O$$

In this experiment, you will prepare a standard solution using **FA 3** and perform titrations to determine the value of *n*, the water of crystallisation in **FA 3**.

You are also provided with

FA 4, 0.0200 mol dm^{-3} potassium manganate(VII), $KMnO_4$,
zinc powder.

You will also need access to the **FA 1** solution you used earlier.

(a) Preparation of standard solution of hydrated iron(III) salt

1. Weigh accurately the sample of **FA 3** provided with the weighing bottle. Transfer all the solid into a 250 cm^3 beaker. Determine the mass of solid **FA 3** used and record all your weighings, to an appropriate level of precision, in the space below.

[1]

2. Use a measuring cylinder to add about 100 cm^3 of **FA 1** to the beaker. Stir the mixture with a glass rod for 3 minutes. Ignore any cloudiness that remains.

3. Transfer the solution into a 250 cm^3 volumetric flask. Rinse the beaker with deionised water and pour the washings into the volumetric flask.

4. Make up to the 250 cm^3 mark with deionised water, stopper and mix thoroughly by inverting the flask a number of times.

5. Label this solution **FA 5**.

(b) Preparation of Fe^{2+} solution from FA 5

6. Use a measuring cylinder to transfer 100 cm^3 of **FA 5** into a **dry** 250 cm^3 beaker.

7. Add all the zinc powder provided in the bottle into the beaker. Cover the beaker with a watch glass.

8. Allow the reaction to take place for about 5 minutes, stirring the reaction mixture from time to time. Record your observations in the space below.

Observations in step 8

[1]

9. Filter the mixture into the **dry** conical flask provided using **dry** filter paper and filter funnel. Ignore any reaction that may still be taking place.

10. Label the filtrate as **FA 6**. Proceed to **2(c)** once you have collected sufficient filtrate.

(c) (i) Titration of **FA 6** against **FA 4**

11. Fill a burette with **FA 4**.

12. Use a pipette to transfer 10.0 cm^3 of **FA 6** into a 250 cm^3 conical flask.

13. Use a measuring cylinder to add about 10 cm^3 of **FA 1** to this flask.

14. Run **FA 4** from the burette into this flask until the appearance of the first permanent pale-pink colour.

15. Record your titration results, to an appropriate level of precision, in the space below.

16. Repeat steps 12 to 15 until consistent results are obtained.

Keep the remaining FA 1 and FA 4 solutions for use in question 4.

Titration results

[3]

(ii) From your titrations, obtain a suitable volume of **FA 4**, $V_{FA\ 4}$, to be used in your calculations. Show clearly how you obtained this volume.

$V_{FA\ 4}$ = ...[3]

(d) **(i)** Calculate the amount of Fe^{2+} in 10.0 cm^3 of **FA 6**.

amount of Fe^{2+} in 10.0 cm^3 of **FA 6** = ... [1]

(ii) In step 7, an excess of zinc was added to convert the Fe^{3+} to Fe^{2+}.

Calculate the amount of Fe^{3+} in 250 cm^3 of **FA 5**.

amount of Fe^{3+} in 250 cm^3 of **FA 5** = ... [1]

(iii) Use your answer from **2(d)(ii)** to calculate the M_r of the hydrated iron(III) sulfate, $Fe_2(SO_4)_3.nH_2O$, in **FA 3**.

M_r of the hydrated iron(III) sulfate = ……

Hence, deduce the value of *n*, the water of crystallisation in the hydrated iron(III) sulfate.

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

n = ……..

[5]

(e) Explain all the observations in step 8 in terms of the chemical processes involved.

……...

.....

…….. [1]

(f) In step 9, excess zinc was filtered off before titration of **FA 6** against **FA 4** to avoid any possible reaction between zinc and **FA 4**.

Suggest another reason why it was necessary to filter off the excess zinc metal, and what effect failing to do it would have on the titre values.

……...

……...

…….. [1]

[Total: 17]

3 Planning

Iron(III) ion, $Fe^{3+}(aq)$, and thiocyanate ion, $SCN^{-}(aq)$, react to give the thiocyanatoiron(III) complex ion, $FeSCN^{2+}(aq)$, as shown in equation 3.

equation 3 $\quad Fe^{3+}(aq) + SCN^{-}(aq) \ll FeSCN^{2+}(aq)$

(a) Write an expression for the equilibrium constant, K_c, of the reaction between $Fe^{3+}(aq)$ and $SCN^{-}(aq)$.

[1]

The colour of $FeSCN^{2+}(aq)$ varies from deep red to orange depending on its concentration. It is possible to determine the concentration of a solution of $FeSCN^{2+}(aq)$ by placing 3 cm^3 of the solution inside a glass sample holder, known as a *cuvette*, into the spectrophotometer. The machine measures the amount of light absorbed when blue light is shone through the solution. The amount of light absorbed is expressed as an *absorbance value*. The more concentrated the solution, the higher the absorbance value.

(b) A plot of absorbance against concentration is known as a *calibration line*. Fig. 3.1 shows the calibration line obtained when the absorbance values of a series of standard solutions containing $FeSCN^{2+}(aq)$ were measured.

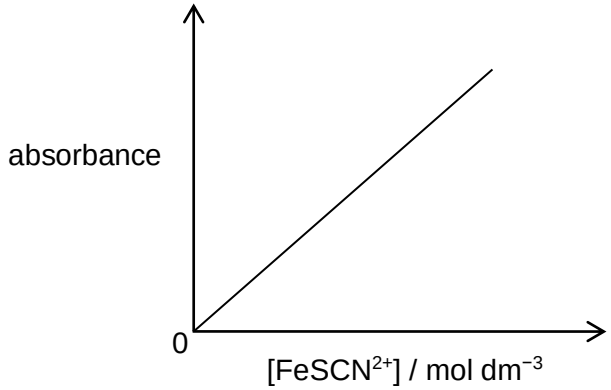


Fig. 3.1

One of the standard solutions was prepared by mixing 5.00 cm^3 of 0.200 mol dm^{-3} aqueous iron(III) nitrate, $Fe(NO_3)_3$, and 5.00 cm^3 of 2.00 x 10^{-3} mol dm^{-3} potassium thiocyanate, KSCN.

Show that the concentration of $FeSCN^{2+}(aq)$ is 1.00 x 10^{-3} mol dm^{-3} in this standard solution, and explain why the amount of aqueous iron(III) nitrate used must be in large excess in the standard solutions used to obtain the calibration line.

……

……[2]

To determine a value for K_c, known amounts of $Fe^{3+}(aq)$ and $SCN^-(aq)$ are mixed to produce a solution of $FeSCN^{2+}(aq)$. The absorbance of this solution is then measured. Using the calibration line in Fig 3.1, the concentration of $FeSCN^{2+}(aq)$ can be determined. This can be used to calculate the concentrations of $Fe^{3+}(aq)$ and $SCN^-(aq)$ in the equilibrium mixture and hence K_c.

The following equation represents the relationship between K_c and temperature in kelvin, T:

$$\ln K_c = -\frac{\Delta H^\ominus}{R}\left(\frac{1}{T}\right) + \frac{\Delta S^\ominus}{R}$$

R is the molar gas constant with a value of 8.31 J K^{-1} mol^{-1}.
$\Delta H^\ominus$ is the standard enthalpy change of reaction.
$\Delta S^\ominus$ is the standard entropy change of reaction.

A plot of ln K_c against 1/T can then be used to graphically determine $\Delta H^\ominus$ and $\Delta S^\ominus$.

(c) Plan an investigation to determine the effect of temperature, T, on the equilibrium constant, K_c of the reaction between $Fe^{3+}(aq)$ and $SCN^-(aq)$.

You may assume that you are provided with:
- 2.00 x 10^{-3} mol dm^{-3} iron(III) nitrate,
- 2.00 x 10^{-3} mol dm^{-3} potassium thiocyanate,
- a cuvette,
- access to a spectrophotometer and instructions for its use,
- the equipment normally found in a school or college laboratory.

In your plan you should include:
- an outline of how you would prepare a solution of $FeSCN^{2+}(aq)$,
- an outline of how you would determine the concentration of $FeSCN^{2+}(aq)$ in the solution at different temperatures,
- brief, but specific, details of how the concentrations, in mol dm^{-3}, of $Fe^{3+}(aq)$ and $SCN^-(aq)$ in the equilibrium mixture can be determined, and how you would use these concentrations to determine K_c for one of your chosen temperatures.

……

……

……

……

……

……

……

……

……

……

……

……

……

……

……

……

……

……

……

……

……

……

……

……

……

……

……

……[6]

(d) **(i)** Given that the reaction between $Fe^{3+}(aq)$ and $SCN^{-}(aq)$ is exothermic, sketch a graph you would expect to obtain using data obtained from **3(c)** on the axes in Fig 3.2.

Explain your answer.

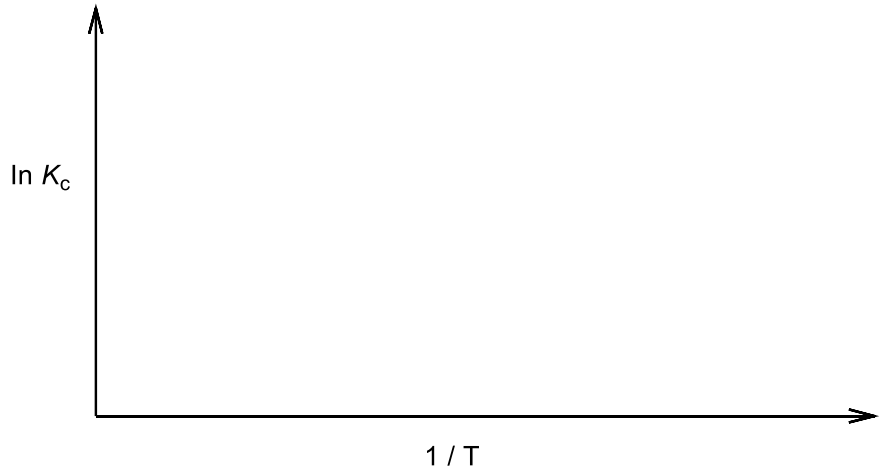


Fig. 3.2

Explanation ……..……..…....

……..…….....

[2]

(ii) Describe how would you use your graph in **3(d)(i)** to determine values for $\Delta H^{\ominus}$ and $\Delta S^{\ominus}$.

……..…….....

……..…….....

……..…….....

……..…….....

[2]

[Total: 13]

4 Investigation of some reactions involving manganese compounds

FA 7 is solid manganese dioxide, MnO_2.
FA 8 is an aqueous solution of hydrogen peroxide, H_2O_2.

In addition to access to the usual bench reagents, you are also provided with the following.

- aqueous sodium thiosulfate, $Na_2S_2O_3(aq)$

You will also need access to the **FA 1**, **FA 2** and **FA 4** solutions you used earlier.

Perform the tests described in Tables 4.1, 4.2 and 4.3 and record your observations. Test any gases produced.

Table 4.1

	test	observations
(a)	Place 1 cm depth of **FA 1** in a test-tube. Add a spatula of **FA 7** to this test-tube, followed by another 2 cm depth of aqueous potassium iodide. Filter the mixture into a clean test-tube and collect the filtrate. To the filtrate, add aqueous sodium thiosulfate dropwise, with shaking, until the solution first becomes colourless. Divide the filtrate into two portions.	
	To the first portion, add **FA 2** slowly until no further change is seen.	
	To the second portion, add aqueous ammonia slowly until no further change is seen.	[3]

(b) Suggest the role of **FA 7** in the reaction occurring in **4(a)**. Explain your answer with **two** observations from your experiment.

Role of **FA 7** ……

Explanation

……

……

……[2]

Table 4.2

	test	observations
(c)	Place 3 cm depth of **FA 8** into a test-tube. Add a spatula of **FA 7** to this test-tube.	[1]

(d) The mixture in **4(c)** was filtered and the filtrate divided into two portions. When **FA 2** and $NH_3(aq)$ were added to the two portions respectively, no precipitate was formed.

Explain why this observation shows that **FA 7** plays a different role in the reaction taking place in **4(c)** as compared to **4(a)**.

…….....

…….....[1]

(e) Hence, suggest the role of **FA 7** in the reaction in **4(c)** and justify your answer with an observation from your experiment.

Role of **FA 7**……..…

Explanation

…….....

…….....[1]

Question 4 continues on the next page.

Table 4.3

	test	observations
(f)	Place 1 cm depth of **FA 2** into a test-tube. Add 5 drops of aqueous potassium iodide and shake. Add **FA 4** to the mixture dropwise, with shaking, until 10 drops have been added. Then add 1 cm depth of aqueous barium nitrate slowly, with shaking, into the same test-tube. Filter the mixture into a boiling tube. Wash the residue thoroughly with deionised water. Discard the washings. Retain the residue for use in **4(g)**.	
(g)	Place the filter funnel with the residue from **4(f)** over a clean test-tube. Carefully add **FA 1** slowly until it covers the residue. Observe until no further changes are seen.	[2]

(h) When aqueous barium nitrate was added to the mixture in **4(f)**, a Mn-containing species was precipitated and collected as residue on filtration. When **FA 1** was then added to this residue in **4(g)**, two Mn-containing species, **X** and **Y**, were produced which could be found in the final residue and filtrate respectively.

Suggest the identities of **X** and **Y**.

Identity of **X** in the residue ……

Identity of **Y** in the filtrate …….. [1]

(i) A white precipitate is also formed after adding **FA 1** in **4(g)** but it is unlikely that you will have noticed it. Suggest the identity of this white precipitate.

…….. [1]

[Total: 12]

Qualitative Analysis Notes
[ppt. = precipitate]

(a) Reactions of aqueous cations

cation	***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., 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

(b) Reactions of aqueous anions

ion	*reaction*
carbonate, CO_3^{2-}	CO_2 liberated by dilute acids
chloride, Cl^-(aq)	gives white ppt. with Ag^+(aq) (soluble in NH_3(aq))
bromide, Br^-(aq)	gives pale cream ppt. with Ag^+(aq) (partially soluble in NH_3(aq))
iodide, I^-(aq)	gives yellow ppt. with Ag^+(aq) (insoluble in NH_3(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 NO → (pale) brown NO_2 in air)
sulfate, SO_4^{2-}(aq)	gives white ppt. with Ba^{2+}(aq) (insoluble in excess dilute strong acids)
sulfite, SO_3^{2-}(aq)	SO_2 liberated on warming with dilute acids; gives white ppt. with Ba^{2+}(aq) (soluble in dilute strong acids)

(c) Tests for gases

gas	*test and test result*
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 aqueous acidified potassium manganate(VII) from purple to colourless

(d) Colour of halogens

halogen	*colour of element*	*colour in aqueous solution*	*colour in hexane*
chlorine, Cl_2	greenish yellow gas	pale yellow	pale yellow
bromine, Br_2	reddish brown gas / liquid	orange	orange-red
iodine, I_2	black solid / purple gas	brown	purple