

NAME		Class	
------	--	-------	--

ST ANDREW'S JUNIOR COLLEGE



JC2 PRELIMINARY EXAMINATION

Chemistry (9729)

27 Aug 2019

Paper 4 Practical

2.5 hours

Candidates answer on the Question Paper

Additional Materials : As listed in the Confidential Instructions

: Insert

READ THESE INSTRUCTIONS FIRST.

Write your name and class on all the work you hand in.

Give details of the practical shift and laboratory in the boxes provided above.

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** the 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 **21** and **22**.

The number of marks is given in the brackets [] at the end of each question or part question.

Shift
Laboratory

For Examiner's use:

Question	Practical	Planning	Total
Marks			
	44	11	55

This document consists of **22** printed pages.

[Turn Over

- 1 To standardise a 0.03 mol dm^{-3} of iron(III) chloride solution and determine the concentration of sodium thiosulfate solution.

(a) Planning

FA1 is a standard solution containing 0.03 mol dm^{-3} of FeCl_3 solution.

You are to design an experiment to show how **FA1** is prepared using solid FeCl_3 .

You are provided with :

a solid sample of approximately 1.50 g of FeCl_3 ($M_r = 162.3$),

250 cm^3 volumetric flask and

the usual laboratory apparatus

In your plan, you should include details on:

- the mass of $\text{FeCl}_3(\text{s})$ you use with justification,
- the apparatus you would use, and
- the procedures which you would follow.

Do not carry out this plan as FA1 is provided for you to continue with 1(b).

.....

.....

.....

.....

.....

.....

.....

.....

.....

.....

- 1 (b) In addition to **FA1**, you are also provided with the following:

FA2 is 0.060 mol dm⁻³ potassium iodide, KI.

FA3 is aqueous sodium thiosulfate, Na₂S₂O₃.

When **FA1** is combined with **FA3**, a complex, [Fe(S₂O₃)₂]⁻, is formed.

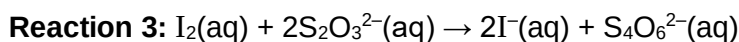
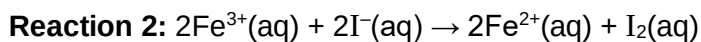


To 1 cm³ of **FA3** in a test-tube, add 2–3 drops of **FA1** and shake them thoroughly. State the colour of the complex formed.

Colour :

[1]

- (c) A student aimed to determine the concentration of **FA3** using volumetric analysis involving the use of **FA1** and **FA2**.



Using the above reactions, the student planned and carried out the following procedure.

1. Fill a 50.00 cm³ burette with **FA3** solution.
2. Using a 10 cm³ measuring cylinder, measure 5 cm³ of **FA1** and place it in a 100 cm³ conical flask.
3. Using another 10 cm³ measuring cylinder, measure 5 cm³ of **FA2** into the conical flask.
4. Run **FA3** from the burette into the conical flask. Near the endpoint, when the brown solution becomes pale, add 2 drops of starch.
5. Continue adding **FA3** slowly, the endpoint is reached when the solution **first becomes colourless**.
6. Record the titration results, to an appropriate level of precision, in an appropriate table and repeat the experiment once more.

- 1 (c) (i) Carry out the student's procedure **twice only** to obtain two titration readings. **Your titre values need not be consistent.**

Note: After the end point is reached, you may observe the appearance of the coloured mixture again. There is no need to titrate any further.

Record your titration in an appropriate table in the space below.

[2]

(ii) **Planning**

A teacher commented that it is appropriate for the student to use 5 cm³ instead of 25 cm³ of **FA1** for this titration. Justify the teacher's comment using suitable calculations.

Assume the concentration of **FA3** is 0.006 mol dm⁻³.

[2]

1 (c) (iii) Planning

Justify whether the amount of **FA2** that the student used in step **3** of the procedure in **1(c)** is sufficient to determine the concentration of **FA3**.

[1]

- (iv)** The student did the titration several times but could not get consistent titre values of **FA3**. Based on your observation during titration, suggest a reason why.

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

[1]

- (v)** Suggest another reason why the results obtained are not reliable.

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

[1]

- 1 (c) (vi) The student performed the titration and had a titration value of 18.50 cm^3 . Given the errors (uncertainties) associated with each reading using a measuring cylinder and burette are $\pm 0.1 \text{ cm}^3$ and $\pm 0.05 \text{ cm}^3$ respectively, calculate the maximum total percentage error (uncertainty) from the apparatus in the titration.

[3]

[Total: 14 marks]

2 To investigate the kinetics of the reaction between iron(III) ions and iodide ions

In this experiment, you are investigating the rate of reaction between iron(III) ions and iodide ions.

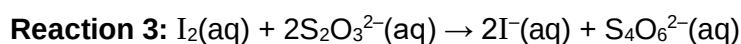
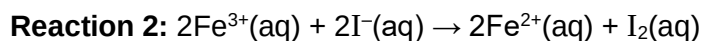
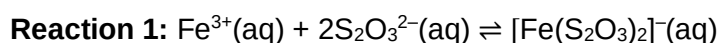
You are provided with:

FA1 is 0.03 mol dm^{-3} of FeCl_3 solution

FA2 is $0.060 \text{ mol dm}^{-3}$ potassium iodide, KI

FA3 is sodium thiosulfate, $\text{Na}_2\text{S}_2\text{O}_3$

The reaction is started by mixing a solution of iron(III) chloride with sodium thiosulfate, potassium iodide, and starch. The iodine, I_2 , produced in **reaction 2** reacts immediately with thiosulfate ions, $\text{S}_2\text{O}_3^{2-}$ in **reaction 3**.



When all the thiosulfate have been used, the iodine produced will turn starch indicator blue–black. The rate of the reaction can therefore be determined by finding the time for the blue–black colour to appear.

You are advised to read the instructions before starting any practical work and draw a table, in an appropriate format, for your results in the space on page 10.

For each of the five experiments, you will need to include the

- volume of **FA2**, V_{FA2} ,
- volume of water, V_{water} ,
- calculated squared volume of **FA2**, $(V_{\text{FA2}})^2$,
- reaction time, t to nearest second, and
- calculated rate in $\text{mol dm}^{-3} \text{ s}^{-1}$

Record all calculated values to 3 significant figures.

2 (a) Method

Experiment 1

1. Use the measuring cylinders to measure the following:
 - 20 cm³ of **FA1**
 - 20 cm³ of **FA3**
 - 1 cm³ of starch indicator
2. Using another measuring cylinder, measure 10 cm³ of **FA2**.
3. Pour the measured **FA1** and starch into a dry 100 cm³ beaker.
4. Pour in the **FA3**, followed by **FA2** immediately into the same beaker. Start the stopwatch on adding **FA2**.
5. Stir the mixture and place the beaker on the printed page on page 2 of the insert.
6. The mixture turns brown and then yellow before turning a blue–black colour. Stop the stopwatch when this blue–black colour appears and obscures the wordings.
7. Record the time to the nearest second in the space on page 10.
8. Wash the beaker thoroughly with water and carefully dry the beaker with paper towel.

Experiment 2

9. Repeat step 1 in **Experiment 1**.
10. Using a measuring cylinder, add 2 cm³ of **FA2**. Make up the volume to 10 cm³ using deionised water using another measuring cylinder.
11. Pour the measured **FA1**, starch and deionised water into a dry 100 cm³ beaker.
12. Pour in the **FA3**, followed by **FA2** immediately into the same beaker. Start the stopwatch on adding **FA2**.
13. Stir the mixture and place the beaker on the printed page on page 2 of the insert.

14. Stop the stopwatch when this blue–black colour appears and obscures the wordings.
15. Record the time to the nearest second in the space on page 10. The timing should not exceed 6 minutes.
16. Wash the beaker thoroughly with water and carefully dry the beaker with paper towel.

Experiments 3–5

Carry out three further experiments to investigate the effect of changing the concentration of **FA2** by altering the volume of aqueous potassium iodide, **FA2**, used.

You should use a volume of **FA2** that is at least 2 cm^3 and the total volume of the reaction mixture must always be 51 cm^3 .

- 2 (a) With reference to **1(b)**, consider the colour change observed when **FA2** was added to the solution prepared in step **6** of **Experiment 1** before the blue–black colour appeared. Suggest an explanation for the sequence of colour change in terms of the chemistry involved.

.....

.....

.....

.....

.....

.....

.....

.....

.....

[2]

- 2 (b) Show, by means of calculation, that the change in the concentration of I^- , $\Delta[\text{I}^-]$, which occurred when the blue-black colour appeared was $2.35 \times 10^{-3} \text{ mol dm}^{-3}$.

Assume the concentration of $\text{Na}_2\text{S}_2\text{O}_3$ to be $0.006 \text{ mol dm}^{-3}$.

[1]

(c) **Results**

The rate of the reaction can be calculated as shown.

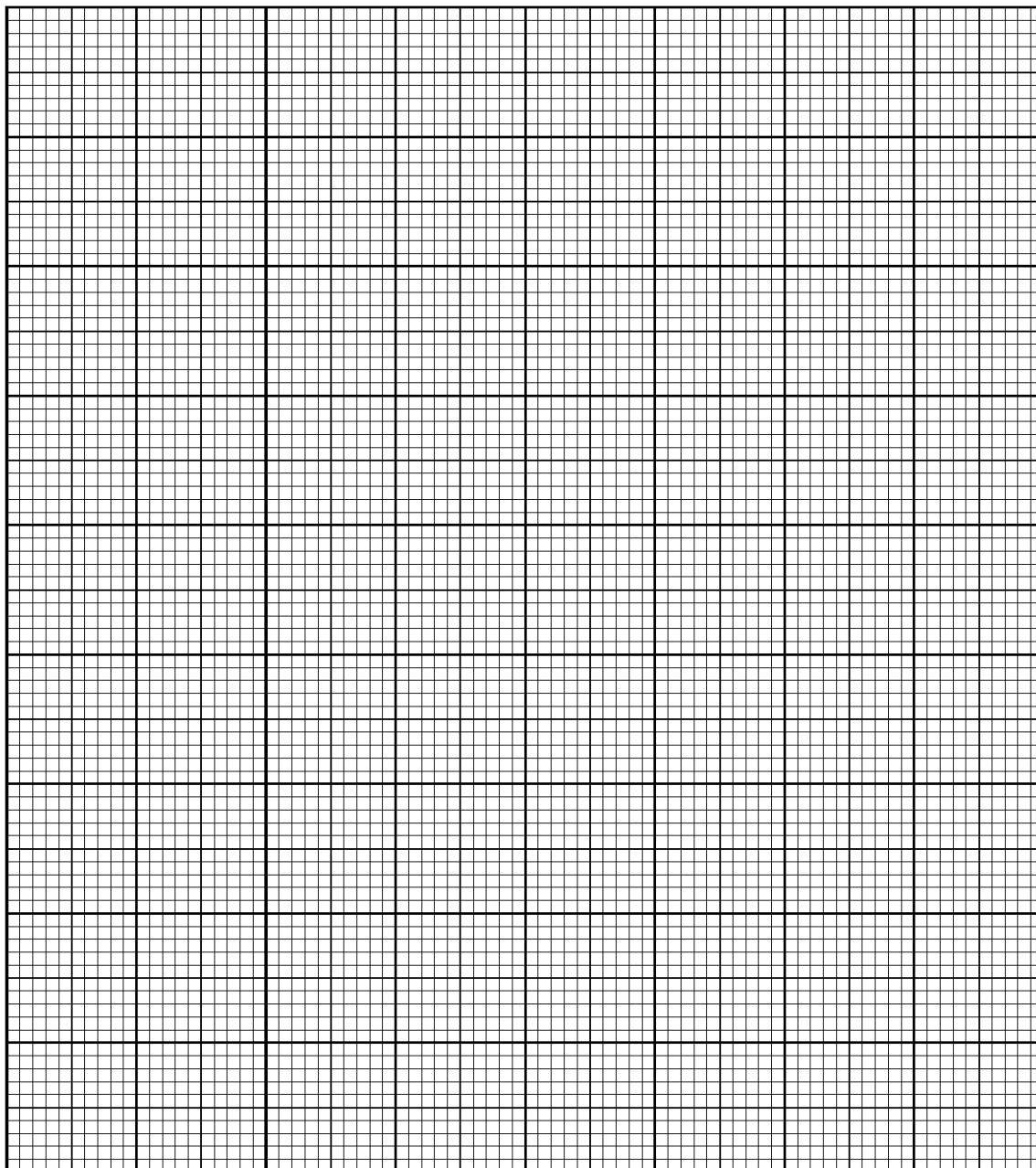
$$\text{rate} = \frac{\Delta[\text{I}^-]}{\text{reaction time}}$$

Calculate the rate of reaction for each experiment and complete your table.

[4]

- 2 (d) (i) Plot a graph of rate on the y-axis against $(V_{FA2})^2$ on the x-axis on the grid in **Figure 2.1**. Draw the best fit line taking account of all the plotted data points.

Figure 2.1



[4]

- 2 (d) (ii) Explain why the volume of **FA2** in each experiment can be used as the $[I^-]$.

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

- (iii) Deduce the order of reaction with respect to $[I^-]$. Use evidence from your graph to support your deduction.

Order of reaction with respect to $[I^-]$: [1]

- (e) (i) Use your graph and the formula in **2(c)** to calculate the time that the reaction would have taken if 5.0 cm^3 of **FA2** had been used. Show your working clearly.

time = [2]

- 2 (e) (ii) Calculate the initial concentration of iron(III) ions and the initial concentration of iodide ions in the reaction mixture if 5.0 cm³ of **FA2** had been used.

initial [Fe³⁺] =
 initial [I⁻] = [1]

- (iii) Given that the reaction is first order with respect to [Fe³⁺], calculate the rate constant.

rate constant = [2]

- (f) At the end of Experiment 1, a student washed the beaker and used it immediately for Experiment 2. State and explain how the student's action would affect the time *t* for Experiment 2.

.....

 [2]

- 2 (g) List 1 possible source of experimental errors and suggest the corresponding improvement to reduce the experimental error.

.....

.....

.....

.....

.....

.....

.....

[2]

[Total: 22 marks]

3 Qualitative Analysis

You are provided the following reagents and the *Qualitative Analysis Notes* on page 21 and 22.

FA1 is 0.03 mol dm^{-3} of FeCl_3 solution

FA4 is 6% volume H_2O_2

FA5 is $0.00215 \text{ mol dm}^{-3}$ of X^- solution

(a) You are to perform the tests described in **Table 3.1** and record your observations in the same table.

Your answers should include

- details of colour changes throughout the tests and precipitates formed,
- the identity of any gases evolved, and details of the test used to identify each one.

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

Marks are not given for chemical equations.

No additional or confirmatory tests for ions present should be attempted.

Table 3.1

Test	Observations
i. To 5 cm^3 of FA1, add aqueous silver nitrate in excess. Filter the mixture. Separate the filtrate into three test tubes for (ii), (iii) and (iv). Place the filter funnel containing the residue on a new test tube, add aqueous ammonia over the residue. Add HNO_3 dropwise to the filtrate until no more change.	

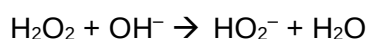
Test	Observations
ii. To a new test tube containing 1 cm³ of FA4, add a few drops of filtrate from the first test tube from (i). Wait and observe the effervescence. Test for the identity of the gas.	
iii. To the second test tube containing 1 cm³ of filtrate from (i), add aqueous sodium hydroxide till excess. Add 1 cm³ of FA4.	
iv. To the last test tube containing 1 cm³ of filtrate from (i), add 1 cm³ of FA5.	
v. To a new test tube containing a fresh sample of FA1, test the pH of the solution with a Universal Indicator paper. Add aqueous ammonia dropwise. Add FA1 dropwise to dissolve the precipitate formed. Stop adding FA1 once the precipitate disappears.	

- 3 (b) With reference to the observation made in test (i), identify the ppt and explain its solubility in aqueous ammonia.

.....

[2]

- (c) In the presence of strong base,



Electrode Reaction	$E^\ominus/\text{V}$
$\text{Fe}^{3+} + \text{e} \rightleftharpoons \text{Fe}^{2+}$	+0.77
$\text{Fe}(\text{OH})_3 + \text{e} \rightleftharpoons \text{Fe}(\text{OH})_2$	-0.56
$\text{O}_2 + 2\text{H}_2\text{O} + 2\text{e} \rightleftharpoons \text{H}_2\text{O}_2$	+0.68
$\text{H}_2\text{O}_2 + 2\text{H}^+ + 2\text{e} \rightleftharpoons 2\text{H}_2\text{O}$	+1.23
$\text{O}_2 + \text{H}_2\text{O} + 2\text{e} \rightleftharpoons \text{HO}_2^- + \text{OH}^-$	-0.08

With reference to the electrode potential given, explain the difference in the observation for effervescence made when **FA4** is added in test (ii) and (iii).

.....

[2]

- (d) Write an equation to explain the colour of the Universal Indicator paper on addition of **FA1** before adding aqueous ammonia.

.....

[1]

- 3 (e) The pH of the resultant solution in test (v) is 7. Explain the chemistry behind the changes of pH in test (v) when aqueous ammonia is added dropwise followed by FA1.

.....

.....

.....

.....

[2]

- (f) Predict what happen when the resultant solution in test (v) is added to phenol.

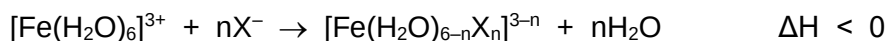
.....

[1]

3 (g) Planning

Thermometry is sometimes used to determine the number of ligands attached to the central metal atom or ion. One of such monodentate ligand is X^- present in **FA5**.

A student performed an experiment to determine the structural formula of the hexa-coordinated complex.



He used varying volumes of **FA1** and **FA5** only, while keeping the total volume of the mixture at 80 cm^3 . He recorded the change in temperature.

(i) Outline how you would:

- determine the effect of changing the volumes of **FA1** and **FA5** on the temperature change of the experiment and hence,
- vary the volumes of **FA1** and **FA5** to form the needed hexa-coordinated complex, $[\text{Fe}(\text{H}_2\text{O})_{6-n}X_n]^{3-n}$.

You are provided with the same solutions, which were used in **3(a)** described. **There is no need to perform the experiment.**

No details regarding the use of specific apparatus are required.

.....

.....

.....

.....

.....

.....

.....

.....

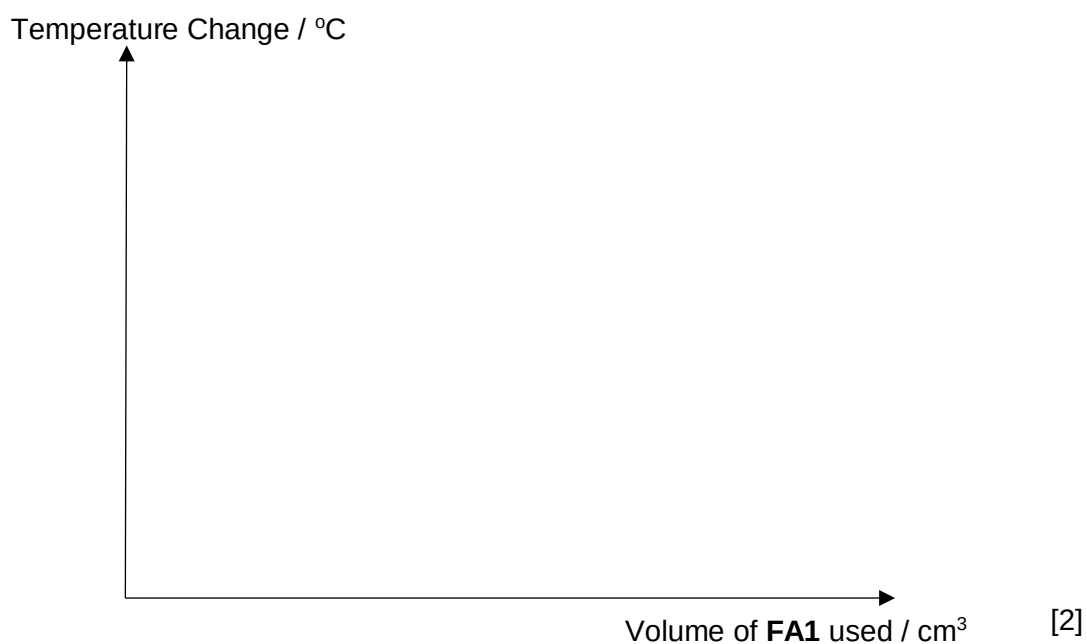
.....

.....

[3]

- (ii) The student plotted a graph and concluded that $n=1$ in the structural formula of $[\text{Fe}(\text{H}_2\text{O})_{6-n}\text{X}_n]^{3-n}$. On the axes of **Figure 3.1**, sketch a graph that the student would obtain from your experiment in **3(g)(i)** to draw this conclusion. Indicate the volume of **FA1** that the student would use to reach this conclusion on the x-axis of the graph.

Figure 3.1



[Total: 19 marks]

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 anions

<i>anion</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 pale 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 dilute strong acids)

(c) 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 aqueous acidified potassium manganate(VII) from purple to colourless

(d) Colour of halogens

<i>halogen</i>	<i>colour of element</i>	<i>colour in aqueous solution</i>	<i>colour in hexane</i>
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