Name:		Class:	

ST ANDREW'S JUNIOR COLLEGE

JC 2 PRELIMINARY EXAMINATION

CHEMISTRY **9729/04**

Paper 4 Practical **16 Aug 2021**

2 hours 30 minutes

Additional Materials: Qualitative Analysis Notes

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 a soft pencil for any diagrams or graphs.
Do not use staples, paper clips, highlighters, 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.

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

Shift
Laboratory

For Examiner's Use	
1	/13
2	/18
3	/10
4	/14
Total	/55

This document consists of **21** printed pages including this page.

1. Determination of the value of *x* in the oxyanion of iodine, IO_x^-

Iodine is able to form more than one oxyanion, IO_x^-, polyatomic ions that contain oxygen, each containing a different number of oxygen atoms.

In this experiment, you will determine the value of ***x*** in the oxyanion of iodine, IO_x^-. You will first react IO_x^- ions with an excess of iodide ions, I^-, to form iodine, I_2 as shown in equation 1.

equation 1 $$IO_x^- + \mathbf{y}\, I^- + \mathbf{z}\, H^+ \rightarrow \frac{1+y}{2} I_2 + \frac{z}{2} H_2O$$

where **x**, **y** and **z** are all integers

The amount of iodine produced will then be determined by titration with thiosulfate ions, $S_2O_3^{2-}$.

$$I_2 + 2S_2O_3^{2-} \rightarrow 2I^- + S_4O_6^{2-}$$

FA 1 is a solution containing 0.0150 mol dm^{-3} IO_x^- ions.

FA 2 is dilute sulfuric acid, H_2SO_4.

FA 3 is 1.00 mol dm^{-3} potassium iodide, KI.

FA 4 is 0.100 mol dm^{-3} sodium thiosulfate, $Na_2S_2O_3$.

starch indicator

(a) Procedure

1. Fill the burette with **FA 4**.
2. Pipette 25.0 cm^3 of **FA 1** into a conical flask.
3. Use a measuring cylinder to add 25 cm^3 of **FA 2** to the conical flask.
4. Use another measuring cylinder to add 10 cm^3 of **FA 3** to the conical flask. The solution will turn brown as iodine is produced.
5. Add **FA 4** from the burette until the solution in the conical flask turns yellow.
6. Add 5 drops of starch indicator to the conical flask. The solution will turn blue-black.
7. Continue to add more **FA 4** from the burette until the blue-black colour just disappears. This is the end-point of the titration.
8. Record your titration results, to an appropriate level of precision, in the space provided on page 3.
9. Repeat points 2 to 7 until consistent results are obtained.

1 (a) Results

M1

M2

M3

[3]

(b) From your titrations, obtain a suitable volume of **FA 4**, to be used in your calculations. Show clearly how you obtained this value. **[2]**

M4

M5

volume of **FA 4** = ……..……..

(c) (i) Calculate the number of moles of iodine formed when **FA 1** reacts with **FA 3**. **[1]**

M6

number of moles of I_2 = ……..……..

1 (c) (ii) Calculate the number of moles of IO_x^- ions in 25.0 cm^3 of **FA 1**. **[1]**

M7

number of moles of IO_x^- ions = ……..……..

(iii) Using equation 1 and your answers in **1(c)(i)** and **1(c)(ii)**, calculate the value of **y**. Show your working. **[1]**
(Note that **y** is an odd integer such as 1, 3, 5, 7 etc.)

M8

y = …….

1 (c) (iv) Use your value of **y** in **1(c)(iii)** and considering the number of electrons transferred, determine the oxidation number of I in IO_x^- ion. Hence, determine the value of **x** in IO_x^- ion. **[2]**

M9

M10

oxidation number of I in IO_x^- ion = ……

x = ……

(v) A student suggested that a more accurate value of **x** could be obtained if a 10.0 cm^3 pipette was used to measure **FA 3** rather than the measuring cylinder. **[1]**

State whether you agree with the student. Explain your answer.

……...

……...

……...

M11

1 (c) (vi) Explain how the titre volume of $Na_2S_2O_3$ will change when the value of **x** in IO_x^- is greater. **[1]**

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……... M12

……...

(d) Chlorine is also able to form more than one oxyanion, ClO_x^-. **[1]**

Similar to IO_x^-, oxyanions of chlorine are also oxidising agents. The table below shows the standard electrode potential of different chlorine oxyanions.

Electrode Reaction	E^e / V
$ClO^- + H_2O + 2e^- \rightleftharpoons Cl^- + 2OH^-$	+0.89
$ClO_2^- + 2H_2O + 4e^- \rightleftharpoons Cl^- + 4OH^-$	+0.78
$ClO_3^- + 3H_2O + 6e^- \rightleftharpoons Cl^- + 6OH^-$	+0.63
$ClO_4^- + 4H_2O + 8e^- \rightleftharpoons Cl^- + 8OH^-$	+0.56
$Cl_2 + 2e^- \rightleftharpoons 2Cl^-$	+1.36

However, unlike IO_x^-, the value of **x** in the oxyanion of chlorine, ClO_x^- cannot be determined by reacting ClO_x^- with its corresponding halide, Cl^-.

Use the data given in the above table, explain why it is not possible to determine the value of **x** in the oxyanion of chlorine, ClO_x^-, with the reaction with Cl^-, under standard conditions.

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……... M13

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[Total: 13]

2. Investigation of the effect of $[S_2O_8^{2-}]$ on the rate of reaction between I^- and $S_2O_8^{2-}$

Sulfur forms the peroxodisulfate anion, $S_2O_8^{2-}$. This ion can oxidise iodide ions, I^-, to iodine, I_2, as shown in the equation.

$$2I^-(aq) + S_2O_8^{2-}(aq) \rightarrow I_2(aq) + 2SO_4^{2-}(aq)$$

You will carry out a series of experiments to investigate how the rate of this reaction is affected by changing the concentration of the solutions.

The rate can be measured by adding thiosulfate ions, $S_2O_3^{2-}$, and starch indicator. As the reaction between $S_2O_8^{2-}$ and I^- occurs, iodine is produced. The I_2 produced reacts immediately with the thiosulfate.

$$I_2(aq) + 2S_2O_3^{2-}(aq) \rightarrow 2I^-(aq) + S_4O_6^{2-}(aq)$$

When all the thiosulfate has reacted, the iodine will remain in the mixture and cause the starch indicator to turn blue-black. The rate of reaction may be determined by measuring the time taken for the reaction mixture to turn blue-black.

FA 5 is 0.0200 mol dm^{-3} potassium peroxodisulfate, $K_2S_2O_8$.

FA 6 is 1.00 mol dm^{-3} potassium iodide, KI.

FA 7 is 0.00500 mol dm^{-3} sodium thiosulfate, $Na_2S_2O_3$.

starch indicator

(a) (i) Procedure

Experiment 1

1. Use the marker to label one of the 100 cm^3 beakers '**A**' and the other 100 cm^3 beaker '**B**'.
2. Use the marker to label one of the measuring cylinders '**A**' and the other measuring cylinder '**B**'.
3. Use the measuring cylinder **A** to transfer 20.0 cm^3 of **FA 5** into beaker **A**.
4. Use the measuring cylinder **B** to add 20.0 cm^3 of **FA 6** into beaker **B**.
5. Use the measuring cylinder **B** to add 10.0 cm^3 of **FA 7** to beaker **B**.
6. Add 10 drops of starch indicator to beaker **B**.
7. Add the contents of beaker **A** to beaker **B**. Start the stopwatch during this addition.

8. Stir the mixture **once** and place the beaker on a white tile.
9. Stop the stopwatch when the solution **first** turns blue-black.
10. Record this reaction time to the nearest second.
11. Wash out both beakers and shake to remove excess water.

Experiment 2

1. Use the measuring cylinder **A** to transfer 10.0 cm^3 of **FA 5** into beaker **A**.
2. Use the measuring cylinder labelled **A** to transfer 10.0 cm^3 of distilled water into beaker **A**.
3. Use the measuring cylinder **B** to add 20.0 cm^3 of **FA 6** into beaker **B**.
4. Use the measuring cylinder **B** to add 10.0 cm^3 of **FA 7** to beaker **B**.
5. Add 10 drops of starch indicator to beaker **B**.
6. Add the contents of beaker **A** to beaker **B**. Start the stopwatch during this addition.
7. Stir the mixture once and place the beaker on a white tile.
8. Stop the stopwatch when the solution **first** turns blue-black.
9. Record this reaction time to the nearest second.
10. Wash out both beakers and shake to remove excess water.

Experiments 3 to 5

Choose suitable volumes that will enable you to investigate further the effect of changing the concentration of potassium peroxodisulfate, **FA 5**, on the rate of the reaction.

Note that the total volume of **FA 5** and distilled water must always be constant. Do not use a volume of **FA 5** that is less than 6.0 cm^3.

The relative rate of the reaction can be calculated as shown.

$$\text{Relative rate} = \frac{900}{\text{reaction time}}$$

In the space provided, record in a single table for each of your five experiments:

- volume of **FA 5**
- volume of distilled water
- the reaction time
- the relative rate of the reaction

2 (a) (i) Results

M14

M15

M16

M17

M18

[5]

(ii) Using data from **Experiments 1** and **2**, show by calculation that the volume of potassium peroxodisulfate, **FA 5**, used was directly proportional to the concentration of peroxodisulfate. You can ignore the volume of starch used. **[2]**

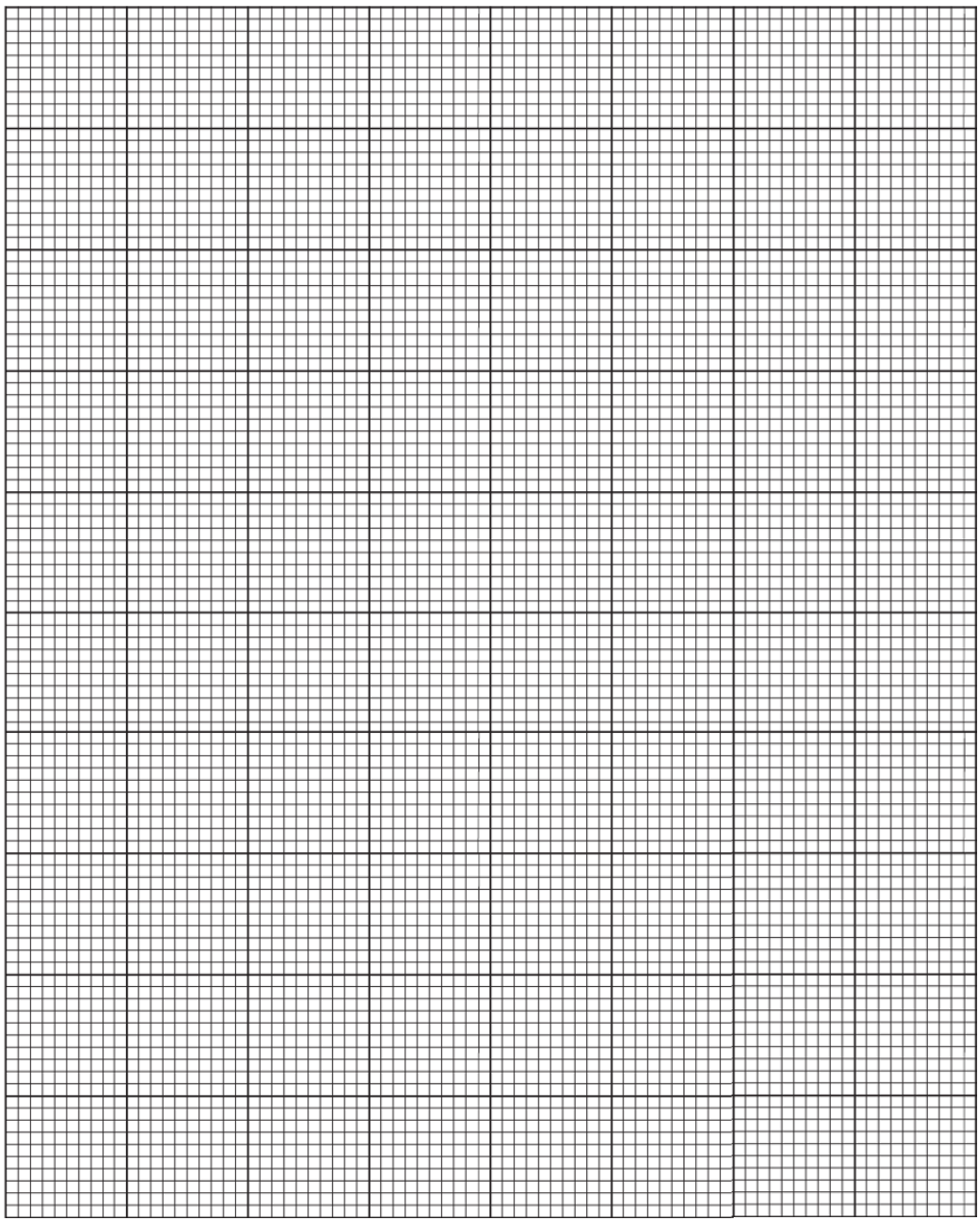
M19

M20

For Examiner's Use

2 (b) (i) Use the grid below to plot a graph of rate against volume of **FA 5**. **[3]**

Include the origin in your plot.



M21

M22

M23

2 (b) (ii) Explain, by referring to your graph or your table of results, how the rate of reaction is affected by an increase in the concentration of potassium peroxodisulfate, **FA 5**. **[1]**

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(iii) Use your graph to calculate the reaction time you would expect to measure if you carried out an experiment using 5.00 cm^3 of **FA 5**. Show on the graph how you obtained your answer. **[2]**

M25

M26

reaction time = …….

(iv) Assume that the error in the time measured for each reaction was ±0.5 s in total. Calculate the percentage error in the reaction time you measured in **Experiment 1**. Show your working. **[2]**

M27

M28

percentage error = …….

2 (b) (v) A student suggested that this error could be reduced if 0.00200 mol dm^{-3} sodium thiosulfate was used in place of **FA 7**. Do you agree with this student? Explain your answer. **[1]**

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……... M29

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(vi) A student carries out the same investigation as in **2(a)(i)** but the solutions are mixed in a different order. The student places **FA 5** and an appropriate volume of distilled water in beaker **A**. He then added **FA 7** and starch into beaker **A**. He added **FA 6** last and started the stopwatch immediately. **[2]**

Tick the box for the statement you consider correct. Explain your answer.

The student's method is better than that in **(a)**.	☐
The two methods are equally good.	☐
The student's method is not as good as that in **(a)**.	☐

You may use the data in the table below to explain your answer.

Electrode Reaction	**$E^{\ominus}$ / V**
$S_2O_8^{2-} + 2e^- \rightleftharpoons 2SO_4^{2-}$	+2.01
$S_4O_6^{2-} + 2e^- \rightleftharpoons 2S_2O_3^{2-}$	+0.09
$I_2 + 2e^- \rightleftharpoons 2I^-$	+0.54

Reason: …… M30

……... M31

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[Total: 18]

3. Planning

A student suggested that the temperature at which the experiment was carried out would also affect the rate of the reaction.

(a) Plan an investigation, based on the experiment described in **2(a)(i)**, to determine the effect of temperature on the rate of reaction. **[4]**

You may assume that you are provided with the same reagents as experiment **2(a)(i)** as well as the equipment normally found in a school laboratory

Give a step-by step description of how you would carry out the experiment by considering

- what you would keep constant in all the experiments,
- a suitable number of experiments you would do, and a reasonable temperature range,
- the apparatus that you would use in addition to that specified in **2(a)(i)**
- the procedure that you would follow and the measurements that you would take
- how you would determine the rate for each experiment.

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…… M34

…… M35

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(b) The activation energy, E_A, for the reaction can be obtained using the "Arrhenius Equation".

$$k = Ae^{\frac{-Ea}{RT}}$$

where k is the rate constant at reaction temperature T in Kelvin, A is the frequency factor and R is the ideal gas constant (8.314 J $K^{-1}mol^{-1}$). A can be regarded as a constant for this experiment.

Taking the natural logarithm of the "Arrhenius equation", gives the following equation.

$$\ln k = \ln A - \frac{E_a}{R}\left(\frac{1}{T_K}\right)$$

(i) Given that rate = k [reactants], state the relationship between k and the time taken for the reaction mixture to turn blue-black. **[1]**

…… M36

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3 (b) (ii) Sketch a graph that you would use to determine the activation energy, E_A, for the reaction on the axes in **Figure 3.1**. Describe how you would use your graph to determine the value of E_A. **[3]**

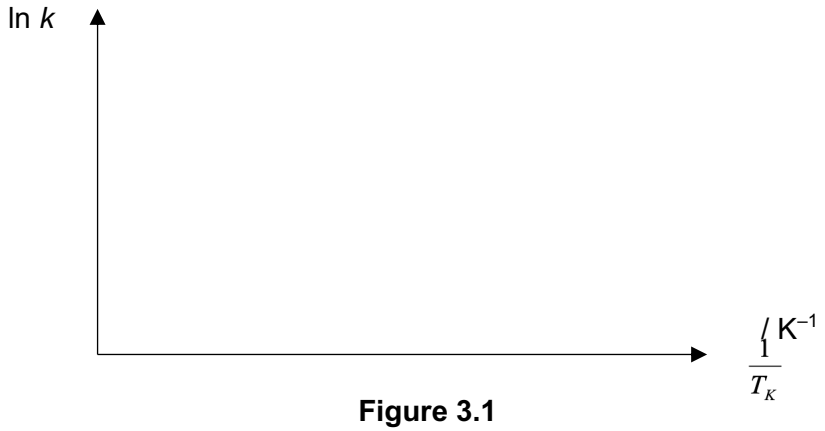

Figure 3.1

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……... M38

……... M39

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(iii) Briefly describe how you would use results obtained from **3(a)** to determine all necessary values in order to plot the graph in **3(b)(ii)**. You do **not** need to calculate any of the calculations. **[2]**

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……... M40

……... M41

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[Total: 10]

4. Investigation of some inorganic and organic reactions

(a) **FA 8** is a mixture that contains two cations and two anions.

Distilled water was added to **FA 8**, where the mixture was stirred and then filtered. You are provided with the dried residue, **FA 9**, and the filtrate, **FA 10**, from this filtration process.

Carry out the tests described in **Table 4.1** and carefully record your observations in the table.

Unless otherwise stated, the volumes given below are approximate and should be estimated rather than measured. Test and identity any gas evolved.

Table 4.1

	tests	observations
1.	(a) Add one spatula of **FA 9** into a dry boiling tube and add dilute hydrochloric acid until no further reaction occurs.	
	(b) To 1 cm depth of the solution from **test 1(a)** in another test tube, add aqueous sodium hydroxide dropwise until no further change is seen. **Keep the remaining solution from test 1(a) for 4(b)(iii).**	

M42

M43

M44

2.	To 1 cm depth of **FA 10** in a test tube, add aqueous ammonia dropwise until no further change is seen. Note: There is no need to perform this test.	Blue ppt formed, soluble in excess aqueous ammonia to form a dark blue solution.
3.	To another 1 cm depth of **FA 10** in a boiling tube, add a piece of aluminium foil and 2 cm depth of aqueous sodium hydroxide. Heat the mixture. Cool the mixture and filter.	
4.	Use a glass rod to transfer the residue in **test 3** into a test tube and add 2 cm^3 of **FA 2**.	

M45

M46

M47

[6]

(b) (i) Identify the cation that is **definitely** present in **FA 8** and **two possible** identities for the other cation present in **FA 8**. **[1]**

Cation that is **definitely** present: ……

Two possible identities for the other cation present: ……

M48

4 (b) (ii) Describe a test, which will allow you to determine which of the two possible cations that you listed in **4(b)(i)** is present in **FA 8**. **[1]**

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……... M49

……...

(iii) Perform the test you describe in **4(b)(ii)** using the remaining solution from **test 1(a)** of **Table 4.1**. Record your observations and hence deduce the other identity of the cation present in **FA 8**. **[1]**

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……... M50

Cation is ……

(iv) Given that **FA 8** does not contain nitrite ions, NO_2^-, or sulfite ions, SO_3^{2-}, identify the two anions that are present in **FA 8**. Use evidence from your observations in **4(a)** to support your deduction. **[2]**

Anion: ……

Evidence: ……

……... M51

Anion: ……

Evidence: ……

……... M52

4 (c) (i) Write an equation to show the change in observation in **test 4**. **[1]**

……...

M53

(ii) The solution prepared in **test 4** can be used to prepare a reagent to test for the carbonyl functional group.
Use this information and the observation given in **Table 4.2** to plan an experiment using the reagent prepared in **test 4** to confirm the presence of aldehyde in 1 cm^3 of C_8H_8O.

In your plan you should include brief details of:

- the quantity and the identity of the reagent to be used
- the condition and apparatus you would use

Table 4.2

Test	Observation
	Brick red ppt is formed.

M54

[1]

(iii) A student carried out the plan in **4(c)(ii)** and obtained the expected observation to confirm the functional group in C_8H_8O. Draw the structure of C_8H_8O. **[1]**

M55

[Total: 14]

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

anion	***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 $\rightarrow$ (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