Class | Adm No

Candidate Name: _____

Shift
Laboratory

2024 Preliminary Examination
Pre-University 3

H2 CHEMISTRY **9729/04**

Paper 4 Practical **27 Aug 2024**

2 hours 30 minutes

Candidates answer on the Question paper.

READ THESE INSTRUCTIONS FIRST

Do not turn over this question paper until you are told to do so

Write your name, class and admission number 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 at the back of the Question Paper.

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.

Question	1	2	3	4	Total
Marks	/14	/18	/14	/9	/55

1 Determination of enthalpy change of reaction

FA 1 is solid sodium hydrogencarbonate, $NaHCO_3$

FA 2 is 1.50 mol dm^{-3} sulfuric acid, H_2SO_4 **(also required in both question 2 and 3)**

Sodium hydrogencarbonate is commonly known as baking soda and is used as a reagent in various reactions. It is soluble in water according to equation 1.

equation 1 $NaHCO_3(s) + aq \longrightarrow Na^+(aq) + HCO_3^-(aq)$ ΔH_1

It can also react with acids in both solid and aqueous state.

equation 2 $2NaHCO_3(s) + H_2SO_4(aq) \longrightarrow Na_2SO_4(aq) + 2H_2O(l) + 2CO_2(g)$ ΔH_2

equation 3 $2NaHCO_3(aq) + H_2SO_4(aq) \longrightarrow Na_2SO_4(aq) + 2H_2O(l) + 2CO_2(g)$ ΔH_3

In this question, you will carry out experiments to determine ΔH_1 and ΔH_2, then use Hess's Law to determine ΔH_3.

(a) In this experiment, you will determine the maximum temperature change when a known mass of solid sodium hydrogencarbonate, **FA 1**, reacts with sulfuric acid, **FA 2**. Then, you will determine ΔH_2.

In an appropriate format in the space provided below, record

- all weighings to an appropriate level of precision,
- all values of temperature to an appropriate level of precision.

Procedure

1. Weigh the capped bottle containing **FA 1**.
2. Place one polystyrene cup inside a second polystyrene cup. Place these in a glass beaker to prevent them from tipping over.
3. Use a measuring cylinder to transfer 25 cm^3 of **FA 2** into the first polystyrene cup. Cover the cup with the lid provided.
4. Stir **FA 2** in the cup gently with the thermometer. Read and record its temperature.
5. Transfer all the **FA 1** to the polystyrene cup. Stir the mixture.
6. Continue to stir the mixture. Observe the temperature and record the value that shows the maximum change from the initial temperature.
7. Reweigh the empty bottle and its cap.
8. Record the maximum temperature change and the mass of **FA 1** used.

(i) Results

[5]

(ii) Calculate the heat change, q, using the values you obtained in **(a)(i)**.

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

heat change = ... [1]

(iii) Hence, determine the enthalpy change of reaction, ΔH_2.

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

ΔH_2 = ... [2]

(iv) Calculate the percentage error of the temperature change when using the thermometer.

percentage error = ... [1]

(v) Suggest the effect on the value for ΔT when 50 cm^3 of **FA 2** is used instead of 25 cm^3 in the experiment.

……

……[1]

(b) A second experiment was conducted to find out ΔH_1 and the results of the experiment are presented in Table 1.1.

Table 1.1

mass of **FA 1** used / g	4.00
volume of water used / cm^3	50.0
initial temperature of water / °C	29.4
minimum temperature reached / °C	21.0

(i) Use the information in Table 1.1 to determine a value of ΔH_1.

ΔH_1 = …… [2]

(ii) Use Hess's Law and your answers from **(a)(iii)** and **(b)(i)** to determine a value for ΔH_3 for the reaction shown in equation 3.

ΔH_3 = …… [2]

[Total: 14]

2 Investigation of the kinetics of the Mn^{2+} catalysed reaction between MnO_4^- and $C_2O_4^{2-}$

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

FA 4 is 0.200 mol dm^{-3} sodium ethanedioate, $Na_2C_2O_4$

FA 5 is 0.0100 mol dm^{-3} sodium thiosulfate, $Na_2S_2O_3$

FA 6 is 0.100 mol dm^{-3} potassium iodide, KI

FA 7 is aqueous Mn^{2+}

Starch indicator

FA 3, FA 4 and FA 7 are also required in question 3.

Ethanedioate ions, $C_2O_4^{2-}$, can be oxidised by manganate(VII) ions, MnO_4^- ions in acidic medium based on the equation below.

$$2MnO_4^-(aq) + 5C_2O_4^{2-}(aq) + 16H^+(aq) \longrightarrow 2Mn^{2+}(aq) + 10CO_2(g) + 8H_2O(l)$$

However, in the absence of a catalyst, the reaction proceeds slowly. The reaction can be catalysed through autocatalysis when Mn^{2+} is formed, or when Mn^{2+} is added at the start of the reaction.

In this experiment, you will prepare investigate the kinetics of a reaction mixture containing **FA 3** and **FA 4**. At timed intervals, you will draw 10 cm^3 aliquots from the reaction mixture. The concentration of MnO_4^- ions in each aliquot will then be determined through titration against $S_2O_3^{2-}$ after adding each aliquot to an excess of **FA 6**.

Acidified potassium manganate(VII) ions oxidises I^- ions according to the following reaction:

$$2MnO_4^-(aq) + 10I^-(aq) + 16H^+(aq) \longrightarrow 2Mn^{2+}(aq) + 5I_2(aq) + 8H_2O(l)$$

The reaction between iodine from the reaction mixture and $S_2O_3^{2-}$ solution is given below.

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

(a) Preparation and titration of the reaction mixture

Notes: You will perform each titration **once** only. Great care must be taken that you do not overshoot the end point.

Once you have started the stopwatch, it must continue running for the duration of the experiment. You must **not** stop it until you have finished this experiment.

In an appropriate format in the space provided in page 7, prepare a table to which to record for each aliquot

- the time of transfer, *t*, in minutes and seconds,
- the decimal time, t_d, in minutes, to 0.1 min, for example if *t* = 4min 33s then t_d = 4min + 33/60 min = 4.6min,
- the burette readings and the volume of **FA 5** added.

1. Fill a burette with **FA 5**.

2. Label each of the boiling tubes **1** to **5**. Using a measuring cylinder, add approximately 10 cm^3 of **FA 6** to each of these boiling tubes.

3. Using appropriate measuring cylinders, transfer 50.0 cm^3 of **FA 4**, 5.0 cm^3 of **FA 2 ,** 5.0 cm^3 of **FA 7** and 40.0 cm^3 of deionised water into a conical flask. Label the conical flask **reaction mixture**.

4. Using an appropriate measuring cylinder, transfer 25.0 cm^3 of **FA 3** to the conical flask labelled **reaction mixture**. Start the stopwatch and swirl the mixture thoroughly to mix its contents.

5. At approximately t = 1 min, use a 10 cm^3 pipette to remove a 10.0 cm^3 aliquot of the reaction mixture. **Immediately** transfer this aliquot into the boiling tube labelled **1** and swirl the mixture. Note and record the transfer time (in minutes and seconds, to the nearest second) when half of the reaction mixture has emptied from the pipette.

6. Pour the contents of the boiling tube labelled **1** into a clean 250 cm^3 conical flask. Transfer all washings from the boiling tube into the conical flask.

7. Titrate the iodine in this solution with **FA 5** until the solution turns pale yellow. Then add about 1 cm^3 of starch indicator. Continue the titration. The end-point is reached when the blue-black colour is discharged. Record your result.

8. Repeat steps 5 to 7 with around three-minute interval until a total of **five** aliquots have been titrated and their results recorded.

Results

[3]

(b) **(i)** On the grid in **Fig. 2.1**, plot a graph of **volume of sodium thiosulfate**, **FA 5**, on the *y*-axis, against decimal time, t_d, on the *x*-axis. You should draw the most suitable line that takes into account all of your plotted points.

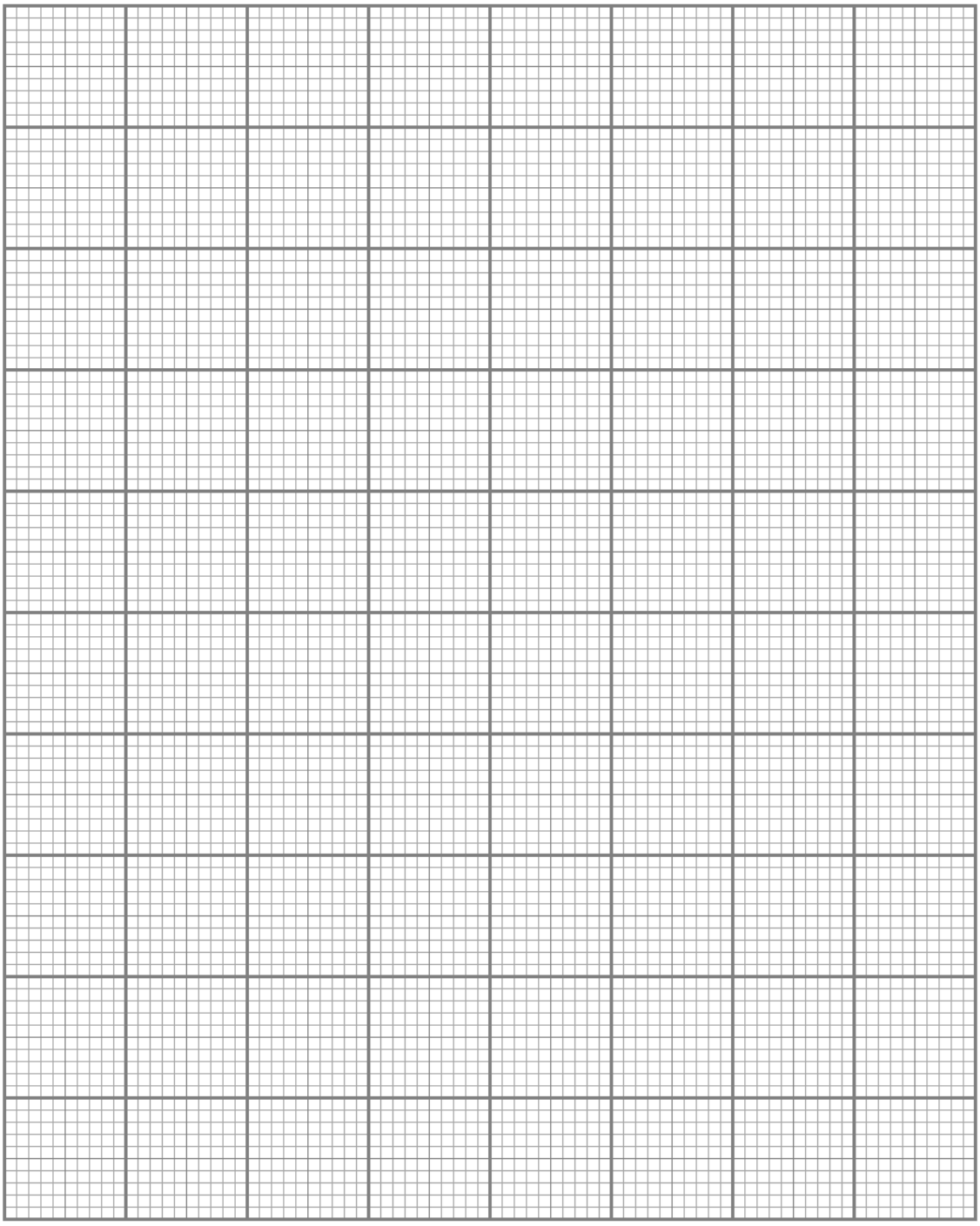

Fig 2.1

[3]

(ii) State and explain the relationship between **[FA 3]** and volume of **FA 5** used in each titration.

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……[1]

(iii) Hence, use your graph in **(b)(i)** and answer in **(b)(ii)** to determine the order of reaction with respect to [MnO_4^-].

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……[2]

(c) **(i)** Using your graph in **(b)(i)**, calculate the concentration of MnO_4^- ions in the reaction mixture at t_d = 10 min.

concentration of MnO_4^- ions = ……. [2]

(ii) Draw a tangent to your graph line in Fig 2.1 at time t_d = 10.0 min.

Determine the gradient of this line, showing clearly how you did this, and hence determine the rate of change of the amount of $S_2O_3^{2-}$ ions required in mol min^{-1}.

rate of change of the amount of $S_2O_3^{2-}$ ions = ... mol min^{-1} [3]

(iii) Using your answer in **(c)(ii)**, calculate the rate of change of the amount of MnO_4^- in mol min^{-1}.

rate of change of the amount of MnO_4^- ions = ... mol min^{-1} [1]

(iv) Hence, calculate the rate of disappearance of $[MnO_4^-]$ in the reaction mixture, at time t_d = 10 min.

rate of disappearance of $[MnO_4^-]$ = ... mol min^{-1} [3]

[Total: 18]

3 Qualitative analysis

(a) In question 2, you used a solution of Mn^{2+}, **FA 7**, containing an unknown anion. The anion in **FA 7** is not a nitrate, nitrite or carbonate ion.

You will devise and perform a series of simple tests to identify the anion present in **FA 7.**

Use the Qualitative Analysis Notes on pages 19 and 20 to deduce the possible anions in **FA 7** and describe **two different** tests, that will allow you to distinguish between the possible anions. You can only use the bench reagents provided.

Perform the tests described and record your observations in the space below. Hence, deduce the identity of the anion in **FA 7.**

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…….. [3]

(b) **FA 8** is a solid sample of a metal oxide.

You will need access to the **FA 2**, **FA 3** and **FA 4** from question 1 and 2.

Perform the tests described in Table 3.1 and record your observations in the table. Test and identify any gases evolved.

The observation in **(b)(iii)** has been completed for you. **There is no need to carry out this test.**

Table 3.1

tests		observations
(i)	Add 2 cm depth of **FA 2** and 2 cm depth of **FA 4** to a test-tube. Add half a spatula of **FA 8** to the mixture with swirling until no further change. Test for any gas evolved. Keep the reaction mixture for **(ii)**.	
(ii)	Filter the mixture from **(b)(i)** into a fresh test-tube. Divide the filtrate into two portions. To one portion, add aqueous sodium hydroxide until no further change. To the other portion, add aqueous ammonia until no further change.	
(iii)	Aqueous hydrogen peroxide is added to an equal volume of H_2SO_4(aq) in a test-tube, followed by addition of one spatula of **FA 8**. The mixture is filtered into another test-tube. Leave the filtrate to stand.	Vigorous effervescence observed. Gas rekindled glowing split. Gas is O_2. A black residue and a colourless filtrate obtained. The filtrate remains colourless.

[5]

(iv) Deduce the metal in **FA 8** using your results in **(b)(ii)**.

……[1]

(v) The filtrate obtained in **(b)(i)** turned pale yellow on standing.

Compare the role of **FA 8** in **(b)(i)** and **(b)(iii)**. Explain your answer with reference to your observations in both tests.

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……[2]

(c) **FA 9** can either be a carbonyl compound with molecular formula C_3H_6O or an alcohol with molecular formula C_3H_8O**.** Perform the tests described in Table 3.2 and record your observations in the table. If there is no observable change, write **no observable change**.

Table 3.2

	tests	observations
(i)	Add about 1 cm depth of **FA 9** in a test-tube. To this test-tube, add 6 drops of sodium hydroxide solution, followed by adding iodine solution, dropwise, until a permanent orange colour is present. Half fill a 250 cm^3 beaker with hot water. Immerse the test-tube into the water bath for two minutes.	
(ii)	Add about 1 cm depth of **FA 9** in a test–tube. To this test–tube, add 2 drops of **FA 3** followed by 1 cm depth of **FA 2**. Half fill a 250 cm^3 beaker with hot water. Immerse the test-tube into the water bath for two minutes.	

[2]

(iii) Using the information in Table 3.2, draw the structure of **FA 9**.

[1]

[Total: 14]

4 Planning

Calcium is an essential element that is vital to human life. Cow's milk is a good source of calcium, and the concentration of calcium can be determined using complexometric titration. A farmer wants to determine the concentration of calcium ions in the milk produced by his cows for marketing purposes. An experiment involving complexometric titration can be performed to find out the concentration.

Calcium ions can form a complex with disodium ethylenediaminetetraacetic acid, disodium EDTA, in a 1:1 ratio. Murexide is a violet indicator in basic conditions, which turns pink when bonded to Ca^{2+} ions. The murexide-Ca^{2+} complex, is however, less stable than the EDTA-Ca^{2+} complex. Hence, when a mixture containing murexide-Ca^{2+} is titrated against disodium EDTA solution, murexide will be displaced by disodium EDTA. The endpoint of the titration is hence determined when the colour of the reaction mixture changes from pink to violet.

EDTA is hazardous to the environment and can cause serious eye damage.

You may assume you are provided with:

- Cow's milk, **FB 1**
- 0.250 mol dm^{-3} disodium EDTA solution, **FB 2**
- 2.00 mol dm^{-3} sodium hydroxide solution
- Murexide indicator
- Waste bottle
- the equipment normally found in a school or college laboratory.

(a) Define the term complex.

…….. [1]

(b) Based on a previous investigation, the estimated calcium concentration in the milk is 100 mg per 100 cm^3 serving. Determine a suitable concentration of disodium EDTA such that 25 cm^3 of milk will react completely with 25 cm^3 of disodium EDTA solution. Justify your answer with calculations.

[A_r: Ca, 40.1]

[2]

(c) Plan an experiment to determine the concentration of calcium ions in a sample of cow's milk using complexometric titration. Your plan should include the preparation of a standard solution of the EDTA solution at the concentration in part **(b)**. In your titration, you will also need to ensure that the reaction mixture is basic by adding 10.0 cm^3 of the provided NaOH solution.

In your plan you should include details of

- the apparatus you would use,
- the procedure you would follow and
- the measurements you would make.

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……[4]

(d) Suggest a safety precaution that should be taken throughout the experiment.

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……...[1]

(e) EDTA can be represented as H_4EDTA because it is a weak acid. The $EDTA^{4-}$ anion can be formed from four successive deprotonation of H_4EDTA.

$$H_4EDTA(aq) \rightleftharpoons EDTA^{4-}(aq) + 4H^+(aq) \qquad \text{equation (1)}$$

At high pH, Mg^{2+} readily forms a complex with $EDTA^{4-}$.

$$[Mg(H_2O)_6]^{2+} + EDTA^{4-} \rightleftharpoons [Mg(EDTA)]^{2-} + 6H_2O \qquad \text{equation (2)}$$

In cow's milk, there is a small amount of Mg^{2+} ions present. Suggest how the presence of Mg^{2+} ions will affect the titration results in part **(c)**.

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…… [1]

[Total: 9]

END OF PAPER

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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 with dilute acids; gives white ppt. with Ba^{2+}(aq) (soluble in dilute strong acids)

(c) Test 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