1 Under certain conditions, iodine forms a series of polyiodide anions with the formula I_{2n+1}^-, where n is an integer.

This question is about the preparation of a polyiodide salt, $(CH_3)_4NI_{2n+1}$. After preparing the polyiodide salt, the value of n in $(CH_3)_4NI_{2n+1}$ is found by titration.

$(CH_3)_4NI_{2n+1}$ is formed when tetramethylammonium iodide, $(CH_3)_4NI$, reacts with methanolic iodine solution, I_2(meth).

$$(CH_3)_4NI + nI_2 \rightarrow (CH_3)_4NI_{2n+1}$$

(a) Methanolic iodine solution, I_2(meth), is prepared by dissolving solid I_2 in methanol.

(i) Calculate the mass, in g, of I_2(s) needed to prepare 100.0 cm^3 of 0.300 mol dm^{-3} I_2(meth).

mass = ... g [1]

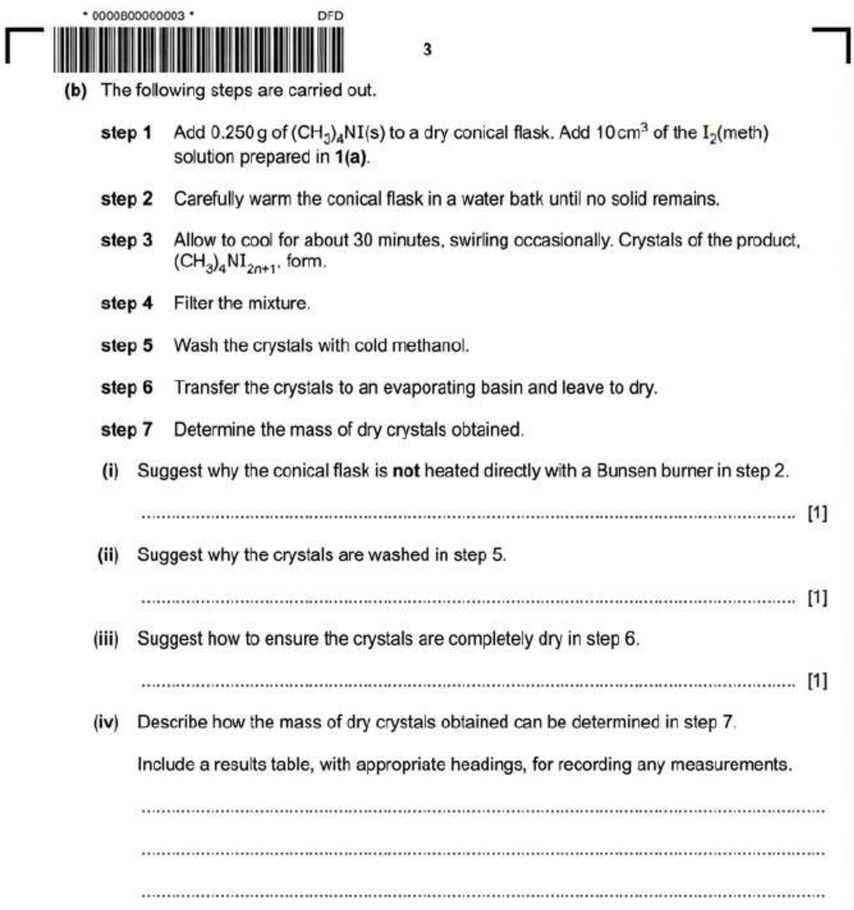
(ii) Describe the steps taken to prepare 100.0 cm^3 of 0.300 mol dm^{-3} I_2(meth), starting from the mass of I_2(s) calculated in **1(a)(i)** in a 50 cm^3 beaker.

Give the name and capacity of any apparatus used.

Write your answer using a series of numbered steps.

.... [3]

)	7.61 g
	M1 Dissolve the solid / KSCN(s) (in the beaker) (using a small volume of) ~~(distilled) water~~ methanol M2 Transfer/add to a (~~250~~ 100 cm^3 volumetric) flask **and** Rinse (with ~~distilled water~~ methanol) M3 Top the ~~250~~ 100 cm^3 volumetric flask up to mark (with ~~distilled water~~ methanol) **and** Invert the (stoppered) flask Allow '250 volumetric flask' (ie units need not be seen) M1 Transfer the I_2 to a 100 cm^3 volumetric flask M2 Wash the beaker with methanol and add washings to flask M3 Top up the (volumetric) flask (with methanol) to the mark and mix well



(b) The following steps are carried out.

step 1 Add 0.250 g of $(CH_3)_4NI(s)$ to a dry conical flask. Add 10 cm^3 of the I_2(meth) solution prepared in **1(a)**.

step 2 Carefully warm the conical flask in a water bath until no solid remains.

step 3 Allow to cool for about 30 minutes, swirling occasionally. Crystals of the product, $(CH_3)_4NI_{2n+1}$, form.

step 4 Filter the mixture.

step 5 Wash the crystals with cold methanol.

step 6 Transfer the crystals to an evaporating basin and leave to dry.

step 7 Determine the mass of dry crystals obtained.

(i) Suggest why the conical flask is **not** heated directly with a Bunsen burner in step 2.

.... [1]

(ii) Suggest why the crystals are washed in step 5.

.... [1]

(iii) Suggest how to ensure the crystals are completely dry in step 6.

.... [1]

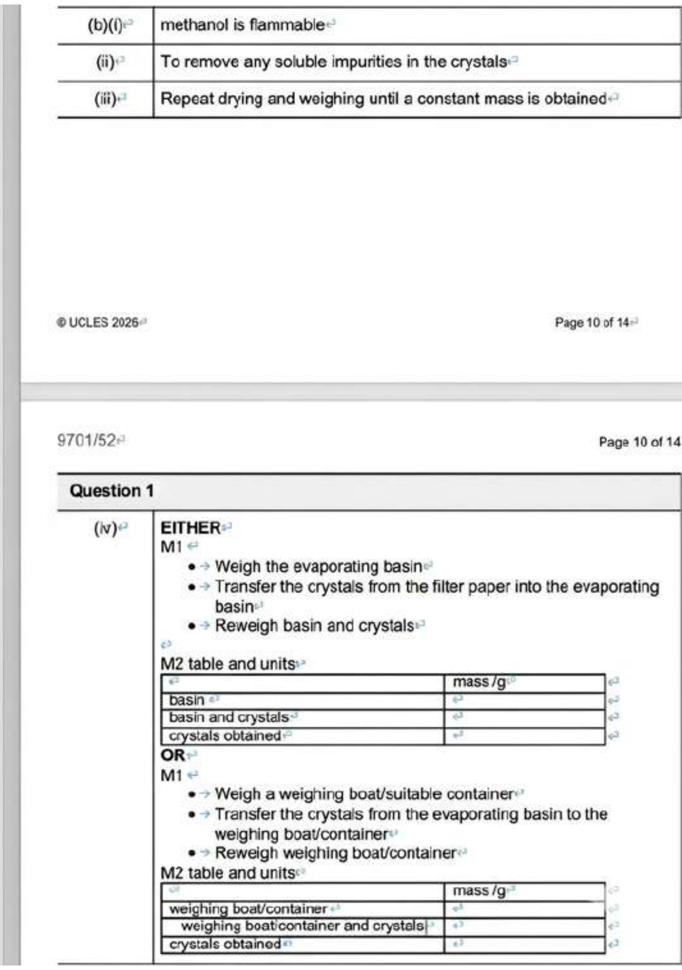
(iv) Describe how the mass of dry crystals obtained can be determined in step 7.

Include a results table, with appropriate headings, for recording any measurements.

....

....

....



(b)(i)	methanol is flammable
(ii)	To remove any soluble impurities in the crystals
(iii)	Repeat drying and weighing until a constant mass is obtained

Question 1

(iv)

EITHER

M1
- Weigh the evaporating basin
- Transfer the crystals from the filter paper into the evaporating basin
- Reweigh basin and crystals

M2 table and units

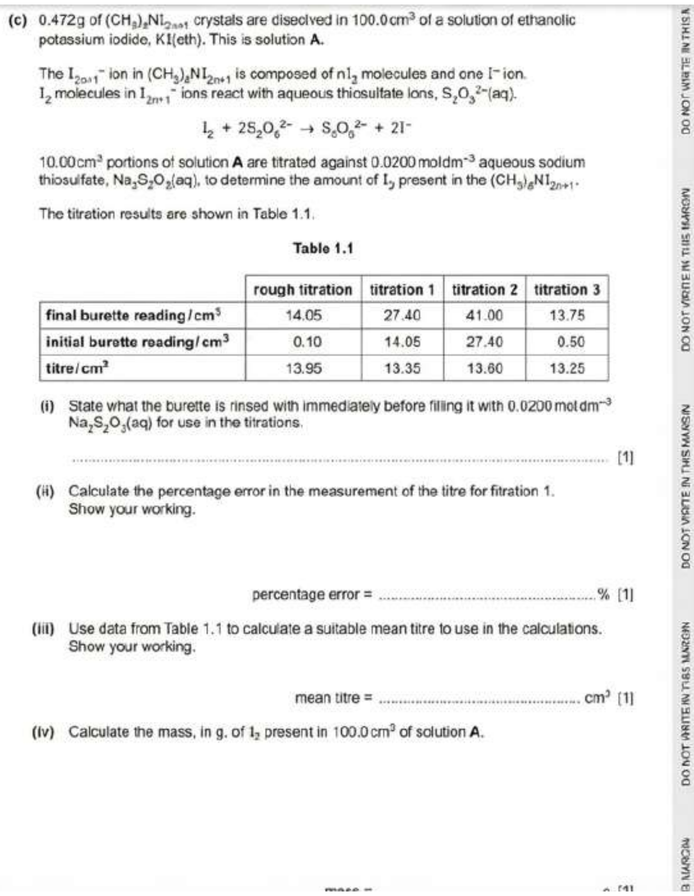
	mass / g
basin	
basin and crystals	
crystals obtained	

OR

M1
- Weigh a weighing boat/suitable container
- Transfer the crystals from the evaporating basin to the weighing boat/container
- Reweigh weighing boat/container

M2 table and units

	mass / g
weighing boat/container	
weighing boat/container and crystals	
crystals obtained	



(c) 0.472 g of $(CH_3)_8NI_{2n+1}$ crystals are disecived in 100.0 cm^3 of a solution of ethanolic potassium iodide, KI(eth). This is solution **A**.

The I_{2n+1}^- ion in $(CH_3)_8NI_{2n+1}$ is composed of nI_2 molecules and one I^- ion. I_2 molecules in I_{2n+1}^- ions react with aqueous thiosulfate ions, $S_2O_3^{2-}$(aq).

$$I_2 + 2S_2O_6^{2-} \rightarrow S_6O_6^{2-} + 2I^-$$

10.00 cm^3 portions of solution **A** are titrated against 0.0200 mol dm^{-3} aqueous sodium thiosulfate, $Na_2S_2O_3$(aq), to determine the amount of I_2 present in the $(CH_3)_8NI_{2n+1}$.

The titration results are shown in Table 1.1.

Table 1.1

	rough titration	titration 1	titration 2	titration 3
final burette reading / cm^3	14.05	27.40	41.00	13.75
initial burette reading / cm^3	0.10	14.05	27.40	0.50
titre / cm^3	13.95	13.35	13.60	13.25

(i) State what the burette is rinsed with immediately before filling it with 0.0200 mol dm^{-3} $Na_2S_2O_3$(aq) for use in the titrations.

.... [1]

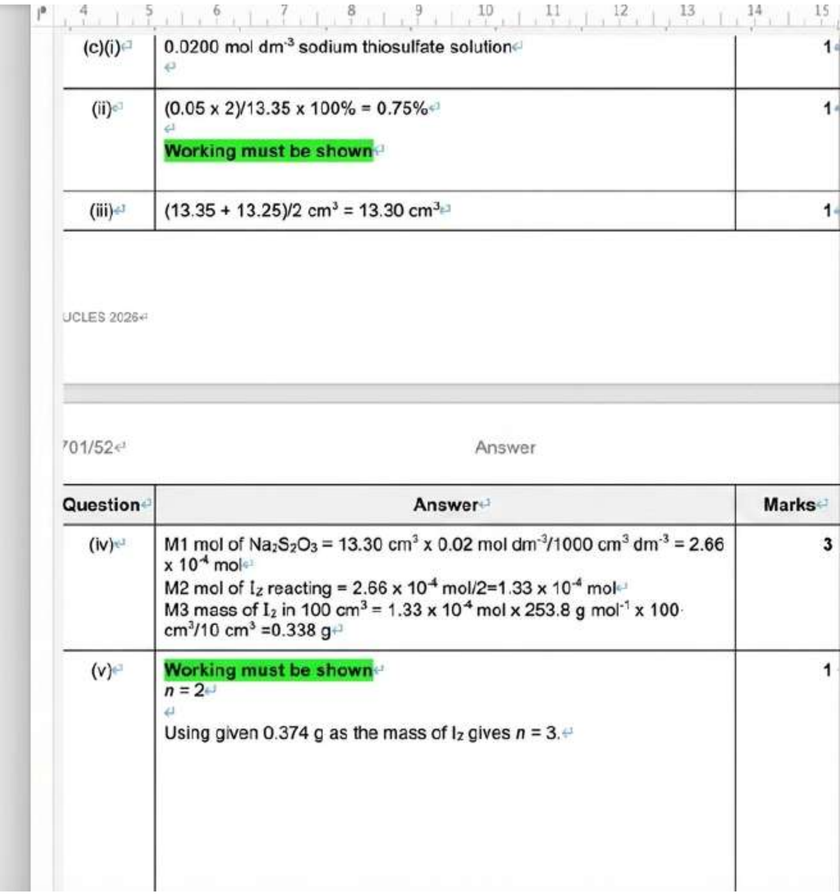
(ii) Calculate the percentage error in the measurement of the titre for fitration 1. Show your working.

percentage error = ... % [1]

(iii) Use data from Table 1.1 to calculate a suitable mean titre to use in the calculations. Show your working.

mean titre = ... cm^3 [1]

(iv) Calculate the mass, in g, of I_2 present in 100.0 cm^3 of solution **A**.



(c)(i)	0.0200 mol dm^{-3} sodium thiosulfate solution	1
(ii)	(0.05 x 2)/13.35 x 100% = 0.75% **Working must be shown**	1
(iii)	(13.35 + 13.25)/2 cm^3 = 13.30 cm^3	1

Question	Answer	Marks
(iv)	M1 mol of $Na_2S_2O_3$ = 13.30 cm^3 x 0.02 mol dm^{-3}/1000 cm^3 dm^{-3} = 2.66 x 10^{-4} mol M2 mol of I_2 reacting = 2.66 x 10^{-4} mol/2=1.33 x 10^{-4} mol M3 mass of I_2 in 100 cm^3 = 1.33 x 10^{-4} mol x 253.8 g mol^{-1} x 100 cm^3/10 cm^3 =0.338 g	3
(v)	**Working must be shown** n = 2 Using given 0.374 g as the mass of I_2 gives n = 3.	1

(v) 0.472 g of $(CH_3)_4NI_{2n+1}$ crystals were used to make solution **A** in **1(c)**. Use this information, the equation shown and your answer to **1(c)(iv)** to determine the value of *n* in $(CH_3)_4NI_{2n+1}$.

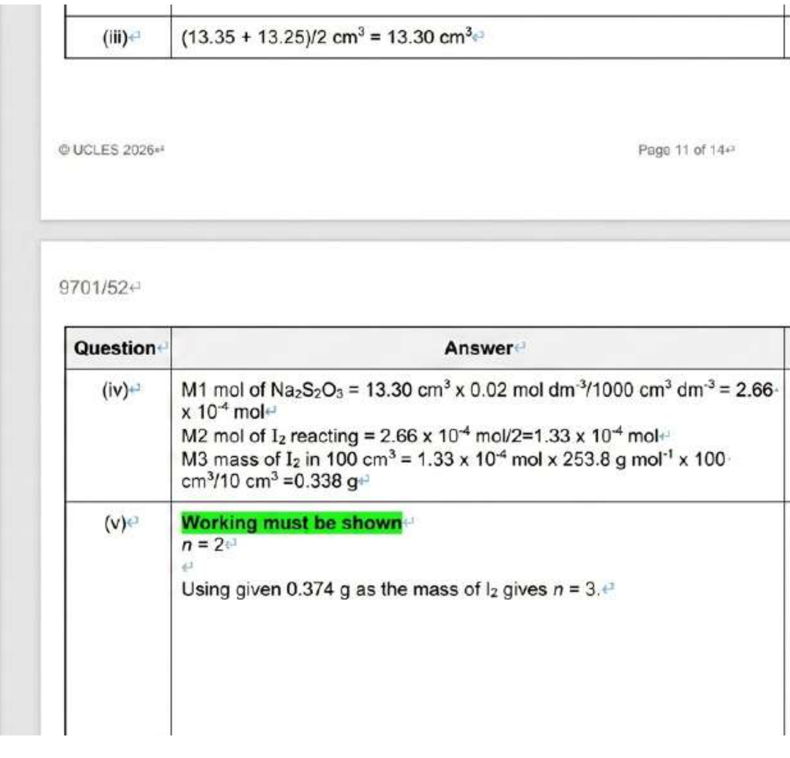
$$(CH_3)_4NI + nI_2 \rightarrow (CH_3)_4NI_{2n+1}$$

[If you were unable to answer **1(c)(iv)**, then assume the mass of I_2 is 0.374 g. This is **not** the correct value.]

Show your working.

n = ... [1]

[Total: 16]



(iii)	(13.35 + 13.25)/2 cm^3 = 13.30 cm^3

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Page 11 of 14

9701/52

Question	Answer
(iv)	M1 mol of $Na_2S_2O_3$ = 13.30 cm^3 x 0.02 mol dm^{-3}/1000 cm^3 dm^{-3} = 2.66 x 10^{-4} mol M2 mol of I_2 reacting = 2.66 x 10^{-4} mol/2=1.33 x 10^{-4} mol M3 mass of I_2 in 100 cm^3 = 1.33 x 10^{-4} mol x 253.8 g mol^{-1} x 100 cm^3/10 cm^3 =0.338 g
(v)	**Working must be shown** *n* = 2 Using given 0.374 g as the mass of I_2 gives *n* = 3.

2 This question is about the rate of the reaction between aqueous sodium ethanedioate, $Na_2C_2O_4(aq)$, and aqueous sodium peroxydisulfate, $Na_2S_2O_8(aq)$.

$$Na_2C_2O_4(aq) + Na_2S_2O_8(aq) \rightarrow 2Na_2SO_4(aq) + 2CO_2(g)$$

When $Na_2C_2O_4(aq)$ is in excess, the rate equation for this reaction is:

$$\text{rate} = k[Na_2S_2O_8]^m$$

where k is the rate constant and m is the order of the reaction with respect to $Na_2S_2O_8(aq)$.

In an experiment, excess $Na_2C_2O_4(aq)$ is added to a sample of $Na_2S_2O_8(aq)$ in a side-arm conical flask. The volume of carbon dioxide gas, $CO_2(g)$, produced is measured at regular time intervals.

Figure 2.1 shows the results from the experiment plotted on the grid. A curved line of best fit has been drawn.

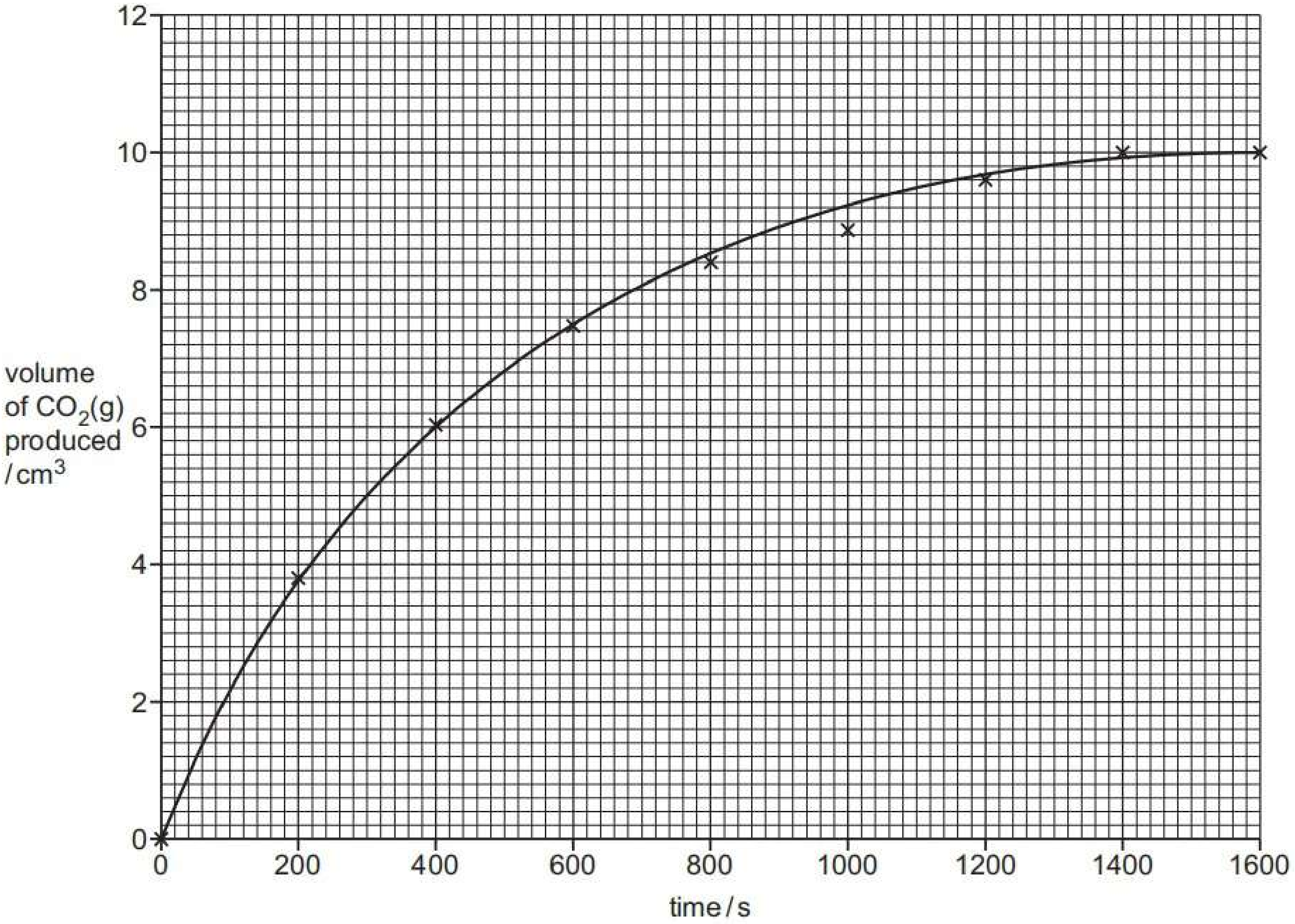
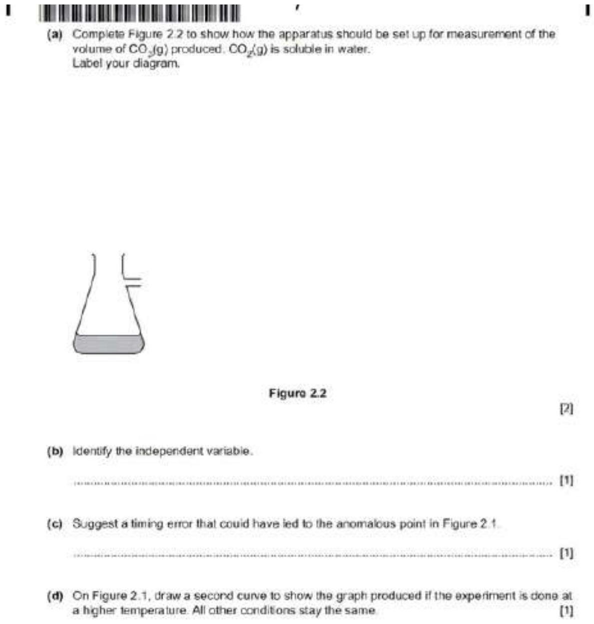


Figure 2.1



(a) Complete Figure 2.2 to show how the apparatus should be set up for measurement of the volume of $CO_2(g)$ produced. $CO_2(g)$ is soluble in water.
Label your diagram.

Figure 2.2

[2]

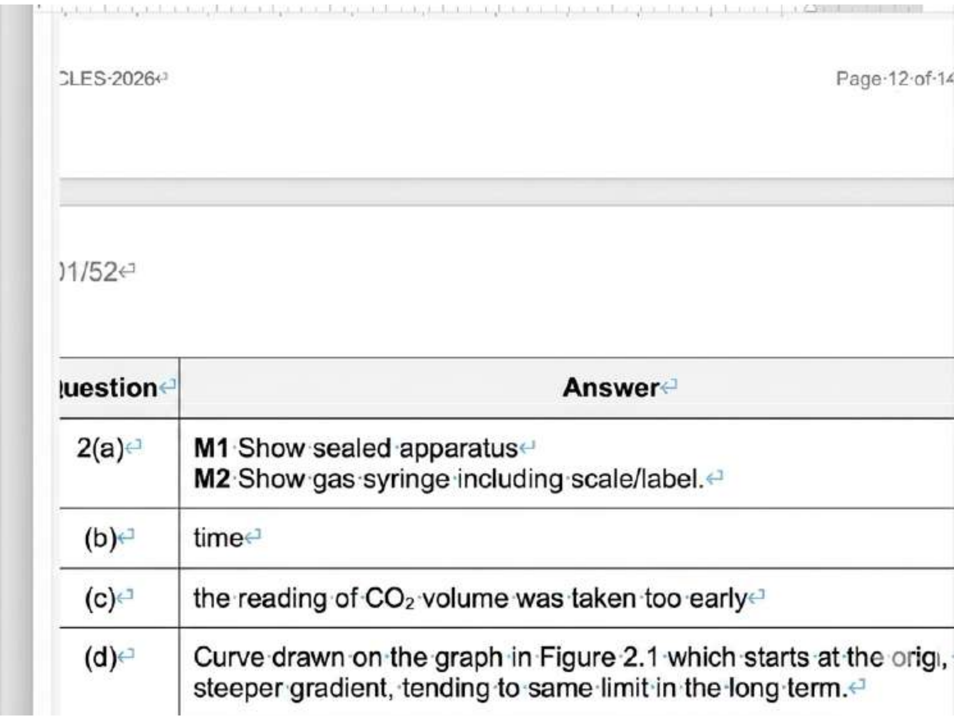
(b) Identify the independent variable.

.... [1]

(c) Suggest a timing error that could have led to the anomalous point in Figure 2.1.

.... [1]

(d) On Figure 2.1, draw a second curve to show the graph produced if the experiment is done at a higher temperature. All other conditions stay the same. [1]

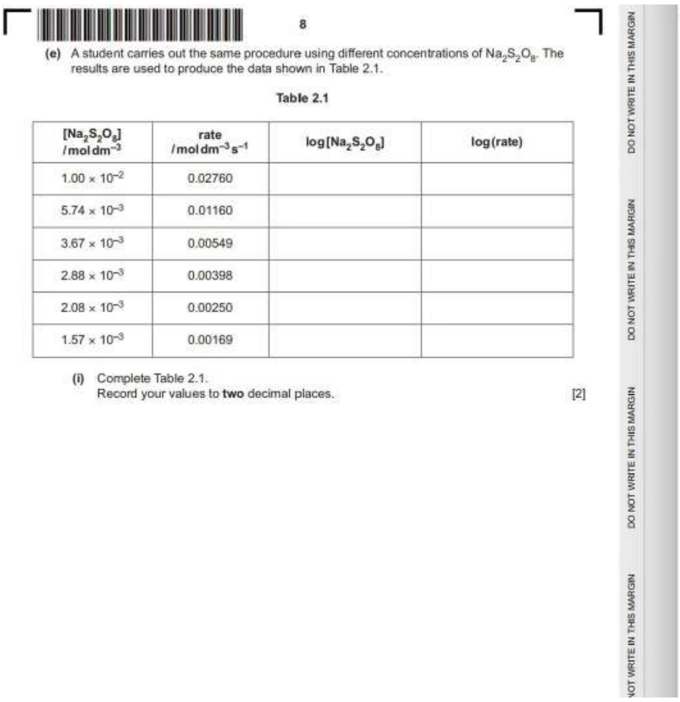


CLES 2026

Page 12 of 14

01/52

uestion	Answer
2(a)	**M1** Show sealed apparatus **M2** Show gas syringe including scale/label.
(b)	time
(c)	the reading of CO_2 volume was taken too early
(d)	Curve drawn on the graph in Figure 2.1 which starts at the origi, steeper gradient, tending to same limit in the long term.

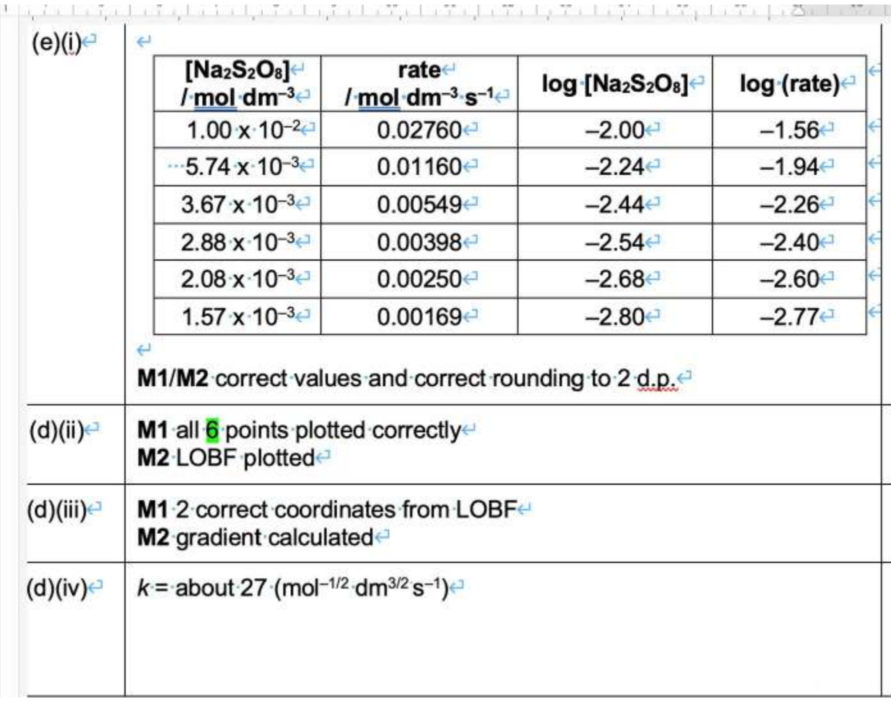


(e) A student carries out the same procedure using different concentrations of $Na_2S_2O_8$. The results are used to produce the data shown in Table 2.1.

Table 2.1

$[Na_2S_2O_8]$ / mol dm^{-3}	rate / mol dm^{-3} s^{-1}	log$[Na_2S_2O_8]$	log(rate)
1.00×10^{-2}	0.02760		
5.74×10^{-3}	0.01160		
3.67×10^{-3}	0.00549		
2.88×10^{-3}	0.00398		
2.08×10^{-3}	0.00250		
1.57×10^{-3}	0.00169		

(i) Complete Table 2.1.
Record your values to **two** decimal places. [2]



(e)(i)

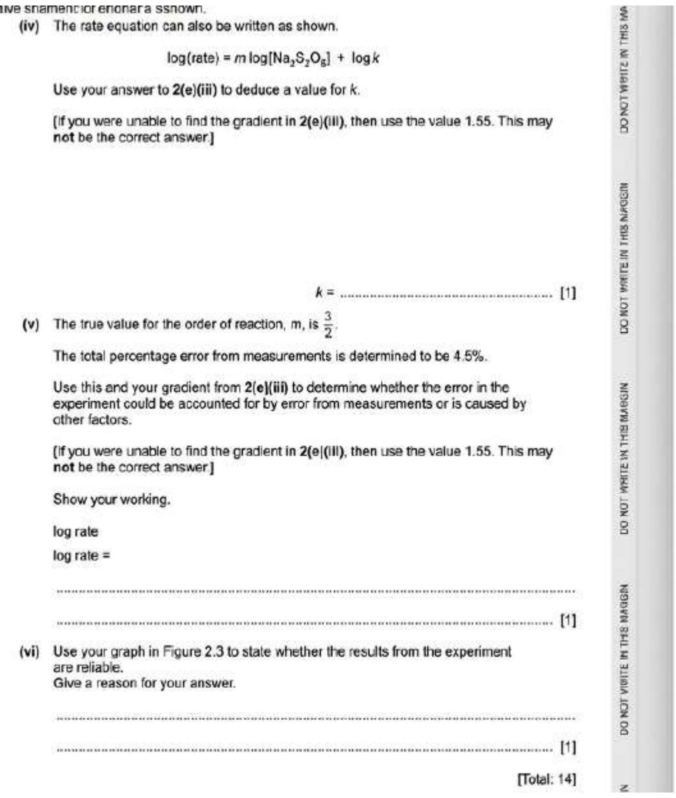
$[Na_2S_2O_8]$ / mol dm^{-3}	rate / mol dm^{-3} s^{-1}	log $[Na_2S_2O_8]$	log (rate)
1.00×10^{-2}	0.02760	–2.00	–1.56
5.74×10^{-3}	0.01160	–2.24	–1.94
3.67×10^{-3}	0.00549	–2.44	–2.26
2.88×10^{-3}	0.00398	–2.54	–2.40
2.08×10^{-3}	0.00250	–2.68	–2.60
1.57×10^{-3}	0.00169	–2.80	–2.77

M1/M2 correct values and correct rounding to 2 d.p.

(d)(ii)
M1 all 6 points plotted correctly
M2 LOBF plotted

(d)(iii)
M1 2 correct coordinates from LOBF
M2 gradient calculated

(d)(iv)
k = about 27 (mol$^{-1/2}$ dm$^{3/2}$ s^{-1})



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(iv) The rate equation can also be written as shown.

$$\log(\text{rate}) = m\log[Na_2S_2O_8] + \log k$$

Use your answer to **2(e)(iii)** to deduce a value for *k*.

[If you were unable to find the gradient in **2(e)(iii)**, then use the value 1.55. This may **not** be the correct answer.]

k = ... [1]

(v) The true value for the order of reaction, m, is $\frac{3}{2}$.

The total percentage error from measurements is determined to be 4.5%.

Use this and your gradient from **2(e)(iii)** to determine whether the error in the experiment could be accounted for by error from measurements or is caused by other factors.

[If you were unable to find the gradient in **2(e|(III)**, then use the value 1.55. This may **not** be the correct answer.]

Show your working.

log rate

log rate =

....

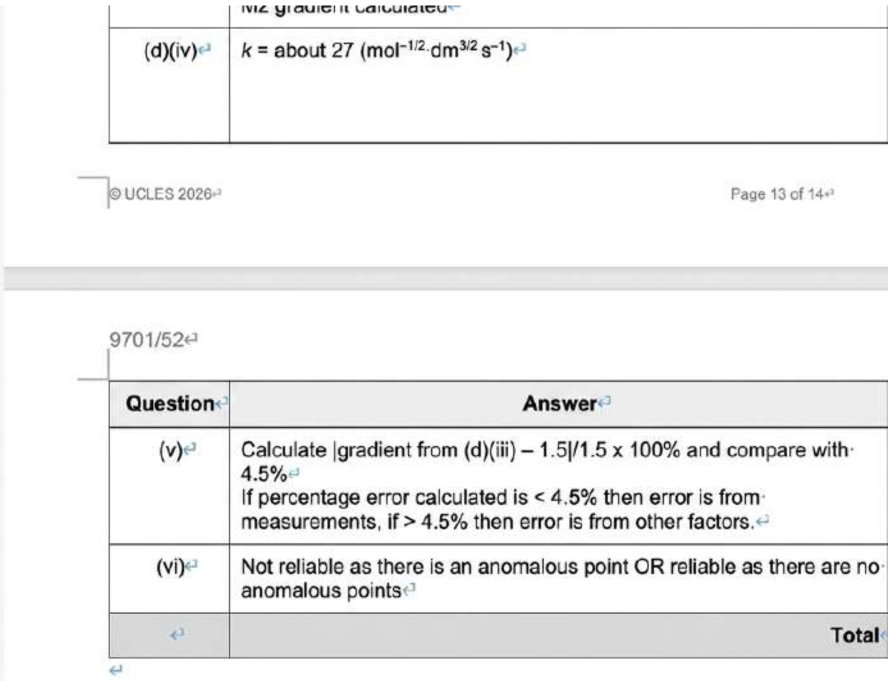
.... [1]

(vi) Use your graph in Figure 2.3 to state whether the results from the experiment are reliable.
Give a reason for your answer.

....

.... [1]

[Total: 14]



	M2 gradient calculated
(d)(iv)	k = about 27 ($mol^{-1/2}\cdot dm^{3/2}\,s^{-1}$)

Question	Answer
(v)	Calculate |gradient from (d)(iii) – 1.5|/1.5 x 100% and compare with 4.5% If percentage error calculated is < 4.5% then error is from measurements, if > 4.5% then error is from other factors.
(vi)	Not reliable as there is an anomalous point OR reliable as there are no anomalous points
	Total