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.

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.... [3]

(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.

....

....

....

(c) 0.472 g of $(CH_3)_4NI_{2n+1}$ crystals are dissolved in 100.0 cm^3 of a solution of ethanolic potassium iodide, KI(eth). This is solution **A**.

The I_{2n+1}^- ion in $(CH_3)_4NI_{2n+1}$ 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).

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

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 $(CH_3)_4NI_{2n+1}$.

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 titration 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**.

mass = ... g [3]

(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]

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.

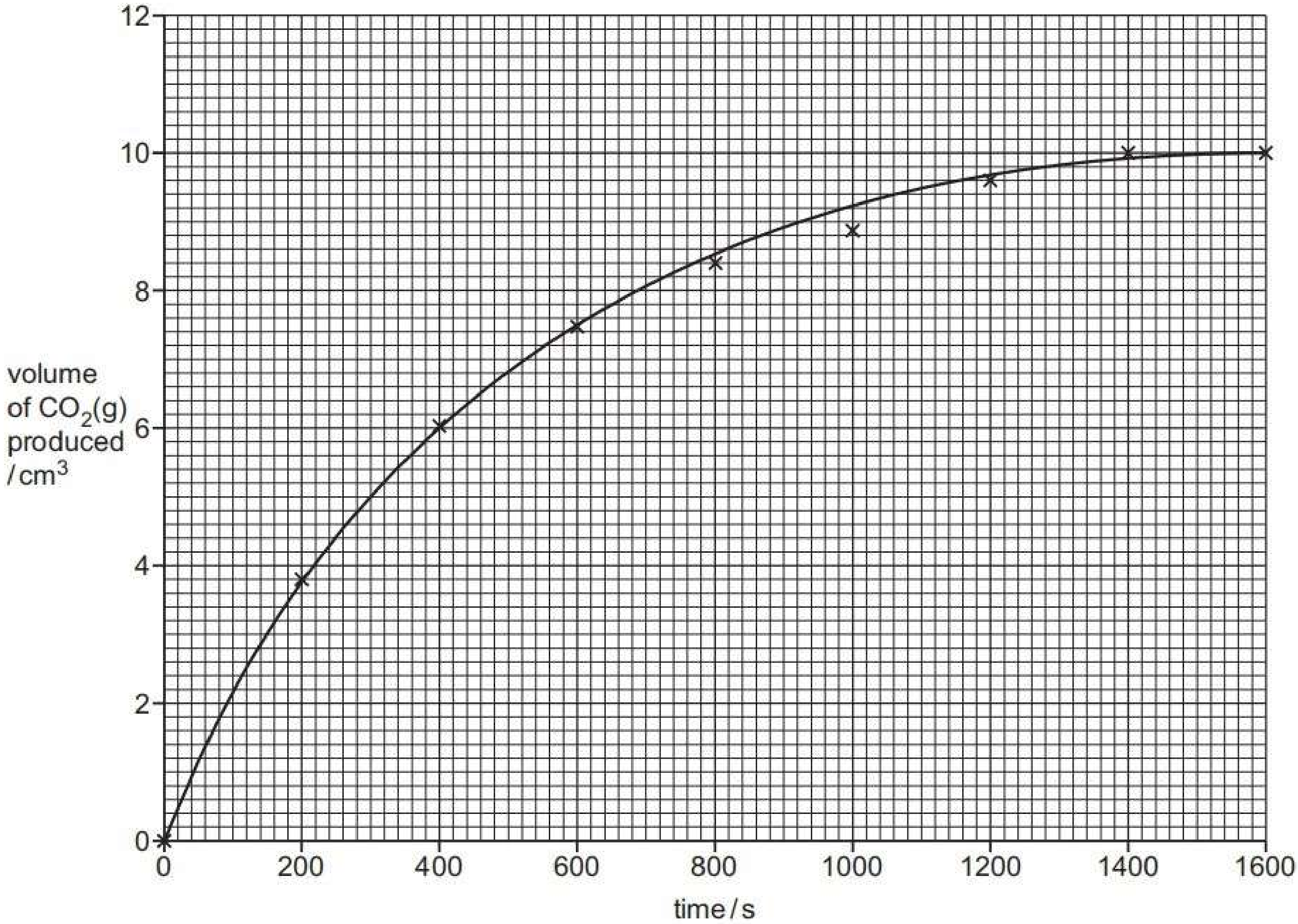

Figure 2.1

(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.

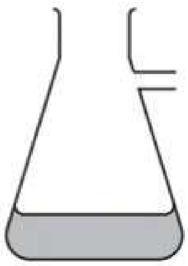

Figure 2.2

[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]

(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]

(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.

....

.... [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]