



TAMPINES MERIDIAN JUNIOR COLLEGE

JC2 PRELIMINARY EXAMINATION

CANDIDATE NAME

MARKS SCHEME

CIVICS GROUP

H2 CHEMISTRY

9476/04

Paper 4 Practical

2 Sep 2026

2 hour 30 minute

Candidates answer on the Question Paper.

Additional Materials: As listed in the *Confidential Instructions*.

READ THESE INSTRUCTIONS FIRST

Write your name and Civics Group in the spaces at the top of the page.

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 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 19 - 21.

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

Shift

Laboratory

For Examiner's Use

1 / 11

2 / 16

3 / 13

4 / 10

Total / **50**

This document consists of **20** printed pages and **2** blank pages.



Answer **all** the questions in the spaces provided.

1 Investigation of some inorganic and organic reactions

In this question, you will be investigating some inorganic and organic reactions by carrying out the stipulated tests. Carefully record your observations in **Tables 1.1** and **1.2**.

Unless otherwise stated, the volumes given below are approximate and should be estimated rather than measured.

(a) Organic analysis

In this question, you will deduce the structure of an organic compound, **FA 1**.

FA 1 has the molecular formula $C_4H_8O_2$ with **two** functional groups present.

Do not carry out the tests for which observations have been recorded.

Do not use the Bunsen burner for heating in this part of the question. Instead, use the hot water provided.

Table 1.1

	tests	observations
(i)	Place about 2 cm depth of aqueous sulfuric acid in a test-tube. To this test-tube, add about 1 cm depth of FA 1, followed by 1 drop of aqueous potassium manganate(VII). Warm the mixture in the hot water bath for two minutes. Hot acidified $KMnO_4 \rightarrow$ Test for functional group that can be oxidised (e.g. alkenes, primary / secondary alcohols, aldehyde etc.) Type of Reaction: Oxidation	<u>Purple $KMnO_4$ decolourises.</u> ✓
(ii)	Place about 1 cm depth of FA 1 in a test-tube. To this test-tube, add 2,4-dinitrophenylhydrazine dropwise. 2,4-DNPH $\rightarrow$ Test for aldehyde / ketone functional group Type of Reaction: Condensation	Orange ppt. formed
(iii)	Add 1 cm depth of aqueous silver nitrate to a test-tube. Then slowly add 1 cm depth of aqueous sodium hydroxide. Add aqueous ammonia slowly, with shaking, until the precipitate just dissolves. To this mixture, add about 1 cm depth of FA 1. Place the test-tube containing the mixture in the hot water bath for two minutes. Preparation of Tollens' Reagent Tollens' Reagent $\rightarrow$ Test for aldehyde functional group Type of Reaction: Oxidation	<u>Brown / grey ppt formed</u> ✓ <u>Brown / grey ppt dissolves to give a colourless solution.</u> ✓ <u>Silver mirror observed.</u> ✓

	tests	observations
(iv)	Place about 1 cm depth of FA 1 and add 8 drops of aqueous sodium hydroxide in a test-tube. Now, add iodine solution dropwise, until a permanent yellow / orange colour is obtained. Warm the mixture in the hot water bath for two minutes. Iodoform Test → Test for $\text{CH}_3\text{CH}(\text{OH})-$ and $\text{CH}_3\text{CO}-$ Type of Reaction: Oxidation	Pale yellow ppt (in yellow / orange solution) observed. ✓
(v)	Place 1 cm depth of FA 1 in a test-tube. To this test-tube, cautiously add a small piece of anhydrous phosphorus pentachloride. Anhydrous PCl_5 → Test for alcohol and carboxylic acid Type of Reaction: Nucleophilic Substitution OR Nucleophilic Acyl Substitution	Dense white fumes, which turns moist blue litmus paper red.

4 to 5 marking points – [M1] and [M2]

[2]

2 to 3 marking points – [M1] only

(b) **FA 1** has the molecular formula $\text{C}_4\text{H}_8\text{O}_2$ with **two** functional groups present.

- (i) Observations from (a)(ii) together with (a)(iii), can be used to identify one of the functional groups present in **FA 1**.

Identify the functional group and explain your answer, showing clearly your reasoning.

Functional group: aldehyde

Explanation: Since FA 1 undergoes condensation with 2,4-DNPH in (a)(ii), FA 1 contains either a ketone or aldehyde. However, since FA 1 undergoes oxidation with Tollens' reagent in (a)(iii), it contains an aldehyde. [M3] for correct type of reaction correlating to the correct deduction of functional group present [1]

- (ii) Observations from either (a)(iv) or (a)(v), can be used to identify the other functional group present in **FA 1**.

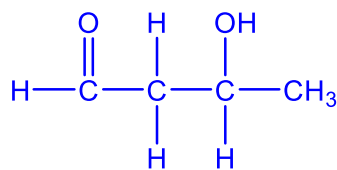
Identify the functional group and explain your answer, showing clearly your reasoning.

Functional group: alcohol must name, cannot only draw structure

Explanation: Since FA 1 undergoes nucleophilic substitution with PCl_5 in (a)(v) to give HCl , it contains an alcohol functional group. OR Since FA 1 undergoes oxidation with alkaline I_2 in (a)(iv), it contains an alcohol functional group. [M4] for correct type of reaction correlating to the correct deduction of functional group present [1]



- (iii) Suggest a possible structure of **FA 1** that is consistent with all the observations in **Table 1.1**.

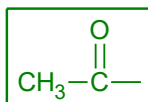


[M5] for correct structure

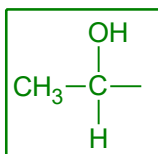
[1]

- Molecular Formula: $\text{C}_4\text{H}_8\text{O}_2$
- Contains aldehyde & alcohol functional group

- Contains



AND / OR



M1	M2	M3	M4	M5

(c) Inorganic analysis

FA 2 contains two cations and one anion listed in the Qualitative Analysis Notes on pages 19 – 20.

You should indicate clearly at what stage in a test a change occurs. Test and identify any gases evolved. If there is no observable change, write **no observable change**.

At each stage of any test, you are to record details of the following:

- colour changes seen
- the formation of any precipitate
- the solubility of such precipitates in an excess of the reagent added

No additional tests should be attempted.

Table 1.2

	tests	observations
(i)	Test the FA 2 solution using Universal Indicator paper.	• <u>Universal indicator paper turns orange-red / orange / pale orange.</u> ✓ • <u>pH 2 – 4</u> ✓
(ii)	Add 2 cm depth of FA 2 into a test-tube. Add aqueous sodium hydroxide, slowly with shaking, until no further change is seen. Filter the mixture into a clean test-tube. To 1 cm depth of the filtrate, add dilute nitric acid dropwise until in excess.	• <u>White</u> $\text{Al}(\text{OH})_3$ (s) <u>ppt</u> / <u>Off-white</u> $\text{Mn}(\text{OH})_2$ (s) <u>ppt</u> formed, <u>insoluble in excess</u> NaOH (aq). ✓ • Off-white <u>ppt turned brown on contact with air</u> $\text{Mn}(\text{OH})_3$ (s). ✓ • <u>Off-white</u> $\text{Mn}(\text{OH})_2$ (s) / <u>brown</u> $\text{Mn}(\text{OH})_3$ (s) <u>residue</u> ✓ • <u>Colourless filtrate</u> $\text{Al}(\text{OH})_4^-$ (aq) ✓ • <u>White ppt formed</u> $\text{Al}(\text{OH})_3$ (s) ✓ • <u>ppt soluble in excess</u> HNO_3 (aq) <u>to give a colourless solution.</u> Al^{3+} (aq) ✓
(iii)	Add 2 cm depth of FA 2 into a test-tube. Add aqueous ammonia, slowly with shaking, until no further change is seen. Filter the mixture into a clean test-tube. To 1 cm depth of the filtrate, add dilute nitric acid dropwise until in excess.	• <u>White</u> $\text{Al}(\text{OH})_3$ (s) <u>ppt</u> / <u>Off-white</u> $\text{Mn}(\text{OH})_2$ (s) <u>ppt</u> formed, <u>insoluble in excess</u> NH_3 (aq). ✓ • Off-white <u>ppt turned brown on contact with air</u> $\text{Mn}(\text{OH})_3$ (s). ✓ • <u>Off-white ppt / brown</u> $\text{Mn}(\text{OH})_2$ (s) and $\text{Al}(\text{OH})_3$ (s) ✓ <u>reject white</u> • <u>Colourless filtrate</u> ✓ • <u>No ppt formed / No observable change</u> ✓

	tests	observations
(iv)	Add 1 cm depth of FA 2 solution into a test-tube. Add a 1 cm depth of aqueous silver nitrate.	<u>White ppt</u> AgCl (s) <u>formed.</u> ✓

12 – 14 marking points – [M6], [M7], [M8], [M9]

[4]

8 – 11 marking points – [M6], [M7] & [M8], only

5 – 7 marking points – [M6] & [M7] only

2 – 4 marking points – [M6] only

(d) Use your observations in **Table 1.2** to identify the two cations and the anion in **FA 2**.

Table 1.3

	identity	explanation
cation in FA 2	✓ Mn^{2+}	In <u>test (c)(ii)</u>, FA 2 reacted with <u>NaOH(aq)</u> to give an <u>off-white ppt of Mn(OH)_2</u> which was <u>insoluble in excess NaOH(aq)</u>. On contact with air, <u>off-white ppt Mn(OH)_2 (s) was oxidised to brown ppt of Mn(OH)_3 (s)</u>
cation in FA 2	✓ Al^{3+}	In <u>test (c)(ii)</u>, FA 2 reacted with <u>excess NaOH(aq)</u> to form a <u>colourless filtrate of soluble Al(OH)_4^-</u> after filtration. On adding <u>dilute nitric acid</u>, a <u>white ppt of Al(OH)_3</u> is formed, which <u>dissolves in excess HNO_3 to form a colourless solution of $[\text{Al}(\text{H}_2\text{O})_6]^{3+}$</u> In <u>test (c)(iii)</u>, FA 2 reacted with <u>NH_3 to form Al(OH)_3 (s)</u>, a white ppt, <u>which is not soluble in excess $\text{NH}_3(\text{aq})$</u> and hence is not present in the filtrate. On adding <u>dilute nitric acid to the filtrate</u>, <u>no ppt was formed.</u>
anion in FA 2	✓ Cl^-	In <u>test (c)(iv)</u>, FA 2 reacted with <u>$\text{AgNO}_3(\text{aq})$</u> to <u>give a white ppt of AgCl.</u>

3 marking points – [M10] & [M11]

[3]

1 to 2 marking points – [M10] only

M6	M7	M8	M9	M10	M11

[Total: 11]



2 Investigating the kinetics of the reaction between iodate(V) ions and hydrogensulfite ions

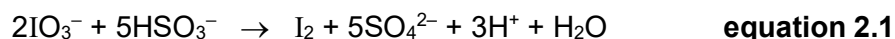
FA 3 is $0.100 \text{ mol dm}^{-3}$ potassium iodate(V), KIO_3

FA 4 is $0.0500 \text{ mol dm}^{-3}$ sodium hydrogen sulfite, NaHSO_3

You are also provided with:

- Solution **S**, 1% starch solution
- deionised water
- a waste container containing sodium carbonate solution

Iodate(V) ions react with hydrogensulfite ions according to the equation shown.



In this experiment, the rate of the reaction is studied by measuring the rate of formation of iodine using a 'clock' method. You will vary the concentration of iodate(V) ions and measure the time taken for a fixed amount of iodine to be produced. This will be indicated by the formation of a blue-black colour in the presence of starch.

The reaction takes place in a series of steps. As a result, the blue-black colour does not appear immediately.

You will perform a series of **five** experiments. For each experiment you will need to ensure that the same total volume of reaction mixture is used, by adding deionised water as required. For each experiment, you will measure the time, t , for the blue-black colour to appear. You should avoid inhaling any gases produced.

- (a) Prepare a table in the space provided on page 7 in which to record, to an appropriate degree of precision:
- the volumes of **FA 3** ($V_{\text{FA 3}}$), **FA 4** ($V_{\text{FA 4}}$) and deionised water (V_{water}) used
 - all values of t
 - calculated values of $\log(1/t)$
 - calculated values of $\log(V_{\text{FA 3}})$



Experiment 1

1. Use a teat pipette, add 0.5 cm^3 of Solution **S** to a 100 cm^3 conical flask.
2. Use a 10 cm^3 measuring cylinder to measure 3.0 cm^3 of **FA 3** into the conical flask.
3. Use a 25 cm^3 measuring cylinder to measure 15.0 cm^3 of deionised water into the same conical flask.
4. Use a second 10 cm^3 measuring cylinder to measure 2.0 cm^3 of **FA 4**.
5. Pour the **FA 4** into the same conical flask, starting the stopwatch during this addition.
6. Mix the contents thoroughly by swirling the flask several times. Place the flask on a white tile.
7. Stop the stopwatch at the first appearance of a blue-black colour.
8. Record the time taken, t , to the nearest second, in your table.
9. Discard the reaction mixture immediately into the waste container containing sodium carbonate solution.
10. Wash out the flask thoroughly with tap water and then with deionised water. Stand the flask upside down on a paper towel to drain.

Experiments 2 to 5

Repeat experiment 1 four times, adding 4.0 cm^3 , 6.0 cm^3 , 8.0 cm^3 and 10.0 cm^3 of **FA 3** respectively in step 2.

In each experiment, you should adjust the volume of deionised water in step 3 to ensure the **same total volume** of reaction mixture is used.

You should alternate the use of the two 100 cm^3 conical flasks.

Record all volumes, the time taken and the calculated values of $\log(1/t)$ and $\log(V_{\text{FA 3}})$ in your table.

Results

	<i>10 cm³ measuring cylinder (to nearest 0.1 cm³)</i>	<i>10 cm³ measuring cylinder (to nearest 0.1 cm³)</i>	<i>25 cm³ measuring cylinder (to nearest 0.5 cm³)</i>	<i>as stipulated in Qn (to nearest s)</i>		<i>calculated values (to 3 s.f.)</i>	
Expt	$V_{\text{FA 3}} / \text{cm}^3$	$V_{\text{FA 4}} / \text{cm}^3$	$V_{\text{water}} / \text{cm}^3$	t / s	$\frac{1}{t} / \text{s}^{-1}$	$\log(V_{\text{FA 3}})$	$\log\left(\frac{1}{t}\right)$
1	3.0	2.0	15.0	32	0.0313	0.477	-1.51
2	4.0	2.0	14.0	24	0.0417	0.602	-1.38
3	6.0	2.0	12.0	16	0.0625	0.778	-1.20
4	8.0	2.0	10.0	12	0.0833	0.903	-1.08
5	10.0	2.0	8.0	10	0.1000	1.00	-1.00

[4]

M12	M13	M14	M15



Marking Points	
<u>CORRECT HEADERS AND UNITS FOR ALL DATA</u> • Tabulates <u>data</u> in appropriate manner. Tabulation may be vertical or horizontal; lines are not essential but there should be no absence of headers. • Tables have correct <u>headers</u> and units, where <u>units</u> have not been included in the header, there should be appropriate unit for <u>each entry</u> in the tables. ○ Volume of FA 3 / cm³ Volume of FA 4 / cm³ Volume of water / cm³ ○ Time taken, t / s $\left(\frac{1}{t} / \text{s}^{-1}\right)$ – OPTIONAL ○ $\log\left(\frac{1}{t}\right)$ and $\log(V_{\text{FA 3}})$ (ignore units for lg values)	[M12]
<u>PRECISION OF VOLUMES USED / TIME TAKEN</u> • Volume of FA 3 and FA 4 recorded to nearest 0.1 cm³ (measured using 10.0 cm³ measuring cylinder) • Volume of deionised water recorded to nearest 0.5 cm³ (measured using 25.0 cm³ measuring cylinder) • Time taken recorded to nearest s. [Award based on students' presented data; Do <u>not</u> award this mark if any measurements (volumes and time taken) are missing]	[M13]
<u>Keeping V_{total} constant by varying $V_{\text{H}_2\text{O}}$ used and V_{FA4} constant at 2.0 cm³</u> • <u>Volume of deionised water + Volume of FA 3 = 18.0 cm³</u> Do not award this mark if V_{water} is not recorded as total volume cannot be assessed to be kept constant • <u>Volume of FA 4 used for experiment to be kept constant at 2.0 cm³</u>; If $V_{\text{solution S}}$ is also shown, then the <u>volumes used must be kept constant</u>; ensure the <u>total volume of the mixture is kept constant</u>.	[M14]
<u>Correct computation of log (values)</u> • Calculate $\log\left(\frac{1}{t}\right)$ and $\log(V_{\text{FA 3}})$ correctly and <u>presented to 3 s.f.</u>	[M15]

- (b) (i) Plot a graph of $\log(1/t)$ on the y-axis against $\log(V_{\text{FA } 3})$ on the x-axis on the grid in Fig. 2.1. Draw the best-fit straight line.

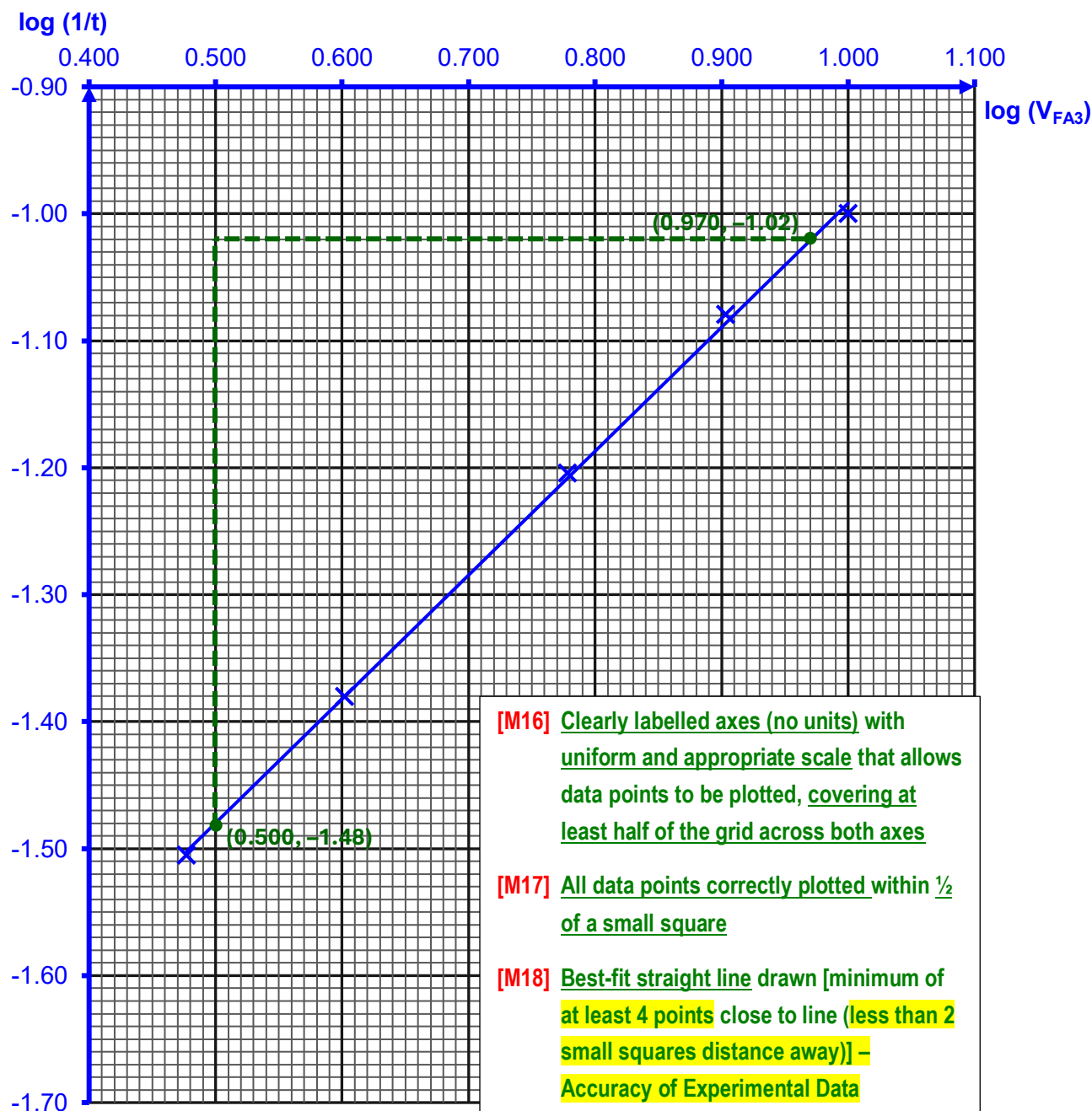


Fig. 2.1

[3]

- (ii) Explain why the volume of **FA 3** used in each experiment can be considered as a measure of $[\text{IO}_3^-]$.

Since total volume of the mixture is kept constant by adding varying volumes of deionised water, volume of FA3 used is proportional to $[\text{IO}_3^-]$. [M19] [1]

[M19] for constant total volume $\rightarrow V_{\text{FA3}} \propto n(\text{IO}_3^-) \propto [\text{IO}_3^-]$

M16	M17	M18	M19

- (iii) Calculate the gradient of the line to three significant figures, showing clearly how you did this. Hence, state the order of the reaction with respect to $[\text{IO}_3^-]$.

$$\text{Gradient} = \frac{-1.48 - (-1.02)}{0.500 - 0.970} = \underline{0.979 \text{ (3 s.f.)}} \quad \text{s.f. penalised under [M38]}$$

[M20] for correct determination of gradient with working

The gradient of the graph of $\lg\left(\frac{1}{t}\right)$ vs $\lg(V_{\text{FA}3})$ is the order of the reaction w.r.t. $[\text{IO}_3^-]$.

Order of reaction with respect to $[\text{IO}_3^-]$ is 1.

[M21] for correct determination of order of reaction based on gradient calculated

[For understanding – not required in answers]

$$\text{Rate} = k [\text{IO}_3^-]^x [\text{HSO}_3^-]^y$$

Since total volume of reaction mixture and volume of HSO_3^- (FA 4) used is kept constant, $[\text{HSO}_3^-]$ is kept constant across all experiments.

$$\text{Hence, rate} = k' [\text{IO}_3^-]^x; \text{ where } k' = k [\text{HSO}_3^-]^y$$

$$\text{Rate} \propto \frac{1}{t} \text{ and } [\text{IO}_3^-] \propto V_{\text{FA}3}$$

$$\frac{1}{t} = k'' (V_{\text{FA}3})^x$$

$$\log\left(\frac{1}{t}\right) = \log k'' + x \log(V_{\text{FA}3})$$

gradient =

order =

$$y = c + m X$$

[2]

With a plot of $\log(1/t)$ on the y-axis against $\log(V_{\text{FA}3})$ on the x-axis

→ Gradient of the best-fit line gives the order of reaction w.r.t. IO_3^- .

- (c) Step 10 of Experiment 1 in 2(a) instructs you to wash and drain the conical flask before it is used again.

State and explain the likely effect on time taken, t , if a student had washed the conical flask but had failed to drain it before starting another experiment.

effect on t : The time taken will be longer than expected.

explanation: The residual water present in the conical flask increases the total volume of the reaction mixture and decreases the concentration of reactants in the reaction mixture, decreasing the rate of reaction. [M22] [1]

[M22] for both

- time taken is **longer than expected**; and
- residual water **dilutes the reaction mixture / lowers reactant concentrations**, causing a **slower rate of reaction**.

M20	M21	M22



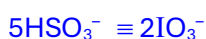
- (d) The average rate of decrease in iodate(V) ions, IO_3^- , up to the appearance of the blue-black colour can be estimated using:

$$\text{rate} = \left| \frac{\Delta[\text{IO}_3^-]}{\Delta t} \right|$$

For the purpose of this calculation, assume that the blue-black colour first appears when the hydrogensulfite ions initially present have just been consumed. **Equation 2.1** represents the overall reaction between iodate(V) ions and hydrogensulfite ions up to this point.

- (i) Calculate the amount of hydrogensulfite ions, HSO_3^- , initially present in each experiment. Hence, using **equation 2.1**, calculate the amount of iodate(V) ions, IO_3^- , consumed by the time the blue-black colour first appears.

Amount of HSO_3^- initially present = $0.0500 \times \frac{2.0}{1000}$ s.f. penalised under [M38]
Units penalised under [M39]
 = 1.00×10^{-4} mol (3 s.f.) with units [M23]



s.f. penalised under [M38]
Units penalised under [M39]

Amount of IO_3^- consumed = $\frac{2}{5} \times 1.00 \times 10^{-4} =$ 4.00×10^{-5} mol (3 s.f.) with units [M24]

amount of HSO_3^- ions initially present [1]

amount of IO_3^- ions consumed [1]

- (ii) Hence, calculate the average rate of decrease in $[\text{IO}_3^-]$ for **Experiment 1** in 2(a).

<i>Expt 1</i>	$V_{\text{FA 3}} / \text{cm}^3$	$V_{\text{FA 4}} / \text{cm}^3$	$V_{\text{water}} / \text{cm}^3$	$V_{\text{Solution S}} / \text{cm}^3$
	3.0	2.0	15.0	0.5

Total volume of reaction mixture = $20.5 \text{ cm}^3 =$ 0.0205 dm^3

Decrease in $[\text{IO}_3^-] = \frac{4.00 \times 10^{-5}}{(0.0205)} =$ $1.951 \times 10^{-3} \text{ mol dm}^{-3}$

For Experiment 1, time taken = 32 s (*Refer to Candidate's Data for Expt 1*) s.f. penalised under [M38]

Units penalised under [M39]

rate of decrease in $[\text{IO}_3^-] = \frac{1.951 \times 10^{-3}}{(32)} =$ $6.10 \times 10^{-5} \text{ mol dm}^{-3} \text{ s}^{-1}$ (3 s.f.) [M25]

rate of decrease in $[\text{IO}_3^-] =$ $\text{mol dm}^{-3} \text{ s}^{-1}$ [1]

M23	M24	M25



- (e) Another student conducted the experiment. However, only one of the student's plotted points lies significantly **above** the best-fit line drawn through the other points. Assume that the point was plotted correctly.

Suggest one experimental error that could have caused this anomaly. Explain your answer with reference to t , $1/t$ and $\log(1/t)$.

The student may have stopped the stopwatch too early, before the blue-black colour had properly appeared. [M26] Suitable error causing time taken to be smaller than expected

This gives a smaller recorded value of t . Hence, $1/t$ is larger and $\log(1/t)$ is less negative / larger, so the point lies above the best-fit line. [M27] correct link to transformed graph value

[2]

Other experimental errors that cause the recorded value of t to be smaller than expected

- The blue-black endpoint was judged too early.
- The stopwatch was started late after adding the final reagent, FA4.
- A larger-than-intended volume of FA 3 was used.
- Less deionised water than intended added, causing higher reactant concentrations

M26	M27

[Total: 16]



3 Standardisation of sodium hydroxide

Sodium hydroxide pellets cannot be obtained pure as they absorb moisture and carbon dioxide from the air. Hence, sodium hydroxide solutions are always standardised before use to determine their exact concentration.

Potassium hydrogen phthalate, $\text{KHC}_8\text{H}_4\text{O}_4$, is an organic acid of high purity that is used to standardise sodium hydroxide solutions. The M_r of $\text{KHC}_8\text{H}_4\text{O}_4$ is 204.2.

FA 5 is an aqueous solution of 8.15 g dm^{-3} $\text{KHC}_8\text{H}_4\text{O}_4$.

FA 6 is aqueous sodium hydroxide, NaOH .

You are to dilute a sample of **FA 6** and titrate **FA 5** with this diluted solution to find the concentration of NaOH in **FA 6**.

Solution **T** is thymolphthalein.

(a) Dilution of FA 6

- Using a pipette, transfer 10.0 cm^3 of **FA 6** into a 250 cm^3 volumetric flask.
- Make the solution up to the mark with deionised water and shake well to mix. Label this solution **FA 7**.

Titration of FA 5 against FA 7

- Fill a burette with **FA 7**.
- Use a pipette to transfer 25.0 cm^3 of **FA 5** into a 250 cm^3 conical flask.
- Add a few drops of solution **T** into the conical flask. Replace the cap on the container of **T** after use.
- Titrate the solution in the conical flask with **FA 7**. The end-point is reached when the solution changes from colourless to a permanent **pale** blue colour.
- Record your titration results, to an appropriate level of precision, in the space provided on page 13.
- Repeat steps 4 to 7 until consistent results are obtained.



(i) Titration results

Final burette reading / cm ³	21.10	41.55	29.10
Initial burette reading / cm ³	0.25	21.10	8.60
Volume of FA 7 used / cm ³	20.85	20.45	20.50
Values used (✓)		✓	✓

Marking Points	
<u>CORRECT HEADERS AND UNITS FOR ALL DATA</u> • Tabulates <u>burette readings</u> in appropriate manner. Tabulation may be vertical or horizontal; lines are not essential but there should be no absence of headers. • Appropriate <u>headers and units</u> for <u>all</u> data given in accurate titration tables (/ cm³); where <u>units</u> have not been included in the header, there should be the appropriate unit for <u>each entry</u> in the tables. • Do <u>not</u> award this mark if: 50.(00) is used as an initial burette reading OR More than one final burette reading is 50.(00) OR any burette reading is greater than 50.(00) OR swapped initial and final burette reading	[M28]
<u>PRECISION OF BURETTE READINGS / TITRE VALUE & CORRECT CALCULATION OF TITRE VALUES</u> • ALL burette readings and volume of FA7 used (including 0.00) are recorded to nearest 0.05 cm³. • Correctly calculated titre values from the difference between initial and final burette readings.	[M29]
<u>CONSISTENT TITRE VALUES WITHIN 0.10 cm³</u> • Has <u>at least two uncorrected titres within 0.10 cm³</u> (even if volumes were not indicated with '✓'s.)	[M30]
<u>ACCURACY OF TITRATION</u> SUPERVISOR'S RESULTS: 20.48 cm³ [2] difference from Supervisor's results: ≤ 0.30 cm³ [1] difference from Supervisor's results: > 0.30 cm³ but ≤ 0.60 cm³ [0] difference from Supervisor's results: > 0.60 cm³	[M31] [M32]

[5]

- (ii) From your titrations, obtain a suitable volume of **FA 7** to be used in your calculations. Show clearly how you obtain this volume.

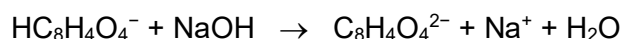
$$\text{Volume of FA 7} = \frac{20.45 + 20.50}{2} = \underline{20.48 \text{ cm}^3} \text{ (2 d.p.)}$$

[M33] with working and choice of uncorrected titres within 0.10 cm³

Units should be penalised under [M39]

volume of **FA 7** = [1]

- (b) The equation for the reaction in the titration is shown.



- (i) Calculate the concentration of NaOH in **FA 7**.

$$[\text{KHC}_8\text{H}_4\text{O}_4] \text{ in FA 5} = \frac{8.15}{204.2} = 0.03991 \text{ mol dm}^{-3}$$

Amount of $\text{KHC}_8\text{H}_4\text{O}_4$ in 25.0 cm^3 of **FA 5** (aliquot for titration)

$$= 0.03991 \times \frac{25.0}{1000} = 9.978 \times 10^{-4} \text{ mol [M34]}$$



Amount of NaOH present in 20.48 cm^3 of **FA 7** = $9.978 \times 10^{-4} \text{ mol}$

$$[\text{NaOH}] \text{ in FA 7} = 9.978 \times 10^{-4} \div \frac{20.48}{1000} = 0.04872 \text{ mol dm}^{-3} \quad \begin{array}{l} \text{s.f. penalised under [M38]} \\ \text{Units penalised under [M39]} \end{array}$$

$$= 0.0487 \text{ mol dm}^{-3} \text{ (3 s.f.) [M35]}$$

concentration of NaOH in **FA 7** = [2]

M28	M29	M30	M31	M32	M33	M34	M35

- (ii) Hence, calculate the concentration of NaOH in **FA 6**.

$$[\text{NaOH}] \text{ in FA 6} = 0.004872 \times \frac{250}{10.0} = 1.218 \text{ mol dm}^{-3} \quad \begin{array}{l} \text{s.f. penalised under [M38]} \\ \text{Units penalised under [M39]} \end{array}$$

$$= 1.22 \text{ mol dm}^{-3} \text{ (3 s.f.) [M36]}$$

concentration of NaOH in **FA 6** = [4]

Marking Points		
Working	Shows working in all calculations in 2(b)(iii), 2(d)(i), 2(d)(ii), 3(b)(i) and 3(b)(ii) . All calculations must be relevant although they may not be complete or correct. Any calculation not attempted loses this mark.	[M37]
Significant Figures	Shows appropriate significant figures (3 or 4 s.f.) in all final answers in • 2(b)(iii) (Accept 3 s.f. only), • 2(d)(i), 2(d)(ii) and • 3(b)(i); 3(b)(ii). Any calculation not attempted loses this mark.	[M38]
Units	Shows appropriate units in all final answers in 2(d)(i), 3(a)(ii), 3(b)(i) and 3(b)(ii) . Any calculation not attempted loses this mark.	[M39]

Calculations should be **relevant** even though it may not be correct; any **missing** calculations loses **the above marks**



- (c) A student used 25.0 cm³ of **FA 5** and obtained a **FA 7** titre value of 20.15 cm³ for the end point.

The errors (uncertainties) associated with each apparatus are given below.

pipette	$\pm 0.06 \text{ cm}^3$
burette	$\pm 0.05 \text{ cm}^3$

Use the above data to calculate the maximum total percentage error (uncertainty) in the volumes used to determine the concentration of **FA 7**.

$$\% \text{ error in pipette} = \frac{\pm 0.06}{25.0} \times 100 \% = \pm 0.240 \%$$

$$\% \text{ error in burette} = \frac{\pm 2 (0.05)}{20.15} \times 100 \% = \pm 0.496 \%$$

$$\text{Total \% error} = 0.240 + 0.496 = \pm 0.736 \% \text{ [M40] (Do not penalise for s.f.)}$$

maximum total percentage error [1]

M36	M37	M38	M39	M40

[Total: 13]



4 Planning

Other than the acid-base titration in **3(a)**, the concentration of sodium hydroxide in **FA 6** can also be determined thermometrically.

This involves performing a series of experiments using different volumes of dilute sulfuric acid and **FA 6** which together give a total volume of 60 cm^3 . The temperature change, ΔT , for each experiment will be determined and a graph of ΔT against the volume of H_2SO_4 used will be plotted. Where the two solutions have different initial temperatures, an appropriate initial temperature of the mixture should be determined, taking into account the volumes of both solutions, before calculating ΔT .

The volume of H_2SO_4 , V_{neut} , which gives the maximum temperature change, ΔT_{max} , is obtained from the graph. V_{neut} can then be used to calculate the concentration of sodium hydroxide in **FA 6**.

- (a) Plan an investigation to determine the concentration of sodium hydroxide in **FA 6** thermometrically.

You may assume that you are provided with:

- the same sodium hydroxide solution, **FA 6**
- 0.500 mol dm^{-3} sulfuric acid, H_2SO_4
- the apparatus normally found in a school or college laboratory

In your plan you should include brief details of:

- the apparatus you would use
- the procedure you would follow to ensure accurate experimental results
- the volumes of **FA 6** and H_2SO_4 used in each experiment
- the temperature measurements you would take, including how you would determine an appropriate initial temperature of the reaction mixture
- how the data measured would be used to determine values needed for the plotting of the graph

1. Place a polystyrene cup, labelled **FA 6**, inside a second polystyrene cup for better thermal insulation. Place both cups in a 250 cm^3 glass beaker to prevent it tipping over.
2. Use a 50 cm^3 measuring cylinder to transfer 10.0 cm^3 of **FA 6** into the Styrofoam cup labelled **FA 6**.
3. Stir the **FA 6** in the cup gently with another thermometer graduated to $0.2\text{ }^\circ\text{C}$. Read and record its temperature, $T_{\text{FA 6}}$, in the data table below.
4. Use another 50 cm^3 measuring cylinder to measure out 50.0 cm^3 of H_2SO_4 .
5. Stir the H_2SO_4 in the measuring cylinder gently with a thermometer graduated to $0.2\text{ }^\circ\text{C}$. Read and record its temperature, $T_{\text{H}_2\text{SO}_4}$, in the data table.
6. Add the H_2SO_4 from the measuring cylinder to the **FA 6** in the Styrofoam cup. Use the thermometer to stir the mixture gently. Read and record the maximum temperature of the mixture, T_{max} , in the data table.



9. Wash and carefully dry the **FA 6** Styrofoam cup.

10. Repeat steps 4 to 9 above using the following volumes:

$V_{\text{FA 6}} / \text{cm}^3$	$T_{\text{FA 6}} / ^\circ\text{C}$	$V_{\text{H}_2\text{SO}_4} / \text{cm}^3$	$T_{\text{H}_2\text{SO}_4} / ^\circ\text{C}$	$T_{\text{avg}} / ^\circ\text{C}$	$T_{\text{max}} / ^\circ\text{C}$	$\Delta T / ^\circ\text{C}$
10.0		50.0				
15.0		45.0				
20.0		40.0				
25.0		25.0				
30.0		30.0				
35.0		25.0				
40.0		20.0				
45.0		15.0				
50.0		10.0				

11. Calculate the average initial temperature, T_{avg} , for all entries using

$$T_{\text{avg}} = \frac{T_{\text{FA 6}} \times V_{\text{FA 6}} + T_{\text{H}_2\text{SO}_4} \times V_{\text{H}_2\text{SO}_4}}{60.00}$$

12. Calculate ΔT using $\Delta T = T_{\text{max}} - T_{\text{avg}}$ for all entries in the data table above.

[5]

[M41] Apparatus: polystyrene cup,

burette / measuring cylinders with capacity for volumes,
thermometer graduated to 0.2°C

[M42] Procedure - Measurement of Initial temperatures of solutions and determining $T_{\text{average, initial}}$

- Take initial temperature of **FA 6** and H_2SO_4 separately and (reject equilibrate / water bath)
- Determine $T_{\text{avg, initial}}$ (weighted average taking into consideration of the volumes of both solutions)
- Read maximum temperature reached, T_{max} or derive in expt context (e.g. graphically)

[M43] Procedure - Measurement of Maximum temperature of reaction mixture & accurate experimental results

- To minimise heat loss - thermal insulation (accept double cup, reject single cup)
- OR stir well after mixing

[M44] Data Treatment - Calculate $\Delta T = T_{\text{max}} - T_{\text{avg}}$ (or T_{initial} for answers without T_{avg}) **b.o.d. expt design**

[M45] At least five data sets with varying volumes of H_2SO_4 and NaOH but keeping the total volume of reaction mixture kept constant at 60 cm^3 reject topping up with DI water



- (b) Sketch the graph that you would expect to obtain on the axes in Fig. 4.1. Explain how the maximum temperature change, $\Delta T_{\max}$, and the corresponding volume of H_2SO_4 , V_{neut} , can be determined. You may find it useful to show how this might be done on your graph.

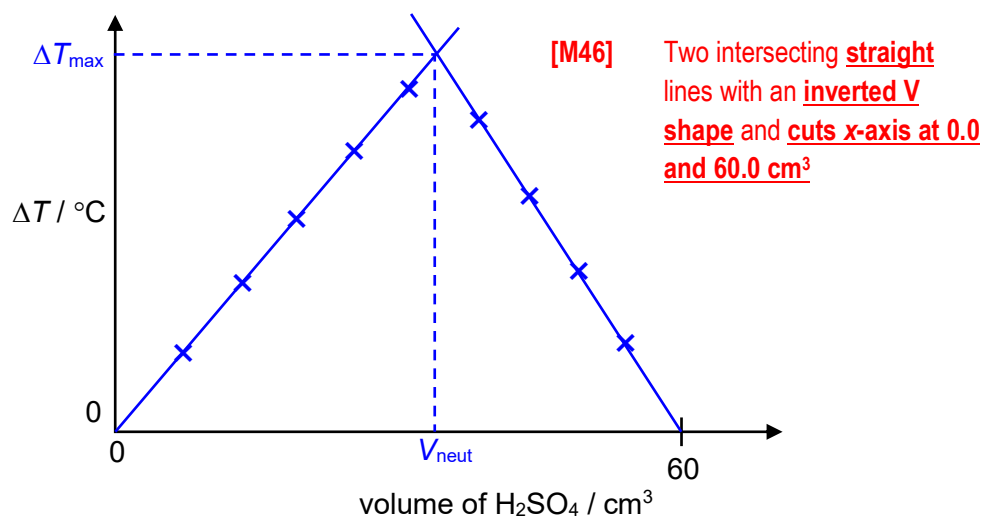


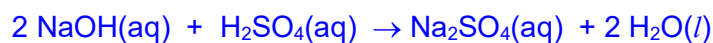
Fig. 4.1

Draw a line of best fit for the points BEFORE the maximum temperature change. Draw a line of best fit for the points AFTER the maximum temperature change. Extrapolate both lines until they meet.

The temperature change at the point of intersection gives the $\Delta T_{\max}$, while the volume of H_2SO_4 corresponds to V_{neut} . [M47] [2]

- (c) Derive an expression for the concentration of NaOH in FA 6, in terms of V_{neut} only.

$$n_{\text{H}_2\text{SO}_4} = \frac{V_{\text{neut}}}{1000} \times 0.500 = \frac{0.500 V_{\text{neut}}}{1000} \text{ mol}$$



$$n_{\text{NaOH}} = 2 n_{\text{H}_2\text{SO}_4} = \frac{0.500 V_{\text{neut}}}{1000} \times 2 = \frac{V_{\text{neut}} \times 10^{-3}}{1000} \text{ mol} \quad \text{[M48] ignore units, can give for method if not explicitly}$$

stated in terms of V_{neut} .

$$V_{\text{NaOH}} = (60.00 - V_{\text{neut}}) \text{ cm}^3$$

$$[\text{NaOH}]_{\text{FA 6}} = \frac{\frac{V_{\text{neut}} \times 10^{-3}}{60.00 - V_{\text{neut}}}}{\frac{1000}{1000}} = \frac{V_{\text{neut}}}{60.00 - V_{\text{neut}}} \text{ mol dm}^{-3} \quad \text{[M49] ignore units}$$

Full marks as long as final answer is correct.

- (d) In **Question 3**, the equivalence volume, V_{eq} , determined by volumetric titration was used to calculate the concentration of NaOH in **FA 6**. Suggest why the method in **Question 3** gives a more accurate determination of the concentration of NaOH.

The volumetric titration method is more accurate because the equivalence volume is determined using a burette, which allows more precise volume measurement.

However, the thermometric titration method used a less precise method of extrapolating the graph to determine V_{eq} . **OR** Volumetric titration allows the equivalence volume to be obtained directly while the thermometric titration requires an estimation of the equivalence volume through drawing of the 2 best-fit lines and extrapolation. **OR** [1]

There is greater inaccuracy in data obtained using the thermometric titration method due to heat loss to surroundings whereas volumetric titration eliminates the need to account for heat exchange with surroundings. **[M50]**

Need to mention heat loss rather than generic “affected by external temperature factor”.

Reject Expt 3 titration was repeated (reliability vs accuracy).

M46	M47	M48	M49	M50

[Total: 10]



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>ion</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 excess 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

(e) Tests for organic compounds

<i>organic compounds</i>	<i>reactions</i>
alkene	decolourises orange $\text{Br}_2(\text{aq})$
chloroalkane	heat with $\text{NaOH}(\text{aq})$, white ppt. formed on adding dilute HNO_3 , followed by $\text{Ag}^+(\text{aq})$
bromoalkane	heat with $\text{NaOH}(\text{aq})$, pale cream ppt. formed on adding dilute HNO_3 , followed by $\text{Ag}^+(\text{aq})$
iodoalkane	heat with $\text{NaOH}(\text{aq})$, yellow ppt. formed on adding dilute HNO_3 , followed by $\text{Ag}^+(\text{aq})$
alcohol	- forms white fumes with solid PCl_5 - decolourises purple acidified $\text{KMnO}_4(\text{aq})$ on heating (for primary and secondary alcohols) - gives pale yellow ppt. with alkaline $\text{I}_2(\text{aq})$ on warming (for alcohols with $\text{CH}_3\text{CH}(\text{OH})-$ group)
phenol	decolourises orange $\text{Br}_2(\text{aq})$ and forms a white ppt
carbonyl compounds (aldehydes and ketones)	- gives orange ppt. with 2,4-dinitrophenylhydrazine - gives pale yellow ppt. with alkaline $\text{I}_2(\text{aq})$ on warming (for carbonyl compounds with $\text{CH}_3\text{CO}-$ group) - gives red-brown ppt. with Fehling's solution on warming (for aliphatic aldehydes) - gives silver mirror with Tollens' reagent on warming (for aldehydes)
carboxylic acid	CO_2 liberated with $\text{Na}_2\text{CO}_3(\text{aq})$ - gives white fumes with solid PCl_5
phenylamine	decolourises orange $\text{Br}_2(\text{aq})$ and forms a white ppt
primary amide	NH_3 liberated on heating with $\text{NaOH}(\text{aq})$

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