



RIVER VALLEY HIGH SCHOOL
Preliminary Examination

CANDIDATE
NAME

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CLASS

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CENTRE
NUMBER

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INDEX
NUMBER

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CHEMISTRY

9476/04

Paper 4 Practical

2 hours 30 minutes

You must answer on the question paper.

You will need: The materials and apparatus listed in the confidential instructions

INSTRUCTIONS

- Answer **all** questions.
- Use a black or dark blue pen. You may use an HB pencil for any diagrams or graphs.
- Write your name, centre number and index number in the boxes at the top of the page.
- Write your answer to each question in the space provided.
- Do not use an erasable pen. Do not use correction fluid or tape.
- You may use an approved calculator.
- Qualitative analysis notes are printed in the question paper.
- Write the details of the shift and laboratory in the boxes provided.

INFORMATION

- The total mark for this paper is 50.
- The number of marks for each question or part question is shown in brackets [].

Shift
Laboratory

For Examiner's Use	
1	
2	
3	
4	
Total	

This document has 20 pages. Any blank pages are indicated.

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

1 To determine the percentage by mass of copper in an alloy

Copper forms compounds containing Cu^{2+} and Cu^+ ions. The compounds containing Cu^{2+} ions tend to be relatively stable.

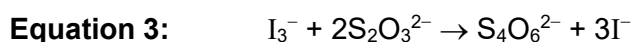
The addition of an excess of potassium iodide, KI, to a solution of Cu^{2+} ions produces iodine, I_2 , and a stable precipitate of CuI. For titration to be accurate, it is necessary that all Cu^{2+} ions are reduced to Cu^+ ions. The I_2 turns the solution brown.



I_2 has a relatively low solubility in water. However, the presence of an excess of I^- ions in the reaction mixture allows the soluble tri-iodide ion, I_3^- , to form as shown by **equation 2**. This ensures that the I_2 formed as shown in **equation 1** is fully dissolved.



The I_3^- ions formed may be titrated against a standard solution of $\text{Na}_2\text{S}_2\text{O}_3$ as shown in equation 3.



In **1(a)**, you will perform titrations to determine the percentage by mass of copper present in an alloy.

FA 1 is an aqueous solution containing 11.54 g dm^{-3} of the alloy.

You are also provided with

FA 2, $0.100 \text{ mol dm}^{-3}$ sodium thiosulfate, $\text{Na}_2\text{S}_2\text{O}_3$,

FA 3, $0.220 \text{ mol dm}^{-3}$ potassium iodide,

starch indicator.

In this experiment, you will determine the percentage by mass of copper in the alloy used to prepare **FA 1**. You will titrate **FA 1** against **FA 2**.

(a) (i) Titration of **FA 1** against **FA 2**

1. Fill a burette with **FA 2**.
2. Use a 25.0 cm^3 pipette to transfer 25.0 cm^3 of **FA 1** into a 250 cm^3 conical flask.
3. Use a measuring cylinder to add about 40 cm^3 of **FA 3** to this flask. A white precipitate forms in a brown solution.
4. Run **FA 2** from the burette into this flask. Near end point, when the brown solution becomes pale, add about 1 cm^3 of starch indicator.
5. Continue adding **FA 2** slowly. The end point is reached when the **solution** first becomes colourless. The white precipitate remains.
6. Record your titration results, to an appropriate level of precision, in the space provided.
7. Repeat steps 2 to 6 until consistent results are obtained.

Results

I	
II	
III	
IV	
V	

- (ii) From your titrations, obtain a suitable volume of **FA 2**, $V_{\text{FA 2}}$, to be used in your calculations. Show clearly how you obtained this volume.

$V_{\text{FA 2}} = \dots\dots\dots$ [1]

- (b) Calculate the mass of copper in the 11.54 g of the alloy used to prepare 1.00 dm³ of **FA 1**, using your answer in **1 (a)(ii)**.

Hence, determine the percentage by mass of copper in the alloy.

[A_r : Cu. 63.5]

Mass of copper in 11.54 g of the alloy = $\dots\dots\dots$ [2]

Percentage by mass of copper in the alloy = $\dots\dots\dots$ [1]

- (c) A large excess of potassium iodide is used in each titration.

Calculate the ratio of the amount of iodide ions added in each titration to the minimum amount of iodide ions required to reduce the copper(II) ions used.

Ratio = : [2]

- (d) Suggest how the titration would be affected if the potassium iodide solution was not added in excess.

.....
.....
..... [1]

[Total: 12]

2 To determine order of reaction with respect to thiosulfate ions, $\text{S}_2\text{O}_3^{2-}$

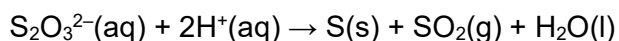
You are provided with the following reagents.

FA 4, containing 37.0 g dm^{-3} of hydrated sodium thiosulfate, $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$,

FA 5, 2.0 mol dm^{-3} hydrochloric acid, HCl ,

deionised water.

Aqueous thiosulfate ions, $\text{S}_2\text{O}_3^{2-}$, decompose in the presence of hydrogen ions, H^+ .



The rate of reaction can be followed by observing the formation of a fixed amount of sulfur as a fine suspension of solid particles in the solution.

The rate of decomposition at a fixed temperature is affected by the

- concentration of thiosulfate ions, $[\text{S}_2\text{O}_3^{2-}]$
- concentration of hydrogen ions, $[\text{H}^+]$

In a series of experiments where the concentration of a reagent is changed

- $1/\text{time}$ can be used as a measure of rate
- the volume of the reagent used can be taken as a measure of its concentration, providing the total volume of the mixture is kept constant in each experiment

The rate equation, where m is the rate order with respect to $\text{S}_2\text{O}_3^{2-}$, can be simplified to

$$\text{rate} = k[\text{S}_2\text{O}_3^{2-}]^m$$

Taking logarithms of the factors in this equation gives

$$\lg(\text{rate}) = m \lg[\text{S}_2\text{O}_3^{2-}] + \lg k$$

By substituting for rate and concentration, as described above, the following is derived

$$\lg(1/t) = m \lg(\text{volume of FA 4}) + \text{constant}$$

Hence, by plotting a graph of $\lg(1/t)$ against $\lg(\text{volume of FA 4})$, a best-fit straight line graph can be drawn, the gradient of which is the required rate order.

The order of reaction with respect to thiosulfate ions can be obtained by plotting a graph of $\lg(1/t)$ against $\lg(\text{volume of FA 4})$.

(a) Method

Experiment 1

- Fill the burette, to be labelled as **FA 4**, with **FA 4**.
- Run 50.00 cm³ of **FA 4** from the burette into a dry 250 cm³ beaker.
- Measure 5 cm³ of **FA 5** in a 10 cm³ measuring cylinder.
- Tip the acid **FA 5** from the measuring cylinder into the beaker and **immediately** start a stopwatch
- Swirl the beaker to mix the reagents and place the beaker over the printing on the insert provided.
- Observe the printing by looking down through the solution in the beaker.
- Stop the stopwatch when the printing is just no longer visible.
- Record the time, t , taken to the **nearest second**.

Note: Small amounts of SO₂ will be produced during the reaction. Minimise inhalation of SO₂. Discard the reaction mixture **immediately** down the sink after recording the time.

Experiment 2

- Empty and rinse the beaker used in experiment 1.
- Carefully dry the beaker with a paper towel.
- Refill the burette labelled **FA 4** with **FA 4**.
- Fill the second burette, to be labelled as water, with deionised water.
- Run 15.00 cm³ of **FA 4** and 35.00 cm³ of deionised water from the burettes into the 250 cm³ beaker.
- Measure 5 cm³ of **FA 5** in a 10 cm³ measuring cylinder.
- Tip the acid **FA 5** into the beaker, start the stopwatch, mix the reagents and place over the printing as in experiment 1.
- Stop the stopwatch when the printing is just no longer visible.
- Record the time taken, t , to the **nearest second**.

- (b) In experiment 1 you will have obtained the time taken for a 'fast' reaction and in experiment 2 the time taken for a 'slow' reaction. You are to repeat the experiment with two further mixtures in which only the concentration of thiosulfate ions is varied.

For all four experiments, prepare a table in the space provided on **page 8** in which to record, to an appropriate level of precision:

- all volumes that vary
- t
- $1/t$
- $\lg(1/t)$
- $\lg(\text{volume of FA 4})$

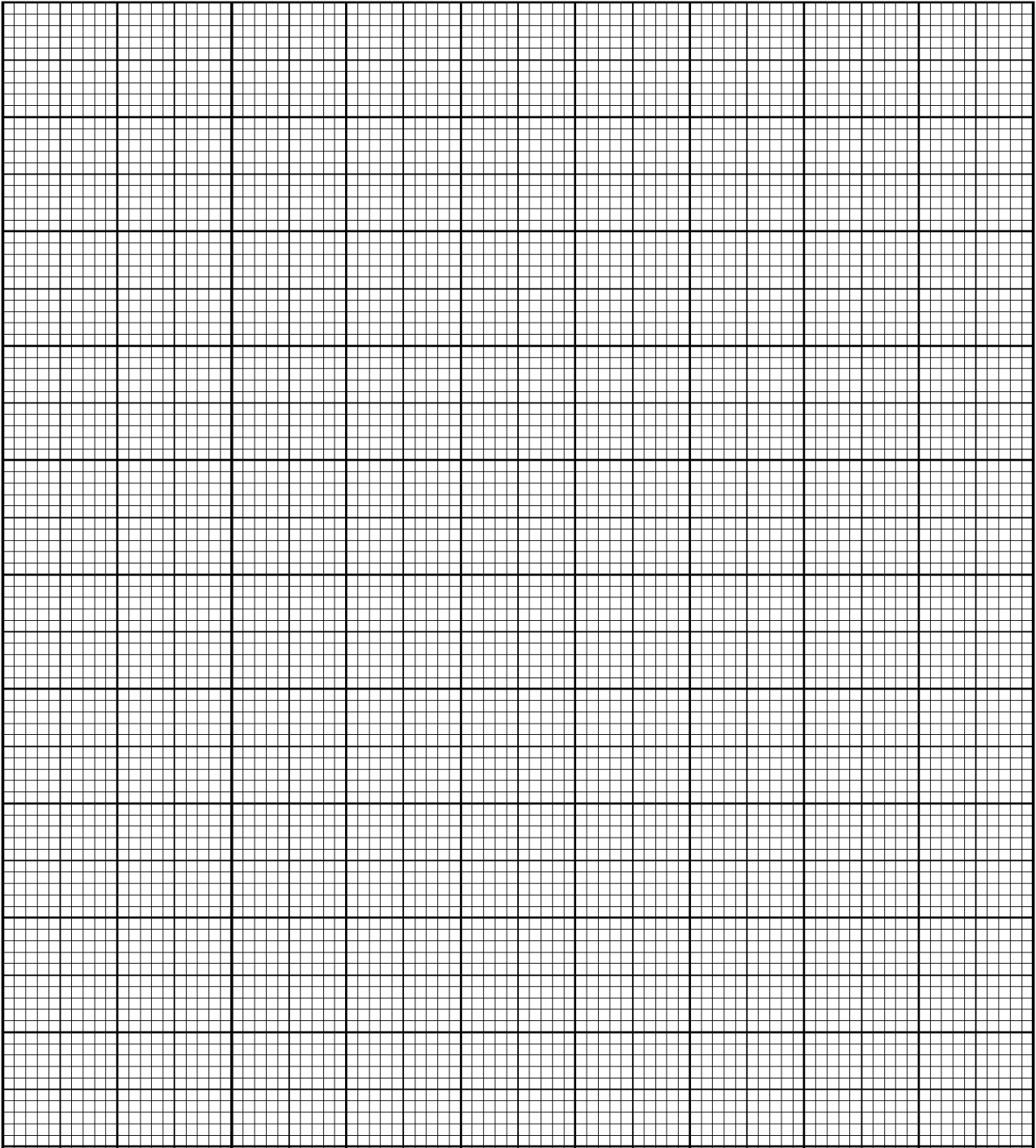
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Results

I	
II	
III	
IV	

- (c) Use the grid below to plot a graph of $\lg(1/t)$ against $\lg(\text{volume of FA 4})$.

Draw the best-fit straight line taking into account all of your plotted points.



I	
II	
III	

- (i) Calculate the gradient of the line drawn and determine the order of reaction with respect to thiosulfate ions, $\text{S}_2\text{O}_3^{2-}$, ***m***.

m = [2]

- (ii) Using your graph, calculate the time, to the nearest second, when the printing is no longer visible if 20.50 cm³ of **FA 4** is used.

Show your working.

time, *t* = [1]

- (iii) The experimental method can be modified to enable the rate of reaction with respect to hydrogen ions, H^+ , to be investigated.

Plan and conduct **one** experiment to determine the order of reaction with respect to H^+ . Explain your answer.

Record the volumes of reagents and any relevant data in the space below.

Order of reaction with respect to H^+ = [2]

Explanation:

.....

..... [1]

- (iv) When you performed this experiment, you were instructed to carefully dry the beaker with a paper towel before using again.

State and explain the likely effect on t of **not** drying the beaker before it is reused.

effect on t

explanation

.....

..... [1]

- (v) The moment at which the printing is just obscured can be difficult to judge and will vary from person to person.

State which percentage uncertainty is higher for **Experiment 1** or **Experiment 2** in section (a). Explain your answer.

.....

.....

..... [1]

[Total: 15]

3 Qualitative Analysis

Solutions **FA 6** and **FA 7** each contain one metal cation and one anion. All the ions are listed in the Qualitative Analysis Notes.

You will carry out tests to identify the ions present in **FA 6** and **FA 7**.

Unless otherwise stated, the volumes given in Table 3.1 are approximate and should be estimated rather than measured.

Test and identify any gas evolved during the tests.

If there is no observable change, write **no observable change**.

- (a) Carry out the following tests in **Table 3.1**, using a 1 cm depth of **FA 6** or **FA 7** in a test-tube for each test.

Complete the table below. Carefully record your observations in **Table 3.1**. [5]

Table 3.1

<i>test</i>		<i>observations</i>	
		FA 6	FA 7
(i)	Add an equal volume of aqueous potassium iodide.		
(ii)	Add half a small spatula measure of zinc powder. Leave the mixture to stand for 3 min.		
(iii)	Add a few drops of aqueous silver nitrate.		

(iv)	Add aqueous sodium hydroxide.		
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- (b) Fill in the boxes below the identity of the metal cations in **FA 6** and **FA 7**.

	FA 6	FA 7
cation		

[2]

- (c) Both **FA 6** and **FA 7** contain the same anion. Experimental observations due to cations can interfere with that of the anion.

Devise and perform a test that will allow you to confirm the identity of this anion. Your test should be based on the Qualitative Analysis Notes and should use only the bench reagents provided.

Record your test and observations in the space below.

[3]

Anion:

<i>test</i>	<i>observations</i>

- (d) Write ionic equations, with state symbols, to account for the observations for **FA 7** in **3(a)(ii)**.

.....

..... [2]

- (e) Using your observations in **3(a)(ii)**, arrange the metal ions in **FA 6**, **FA 7** and Zn^{2+} in increasing oxidising power. Explain your answer.

.....

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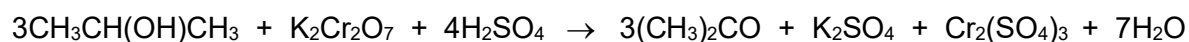
..... [2]

[Total: 14]

4 Planning

Propanone, commonly known as acetone, is primarily used as a highly versatile industrial solvent and an essential chemical building block. It is widely utilised across households, laboratories, and manufacturing sectors.

Propanone is prepared by the oxidation of propan-2-ol. The full equation for this reaction between propan-2-ol and acidified potassium dichromate(VI) to produce propanone is as shown.



Propan-2-ol and aqueous potassium dichromate(VI) are mixed, together with about 6 to 10 drops of concentrated sulfuric acid which catalyse the reaction. The reaction mixture is then heated for about twenty minutes.

The product, propanone, is subsequently separated using simple distillation by maintaining the reaction setup at a suitable temperature.

You are provided with information of some chemicals:

compounds	M_r	melting point / °C	boiling point / °C
propan-2-ol	60.0	-89.0	82.5
propanone	58.0	-94.9	56.5
$\text{K}_2\text{Cr}_2\text{O}_7$	294.2	398.0	500.0

Solubility of $\text{K}_2\text{Cr}_2\text{O}_7 = 0.130 \text{ g cm}^{-3}$

- (a) Calculate the minimum mass of potassium dichromate(VI) that is needed for oxidation of 5.00 g of propan-2-ol to propanone.

[2]

- (b) Write a plan for the oxidation of 5.00 g of propan-2-ol to produce propanone.

In your plan you should include details of:

- the preparation of aqueous potassium dichromate solution.
- the procedure you would follow to produce propanone and obtain pure propanone.
- a labelled diagram of the apparatus you would use to carry out simple distillation.

I	
II	
III	
IV	
V	
VI	

(c) Explain how the student would ensure that all the acid is no longer present.

.....

[1]

[Total: 9]

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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

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(b) Reactions of anions

<i>anion</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 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

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(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 form a white ppt
primary amide	NH_3 liberated on heating with $\text{NaOH}(\text{aq})$

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