CAMBRIDGE
International Examinations

edexcel
advancing learning, changing lives

EXPERIMENTAL CHEMISTRY

Practical No: 1

Duration : 2 hours

Since 2005
Chembase
School of Chemistry

IMPORTANT INSTRUCTIONS:

1. All students are requested to read and understand the laboratory safety rules before entering the laboratory.
2. Instructions for the performance of an experiment should be thoroughly understood and must be followed closely. In case of any doubt DO NOT PROCEED! Ask the teacher.
3. Always work quietly and attentively. Playing practical jokes and other acts of carelessness are strictly prohibited. Remember all times that the laboratory is a place for serious work.
4. Report any accidents and spillages immediately to the teacher.
5. Discard solids into the waste paper baskets. Never throw litmus paper, filter paper, metal pieces or any insoluble solids in to the sinks. Wash down liquids in to the sinks with water.
6. Keep your working area always tidy so that you have sufficient work space and your apparatus clean. Keep your table & floor dry and clear unnecessary obstacles.
7. Report any equipment damaged to the teacher. Never dispose broken glassware without informing the teacher. Never use cracked or broken glassware.

BLANK PAGE

1 You are provided with the following reagents.

- two weighing bottles labelled **FA 1**, each containing between 2.90 g and 3.00 g of zinc powder
- **FA 2**, 0.80 mol dm^{-3} copper sulfate, $CuSO_4$

You are to determine the enthalpy change, Δ*H*, for the following reaction.

$$Zn(s) + CuSO_4(aq) \longrightarrow Cu(s) + ZnSO_4(aq)$$

You will carry out the experimental procedure twice.

Read through the instructions below before starting the experiment.

(a) You will weigh each bottle and later in the experiment weigh it again after the zinc powder has been tipped into copper sulfate solution.
In the space below prepare a table to record the weighings and the mass of zinc powder used in **each** experiment.

Weigh accurately, to at least one decimal place, one of the weighing bottles labelled **FA 1**.
Record this mass in the table you have prepared. [1]

(b) Procedure

- Support the plastic cup in the 250 cm^3 beaker and, using a pipette, place 25.0 cm^3 of **FA 2** into the plastic cup.
- Stir gently, taking a temperature reading every ½ minute until a steady temperature has been obtained for a period of at least 2 minutes. You may need to tilt the beaker in order to cover the bulb of the thermometer with solution.
- On a precise minute reading tip the zinc powder from the weighing bottle into the plastic cup.
  Do not read the temperature at this time or at the following ½ minute.
- Continue to stir the mixture thoroughly. Starting 1 minute after the addition of the zinc powder, record the temperature every ½ minute until the temperature has reached a maximum value and then decreased steadily for at least 5 minutes.
- Reweigh the empty weighing bottle. Record the mass of the bottle + any residual zinc powder and the mass of zinc powder used in the experiment in the table you prepared in **(a)**.
- Record your results in an appropriate form in the space on the following page.

  Repeat the experiment using the contents of the second weighing bottle and 25.0 cm^3 copper sulfate solution pipetted into a clean plastic cup.

For Examiner's Use

(b) continued

Results Make certain your readings of temperature display the precision of the apparatus used.

[11]

(c) Plot your temperature and time readings separately for each experiment on the grids on the next page. Your temperature axis should extend 10 °C **above** the highest temperature you recorded.
Draw lines as instructed below.

On each graph draw a horizontal straight line through the steady initial temperature.

Extrapolate the cooling section of each graph back to the time when you added the zinc powder.
Draw construction lines on the graphs to deduce the "theoretical" **temperature rise** at the moment of mixing the reagents.

experiment 1

experiment 2

For Examiner's Use

[4]

For Examiner's Use

(d) The “theoretical” **temperature rises** are ……………… °C and ……………… °C.

The mean “theoretical” **temperature rise** is ……………… °C. [1]

Calculations

Show working and appropriate significant figures in **all** of your calculations. [2]

(e) Calculate how many moles of copper sulfate, $CuSO_4$, were pipetted into the plastic cup.

……………… mol of $CuSO_4$ were pipetted into the cup

For each experiment calculate how many moles of zinc powder were added to the plastic cup.
[A_r: Zn, 65.4]

1st experiment	2nd experiment

In the 1st experiment ……………… mol of zinc powder were added to the plastic cup.

In the 2nd experiment ……………… mol of zinc powder were added to the plastic cup. [1]

(f) Use your answers to **(e)** and the equation for the reaction to determine which reagent was in excess and which was the limiting reagent. Explain your answer.

$$Zn(s) + CuSO_4(aq) \longrightarrow Cu(s) + ZnSO_4(aq)$$

....

....

.... [1]

For Examiner's Use

(g) From your mean "theoretical" temperature rise at the time of mixing, calculate the heat energy released in the plastic cup by the reaction of zinc powder with copper sulfate solution.
[You may assume that 4.3 J are required to raise the temperature of 1 cm^3 of any solution by 1 °C and that the mass of any solid may be ignored.]

……………… …… of heat energy are released. [1]

(h) Calculate, correct to 3 significant figures, the enthalpy change in $kJ\,mol^{-1}$ for the following reaction.

$$Zn(s) + CuSO_4(aq) \longrightarrow Cu(s) + ZnSO_4(aq)$$

ΔH = ……………………………… $kJ\,mol^{-1}$
[2]

(i) Identify and explain **one** source of error in the experiment you have carried out.

....

....

....

.... [1]

(j) Suggest a way in which the experimental method you used could be improved in a school or college laboratory in order to minimise this error.

....

....

....

.... [1]

[Total: 26]

For Examiner's Use

2 The three boiling-tubes, labelled **FA 3**, **FA 4**, and **FA 5**, each contain a solid with one cation and one anion from those listed on pages 11 and 12.

You will carry out specified tests to deduce the cations and anions present in **FA 3**, **FA 4** and **FA 5**.

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

Where gases are released they should be identified by a test, **described in the appropriate place in your observations**.

You should indicate clearly at what stage in a test a change occurs.
Marks are **not** given for chemical equations.
No additional tests for ions present should be attempted.
If any solution is warmed a boiling-tube MUST be used.

(a) Heat the boiling-tube containing **FA 5** gently at first then more strongly.
Record your observations in the space below.

[2]

(b) In their boiling-tubes, dissolve **FA 3**, **FA 4** and the **cold** residue after heating **FA 5** in a minimum of dilute nitric acid and then add distilled water so that each boiling-tube is approximately ⅔ full. Warm to dissolve if necessary.
Record your observations in the space below.
Use these solutions for tests **(d)**, **(e)** and **(f)**.

[1]

For Examiner's Use

(c) Which anion can be identified from your observations in **(a)** and **(b)**?
Explain your answer.

....

....

....

.... [1]

(d) The cations present in **FA 3**, **FA 4** and **FA 5** can be identified by reaction of each solution with aqueous sodium hydroxide and with aqueous ammonia.
React 1 cm depth of each of the solutions prepared in **(b)** with each of these two reagents.

Record, in an appropriate form in the space below, your observations for these reactions.

i	
ii	
iii	
iv	
v	
vi	

Conclusions

Using your observations you should be able to identify the cation present in two of the solutions. For the remaining solution you should be able to identify two possible cations.

FA 3 contains the cation(s) ……………………………… .

FA 4 contains the cation(s) ……………………………… .

FA 5 contains the cation(s) ……………………………… .

[6]

(e) Use the information on pages 11 and 12 to select a reagent to distinguish between the two possible cations identified as present in one of the solutions in **(d)**.

Carry out the test with the selected reagent.

reagent ....

observation ....

conclusion ....
[2]

(f) Carry out the following tests.

test	*observations*		
	FA 3	**FA 4**	**FA 5**
To 1 cm depth of solution in a test-tube, add 1 cm depth of aqueous barium nitrate, then			
add 2 cm depth of dilute nitric acid.			
To 1 cm depth of solution in a test-tube, add 1 cm depth of aqueous silver nitrate, then			
allow any precipitate formed to settle, pour off the solution and add aqueous ammonia to the precipitate.			

What conclusions can be made from the observations above?

....

....

.... [2]

[Total: 14]

Qualitative Analysis Notes

Key: [ppt. = precipitate]

1 Reactions of aqueous cations

	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)	no ppt. 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
lead(II), Pb^{2+}(aq)	white ppt. soluble in excess	white 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

[Lead(II) ions can be distinguished from aluminium ions by the insolubility of lead(II) chloride.]

2 Reactions of anions

ion	*reaction*
carbonate, CO_3^{2-}	CO_2 liberated by dilute acids
chromate(VI), CrO_4^{2-} (aq)	yellow solution turns orange with H^+(aq); gives yellow ppt. with Ba^{2+}(aq); gives bright yellow ppt. with Pb^{2+}(aq)
chloride, Cl^- (aq)	gives white ppt. with Ag^+(aq) (soluble in NH_3(aq)); gives white ppt. with Pb^{2+}(aq)
bromide, Br^- (aq)	gives pale cream ppt. with Ag^+(aq) (partially soluble in NH_3(aq)); gives white ppt. with Pb^{2+}(aq)
iodide, I^- (aq)	gives yellow ppt. with Ag^+(aq) (insoluble in NH_3(aq)); gives yellow ppt. with Pb^{2+}(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) or with Pb^{2+}(aq) (insoluble in excess dilute strong acid)
sulfite, SO_3^{2-} (aq)	SO_2 liberated with dilute acids; gives white ppt. with Ba^{2+}(aq) (soluble in excess dilute strong acid)

3 Tests 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 acidified aqueous potassium dichromate(VI) (aq) from orange to green