

UNIVERSITY OF CAMBRIDGE INTERNATIONAL EXAMINATIONS General Certificate of Education

Advanced Subsidiary Level and Advanced Level

CANDIDATE NAME				
CENTRE NUMBER		CANDIDATE NUMBER		
CHEMISTRY			 97	701/3

May/June 2011

Advanced Practical Skills

2 hours

Candidates answer on the Question Paper.

Additional Materials:

As listed in the Instructions to Supervisors

READ THESE INSTRUCTIONS FIRST

Write your Centre number, candidate number and name on all the work you hand in.

Give details of the practical session and laboratory where appropriate, in the boxes provided.

Write in dark blue or black pen.

You may use a soft pencil for any diagrams, graphs or rough working.

Do not use staples, paper clips, highlighters, glue or correction fluid.

DO NOT WRITE IN ANY BARCODES.

Answer all questions.

You may lose marks if you do not show your working or if you do not use appropriate units.

Use of a Data Booklet is unnecessary.

Qualitative Analysis Notes are printed on pages 14 and 15.

At the end of the examination, fasten all your work securely together. The number of marks is given in brackets [] at the end of each question or part question.

	Session
ľ	Laboratory

For Examiner's Use		
1		
2		
3		
Total		

This document consists of 15 printed pages and 1 blank page.



You are to determine the percentage by mass of water in the borax crystals.

For Examiner's Use

Borax reacts with hydrochloric acid according to the equation.

$$Na_2B_4O_7(aq) + 2HCl(aq) + 5H_2O(l) \rightarrow 2NaCl(aq) + 4H_3BO_3(aq)$$

1 FA 1 is an aqueous solution containing $38.10\,\mathrm{g\,dm^{-3}}$ of **borax** crystals. **Borax** has the formula, $\mathrm{Na_2B_4O_7.xH_2O.}$

FA 2 is 1.00 mol dm⁻³ hydrochloric acid, HCl.

You are also provided with an indicator suitable for the titration of a strong acid and a weak base.

The indicator provided is

(a) Method

Dilution

- Fill the burette with FA 2.
- Run between 44.50 cm³ and 45.50 cm³ of **FA 2** from the burette into the 250 cm³ graduated (volumetric) flask, labelled **FA 3**.
- Make the solution up to the mark with distilled water.
- Shake the flask to mix the solution of FA 3.

In the space below record your burette readings and the volume of **FA 2** added to the graduated flask.

Titration

- Fill a second burette with **FA 3**, the diluted hydrochloric acid.
- Pipette 25.0 cm³ of **FA 1** into a conical flask.
- Add to the flask a few drops of the indicator provided.
- Titrate the borax in the flask with FA 3 until the appropriate colour change is observed for the end-point.

You should perform a **rough titration**.

In the space below record your burette readings for this rough titration.

The rough titre is cm³.

	J
•	Carry out as many accurate titrations as you think necessary to obtain consistent results.
•	Make certain any recorded results show the precision of your practical work.
•	Record in a suitable form below all of your burette readings and the volume of FA 3 added in each accurate titration.

For Examiner's Use

I	
II	
III	
IV	
V	
VI	
VII	

[7]

(b) From your accurate titration results obtain a suitable value to be used in your calculations. Show clearly how you have obtained this value.

 $25.0 \,\mathrm{cm^3}$ of **FA 1** required cm³ of **FA 3**. [1]

(c) Calculations

Show your working and appropriate significant figures in the final answer to **each** step of your calculations.

(i) Calculate the concentration of hydrochloric acid, in mol dm⁻³, in the diluted solution, FA 3.

The concentration of HCl in **FA 3** was mol dm⁻³.

(ii) Calculate how many moles of HC1 were present in the volume of FA 3 calculated in (b).

..... mol of HC1

	(iii)	Calculate how many moles of $Na_2B_4O_7$ reacted with the HC l in (ii).	For Examiner's Use
		$Na_2B_4O_7(aq) + 2HCl(aq) + 5H_2O(l) \rightarrow 2NaCl(aq) + 4H_3BO_3(aq)$	USE
		The HCl run from the burette reacted with mol of Na ₂ B ₄ O ₇ .	
	(iv)	Calculate the concentration, in $g dm^{-3}$, of $Na_2 B_4 O_7$ in FA 1 . [A_r : B, 10.8; O, 16.0; Na, 23.0]	
			I
			II
		The concentration of $Na_2B_4O_7$ in FA 1 is gdm ⁻³ .	III
	(v)	Use your answer to (iv) and the information at the start of question 1 to calculate	IV
		the percentage by mass of water in the borax crystals, $Na_2B_4O_7$. x H_2O . [A_r : H, 1.0; B, 10.8; O, 16.0; Na, 23.0]	V
			VI
		Borax crystals contain % water.	
(d)		maximum error for a 25 cm ³ pipette commonly used in schools is ±0.06 cm ³ . maximum error in any single burette reading is ±0.05 cm ³ .	
	Cal	culate the maximum percentage error in each of the following.	
	(i)	The volume of FA 1 pipetted into the conical flask.	
		maximum percentage error in the pipetted volume =%.	
	(ii)	The titre volume calculated in (b) .	
		maximum percentage error in titre volume =%. [1]	
		[Total: 15]	

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2 Read through the question carefully before starting any practical work.

For Examiner's Use

Sodium carbonate, Na₂CO₃,

- reacts exothermically with hydrochloric acid,
- does not decompose when heated,
- reacts with acids. $CO_3^{2-}(s) + 2H^+(aq) \rightarrow H_2O(I) + CO_2(g)$

Sodium hydrogencarbonate, NaHCO₃,

- reacts endothermically with hydrochloric acid,
- decomposes when heated, $2HCO_3^-(s) \rightarrow H_2O(g) + CO_2(g) + CO_3^{2-}(s)$
- reacts with acids. $HCO_3^-(s) + H^+(aq) \rightarrow H_2O(l) + CO_2(g)$

You are to measure the temperature changes when samples of

- (i) sodium carbonate,
- (ii) sodium hydrogencarbonate,
- (iii) a mixture of sodium carbonate and sodium hydrogencarbonate, react with an excess of hydrochloric acid.

FA 4 is sodium carbonate, Na₂CO₃.

FA 5 is sodium hydrogencarbonate, NaHCO₃.

FA 6 is a mixture of sodium carbonate and sodium hydrogencarbonate.

FA 7 is 3.0 mol dm⁻³ hydrochloric acid, HC*l*.

Method

(a) sodium carbonate

- Support the plastic cup in a 250 cm³ beaker.
- Use a measuring cylinder to transfer 50 cm³ of **FA 7** into the plastic cup.
- Measure and record the temperature of the acid in the cup.
- Measure and record the mass of the container labelled FA 4, containing Na₂CO₃.
- Carefully tip the sodium carbonate from the weighed container FA 4 into the hydrochloric acid in the plastic cup.

Note: There will be vigorous effervescence. Do not breathe the vapour. Add the solid in small portions with constant stirring using the thermometer.

- Record the highest temperature obtained.
- Reweigh the container **FA 4** with any residual sodium carbonate. Record the mass.
- Empty and rinse the plastic cup and dry it using a paper towel.

In the space at the top of the next page, record, in an appropriate form,

- both balance readings and both temperature measurements,
- the mass of sodium carbonate, **m**₄, used in the experiment,
- the temperature rise, ΔT_1 .

For Examiner's Use			
I			
II			
III			
IV			

[4]

Calculate the rise in temperature for each gram of sodium carbonate used in the experiment.

$$\frac{\Delta T_1}{m_1} = \boxed{+}$$
 sign value

(b) sodium hydrogencarbonate

- Support the plastic cup in a 250 cm³ beaker.
- Use a measuring cylinder to transfer 50 cm³ of **FA 7** into the plastic cup.
- Measure and record the temperature of the acid in the cup.
- Measure and record the mass of the container labelled FA 5, containing NaHCO₃.
- Carefully tip the sodium hydrogencarbonate from the weighed container FA 5 into the hydrochloric acid in the plastic cup.

Note: There will be vigorous effervescence. Add the solid in small portions with constant stirring using the thermometer.

- Record the lowest temperature obtained.
- Reweigh the container **FA 5** with any residual sodium hydrogencarbonate. Record the mass.
- Empty and rinse the plastic cup and dry it using a paper towel.

In the space below, record, in an appropriate form,

- both balance readings and both temperature measurements,
- the mass of sodium hydrogencarbonate, **m**₂, used in the experiment,
- the temperature fall, ΔT_2 .

I II

[2]

Calculate the fall in temperature for each gram of sodium hydrogencarbonate used in the experiment.

(c) mixture of sodium carbonate and sodium hydrogencarbonate

For Examiner's Use

- Support the plastic cup in a 250 cm³ beaker.
- Use a measuring cylinder to transfer 50 cm³ of **FA 7** into the plastic cup.
- Measure and record the temperature of the acid in the cup.
- Measure and record the mass of a clean, dry, weighing-bottle or tube.
- Add to the tube between **8.5 g and 9.5 g** of the mixture **FA 6**.
- Record the mass of the weighing-bottle or tube + FA 6.
- Carefully tip the weighed mixture into the hydrochloric acid in the plastic cup.
- Note: There will be vigorous effervescence. Add the solid in small portions with constant stirring using the thermometer.
- Record the highest or lowest temperature obtained.
- Reweigh the weighing-bottle or tube with any residual mixture. Record the mass.

In the space below, record, in an appropriate form,

- all balance readings and temperature measurements,
- the mass of the mixture, **m**₃, used in the experiment,
- the temperature change, $\Delta \tilde{\mathbf{T}}_3$.

Make certain that your recorded temperature change carries an appropriate sign.

I	
II	

[2]

(d) Transfer the following data from parts (a), (b) and (c).

(a)
$$\frac{\Delta T_1}{m_1} = +$$
°C g⁻¹

(b)
$$\frac{\Delta T_2}{m_2} = -$$
°Cg⁻¹

(c)
$$m_3 = \dots$$
 g $\Delta T_3 =$ sign

The masses of sodium carbonate and sodium hydrogencarbonate in the weighed sample of the mixture used in experiment (c) can be represented as follows.

For Examiner's Use

mass of sodium carbonate / g = W

mass of sodium hydrogenearbonate / $g = (m_3 - W)$

Evaluate the following equation to determine a value for W.

$$[W \times \frac{\Delta T_1}{m_1}] + [(m_3 - W) \times \frac{\Delta T_2}{m_2}] = \Delta T_3$$

(e)	Use the information at the beginning of question 2 to outline an alternative method the could be used in a school laboratory to find the mass of sodium carbonate and the mass of sodium hydrogenicarbonate in the mixture FA 6 .	
		[1]

The mass of sodium carbonate wasg.

[Total: 10]

3 Qualitative Analysis

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

For Examiner's Use

- 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.

Rinse and reuse test-tubes and boiling tubes where possible.

Where reagents are selected for use in a test, the full name or correct formula of the reagents must be given.

FA 8, **FA 9** and **FA 10** are aqueous solutions each containing a sodium cation and a single anion which could be a nitrite, a nitrate or a halide.

(a) By reference to the Qualitative Analysis Notes on page 15, select a single reagent that

would enable you to identify any solution containing the nitrite ion, NO ₂ ⁻ .
reagent
Use this reagent to test each of the solutions. Record your observations in the table below. State clearly where no reaction has been observed.

solution	observation
FA 8	
FA 9	
FA 10	

	·~·	
•	٠,	

(b)	By reference to the Qualitative Analysis	Notes	on page	15,	select	one	reagent	that
	would show that a halide ion is present.							

Use this reagent to test each of the solutions.
Record your observations in an appropriate form in the space below.

For Examiner's Use

	Select another reagent to identify or confirm which halide ions are present in t olutions.	he
r	eagent	
T	ick the appropriate statement about the use of this reagent.	
	It is added to the tube already containing the first reagent.	
	It is added to a fresh sample of solution.	

Use **this reagent** to identify or confirm which halide ions are present in the solutions and record your observations in an appropriate form in the space below.

[3]

I

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III

(c) From the results in (a) and (b) state which anions have been identified in the solutions.

Complete the following table.

Place a cross in any box if no anion has been identified.

solution	FA 8	FA 9	FA 10
anion present			

[1]

FA 11 and FA 12 are aqueous	solutions each containing	one cation from	n those listed in the
Qualitative Analysis Notes prin	ted on page 14.		

For Examiner's Use

(d)	Use aqueous sodium hydroxide and aqueous ammonia in separate tests to identify the
	cation present in each of the solutions.

You will also require some of the solution, FA 11, for tests in (f).

Record the results of your experiments with sodium hydroxide and ammonia in an appropriate form in the space below.

I	
II	
III	

I

II

[3]

(e) Identification of the cations in FA 11 and FA 12

Complete the table below.

solution	FA 11	FA 12
cation present		

What is the evidence **from your observations in (d)** that enables you to identify the cation present in each of the solutions?

The evidence supporting the conclusion for the cation in FA 11 is
The evidence supporting the conclusion for the cation in FA 12 is
[2]

(f) Complete the following table.

For Examiner's Use

	test	observations
(i)	Pour 1 cm depth of FA 11 into a test-tube.	
	Add 1 cm depth of aqueous potassium iodide.	
	Divide this mixture into two parts for use in (ii) and (iii)	
(ii)	To the first part of the mixture from (i) add a few drops of starch solution.	
(iii)	To the second part of the mixture from (i) add aqueous sodium thiosulfate, a drop at a time, until no further change is observed.	

In part (i) and in part (iii) redox reactions have taken place.

Complete the table below to show the ion or molecule which has been oxidised and the ion or molecule which has been reduced in each of these reactions.

racation	the ion or molecule which has been		
reaction	oxidised	reduced	
(i)			
(iii)			

I	
II	
III	
IV	

[4]

[Total: 15]

Qualitative Analysis Notes

Key: [ppt. = precipitate]

1 Reactions of aqueous cations

_	reaction with		
ion	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 ₂ liberated by dilute acids	
CO ₃ ²⁻		
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,	gives white ppt. with Ag ⁺ (aq) (soluble in NH ₃ (aq));	
C <i>l</i> ⁻(aq)	gives white ppt. with Pb ²⁺ (aq)	
bromide,	gives cream ppt. with Ag ⁺ (aq) (partially soluble in NH ₃ (aq));	
Br ⁻ (aq)	gives white ppt. with Pb ²⁺ (aq)	
iodide,	gives yellow ppt. with Ag ⁺ (aq) (insoluble in NH ₃ (aq));	
I⁻(aq)	gives yellow ppt. with Pb ²⁺ (aq)	
nitrate,	$\mathrm{NH_3}$ liberated on heating with $\mathrm{OH^-}(\mathrm{aq})$ and $\mathrm{A}\mathit{l}$ foil	
NO ₃ ⁻ (aq)		
nitrite,	$\mathrm{NH_3}$ liberated on heating with $\mathrm{OH^-}(\mathrm{aq})$ and $\mathrm{A}\mathit{l}$ foil;	
NO ₂ ⁻ (aq)	NO liberated by dilute acids (colourless NO → (pale) brown NO ₂ in air)	
sulfate,	gives white ppt. with Ba ²⁺ (aq) or with Pb ²⁺ (aq) (insoluble in excess dilute	
SO ₄ ²⁻ (aq)	strong acids)	
sulfite,	SO ₂ liberated with dilute acids;	
SO ₃ ²⁻ (aq)	gives white ppt. with Ba ²⁺ (aq) (soluble in excess dilute strong acids)	

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) from orange to green

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