

CANDIDATE
NAME

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CENTRE
NUMBER

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CANDIDATE
NUMBER

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CHEMISTRY

9701/33

Paper 3 Advanced Practical Skills 1

May/June 2015

2 hours

Candidates answer on the Question Paper.

Additional Materials: As listed in the Confidential Instructions

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 an HB pencil for any diagrams or graphs.
Do not use staples, paper clips, glue or correction fluid.
DO NOT WRITE IN ANY BARCODES.

Answer **all** questions.
Electronic calculators may be used.
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 10 and 11.

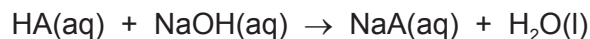
At the end of the examination, fasten all your work securely together.
The number of marks is given in brackets [] at the end of each question or part question.

Session	
Laboratory	

For Examiner's Use	
1	
2	
3	
Total	

This document consists of **10** printed pages and **2** blank pages.

- 1 HA is an organic acid. Solution **FA 1** was prepared by dissolving 13.1 g of solid HA in each dm³ of solution. You are to determine the relative molecular mass, M_r , of HA by titration with aqueous sodium hydroxide. The equation for the reaction between HA and sodium hydroxide is given below.



FA 1 is a solution containing 13.1 g dm⁻³ of organic acid, HA.

FA 2 is 0.100 mol dm⁻³ sodium hydroxide, NaOH.

bromothymol blue indicator

(a) Method

- Fill the burette with **FA 1**.
- Use the pipette to transfer 25.0 cm³ of **FA 2** into a conical flask.
- Add a few drops of bromothymol blue indicator. This indicator is blue in alkali and yellow in acid solutions.
- Perform a **rough titration** and record your burette readings in the space below.

The rough titre is cm³.

- 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 1** added in each accurate titration.

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 obtained this value.

25.0 cm³ of **FA 2** required cm³ of **FA 1**. [1]

(c) Calculations

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

- (i) Calculate the number of moles of sodium hydroxide present in the volume of **FA 2** pipetted into the conical flask.

moles of NaOH = mol

- (ii) Use your answer to **(c)(i)** and the equation on page 2 to determine the number of moles of organic acid, HA, used to neutralise 25.0 cm³ of **FA 2**.

moles of HA = mol

- (iii) Use your answers to **(b)** and **(c)(ii)** to calculate the number of moles of HA in 1 dm³ of **FA 1**.

moles of HA in 1 dm³ of **FA 1** = mol

- (iv) Calculate the relative molecular mass, M_r , of the organic acid, HA.

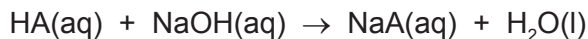
M_r of HA =
[4]

- (d) A student carrying out this method correctly with the same concentration of reactants obtained a titre of 28.30 cm³. Would this give a larger or smaller value of M_r than yours?
Explain your answer.

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

[Total: 13]

- 2 You are to determine the enthalpy change for the neutralisation reaction given below.



You will be using solutions of different concentrations from those in **Question 1**.

FA 3 is 1.80 mol dm^{-3} HA.

FA 4 is aqueous sodium hydroxide, NaOH.

(a) Method

Read through the instructions carefully and prepare a table below for your results before starting any practical work.

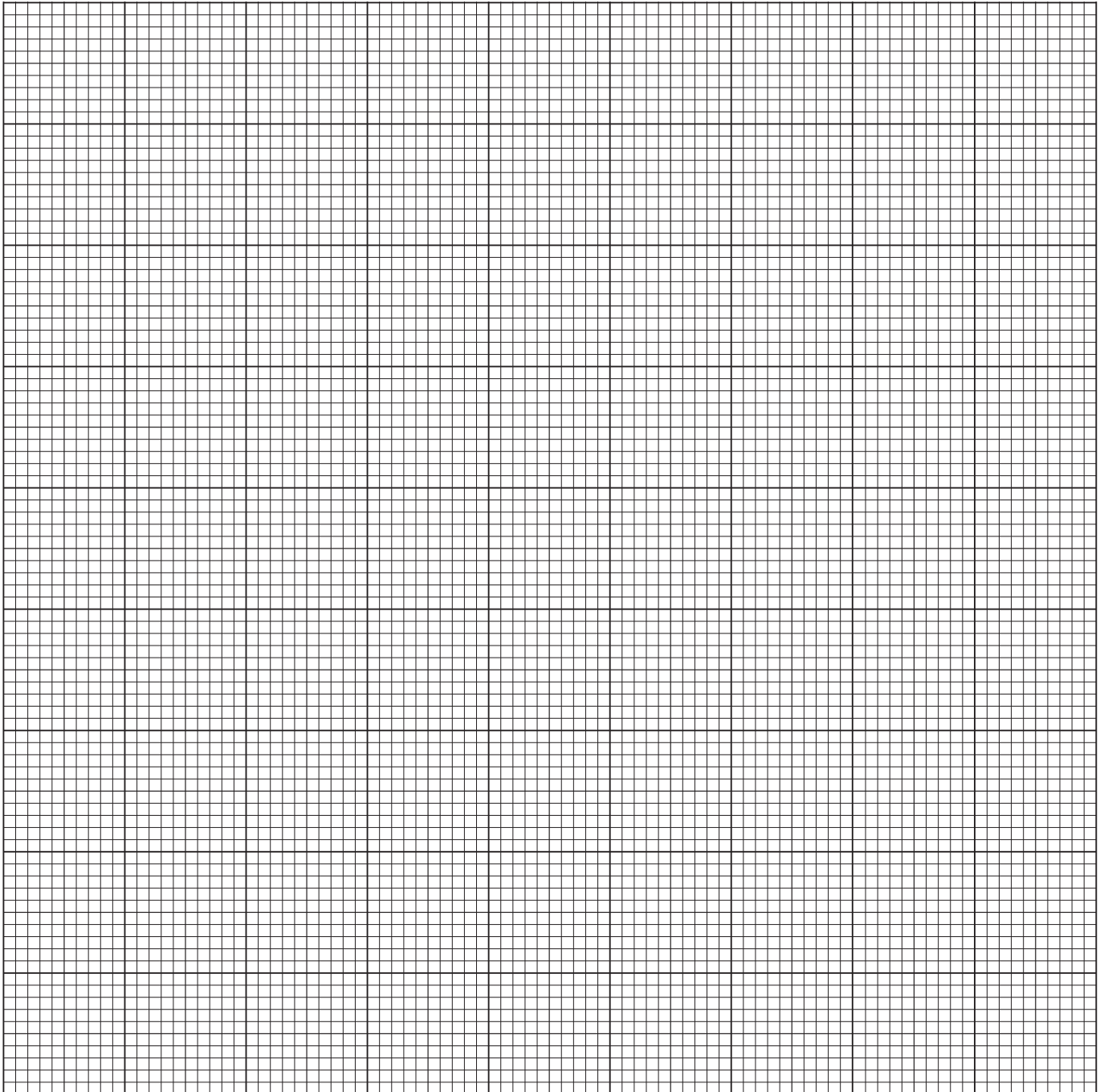
- Support the plastic cup in the 250 cm^3 beaker.
- Rinse and fill the burette with **FA 3**.
- Use the measuring cylinder to transfer 25 cm^3 of **FA 4** into the plastic cup.
- Place the thermometer in the plastic cup and record the temperature of the solution. Tilt the cup if necessary to ensure the thermometer bulb is fully immersed.
- Run 5.00 cm^3 of **FA 3** into the cup. Stir, and record the new temperature of the solution and the volume of **FA 3** added.
- Run a second 5.00 cm^3 of **FA 3** into the cup. Stir and record the new temperature and the total volume of **FA 3** added.
- Continue adding **FA 3** in 5.00 cm^3 portions. Stir and record each new temperature and total volume of **FA 3** until a total of 45.00 cm^3 has been added.

Results

I	
II	
III	
IV	

[4]

- (b)** Plot a graph of temperature (*y*-axis) against total volume of **FA 3** added (*x*-axis) on the grid opposite. The temperature axis should allow you to include a point at least 2°C greater than the maximum temperature recorded.



Draw the best fit smooth curve or straight line through the two sets of points, one for the increase in temperature of the mixture and the other for the cooling of the solution once the reaction is complete. Extrapolate the two lines and determine the maximum **increase** in temperature and the corresponding volume of **FA 3** added for this increase in temperature.

I	
II	
III	
IV	

maximum temperature **increase** = °C

volume of **FA 3** = cm³
[4]

(c) Calculations

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

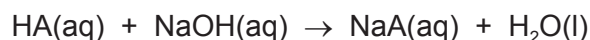
- (i) Calculate the number of moles of HA present in the volume of **FA 3** recorded in (b).

moles of HA = mol

- (ii) Using your answers to (b), calculate the heat energy produced when **FA 3** neutralised 25 cm³ of sodium hydroxide.
(Assume that **4.2 J** of heat energy changes the temperature of 1.0 cm³ of solution by 1.0 °C.)

heat energy produced = J

- (iii) Calculate the enthalpy change of neutralisation, in kJ mol⁻¹, for the reaction below.



enthalpy change = kJ mol⁻¹
(sign) (value)

[4]

- (d) The maximum error in a single thermometer reading is ±0.5 °C.
Calculate the maximum percentage error in the increase in temperature recorded in (b).

maximum percentage error = % [1]

- (e) When carrying out thermochemistry experiments in an A Level laboratory, the plastic cup is usually placed in a glass beaker. Give a reason for the use of the glass beaker.

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

- (f) Apart from using a thermometer calibrated to a greater level of precision, suggest one improvement that could be made to the **method** carried out in (a).

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

[Total: 15]

3 Qualitative Analysis

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.

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

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

Before starting your practical work, half fill the 250 cm³ beaker with water. Heat it to approximately 80 °C, then turn off the Bunsen burner. This will be used as a hot water bath.

(a) **FA 5** is the salt of another organic acid.

FA 6 is a salt containing two cations and one anion from those listed in the Qualitative Analysis Notes on pages 10 and 11.

Carry out the following tests and record your observations in the table below.

<i>test</i>	<i>observations</i>
(i) To a 1 cm depth of dilute sulfuric acid in a test-tube, add five drops of potassium manganate(VII) then a small spatula measure of FA 5 . Shake the tube then place it in the hot water bath.	
(ii) Place a spatula measure of FA 6 in a hard-glass test-tube. Heat gently and test any gas evolved with litmus paper.	
(iii) To a 4 cm depth of water in a boiling tube, add and dissolve a spatula measure of FA 6 . Use portions of this solution, aqueous FA 6 , for tests (iv)–(vi).	X

<i>test</i>	<i>observations</i>
(iv) To a 1 cm depth of aqueous FA 6 in a test-tube, add aqueous ammonia.	
(v) To a 1 cm depth of aqueous FA 6 in a test-tube, add a 1 cm depth of aqueous barium chloride followed by dilute hydrochloric acid or a 1 cm depth of aqueous barium nitrate followed by dilute nitric acid.	
(vi) To a 1 cm depth of aqueous FA 6 in a test-tube, add a small spatula measure of FA 5 and shake the mixture. Keep this mixture for the test in part (b).	

(vii) Identify as many of the ions in **FA 6** as possible from your observations.

FA 6 contains

[9]

(b) A student suggests that one of the cations in **FA 6** was oxidised in test (a)(vi).

(i) Name a reagent you would use to find out whether the student was correct.

reagent

(ii) Use your reagent to carry out a test on the mixture from (a)(vi). Record your observations below. State and explain whether the student was correct.

observations

.....

conclusion

[3]

[Total: 12]

Qualitative Analysis Notes

Key: [ppt. = precipitate]

1 Reactions of aqueous cations

ion	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
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

2 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 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)

3 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 acidified aqueous potassium manganate(VII) from purple to colourless

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