





UNIVERSITY OF CAMBRIDGE INTERNATIONAL EXAMINATIONS General Certificate of Education Advanced Subsidiary Level and Advanced Level

CANDIDATE NAME		
CENTRE NUMBER	CANDIDATE NUMBER	
CHEMISTRY		9701/35

CHEMISIKI

Advanced Practical Skills 1

October/November 2013

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 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.

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	
Labaratani	
Laboratory	
	Session Laboratory

For Examiner's Use		
1		
2		
3		
Total		

This document consists of 11 printed pages and 1 blank page.



1 You are provided with a solution of an organic acid which is known to be one of the following.

For Examiner's Use

```
methanoic acid, HCOOH
propanoic acid, C<sub>2</sub>H<sub>5</sub>COOH
pent-2-enoic acid, CH<sub>3</sub>CH<sub>2</sub>CH=CHCOOH
```

The solution was made by dissolving 1.85 g of acid in 250 cm³ of solution.

You are to suggest the identity of the acid by finding its relative molecular mass, M_r , using a titration method.

FA 1 is the solution of the unknown organic acid. **FA 2** is 0.100 mol dm⁻³ sodium hydroxide, NaOH. phenolphthalein indicator

(a) Method

- Fill the burette with FA 2.
- Pipette 25.0 cm³ of **FA 1** into a conical flask.
- Titrate FA 1 with FA 2 using phenolphthalein as indicator.
- Perform a rough titration and record your burette readings in the space below.

The rough titre is	cm ³
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- 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 2 added in each accurate titration.

I	
II	
III	
IV	
V	
VI	
VII	

[7]

(b)		m your accurate titration results, obtain a suitable value to be used in your calculations. by clearly how you obtained this value.	For Examiner's Use
		25.0 cm ³ of FA 1 required cm ³ of FA 2 [1]	
(c)	Cal	culations	
		ow your working and appropriate significant figures in the final answer to each step of a calculations.	
	(i)	Calculate the number of moles of sodium hydroxide in the volume of FA 2 you have calculated in (b) .	
		moles of NaOH = mol	
	(ii)	One mole of any of the organic acids reacts with one mole of sodium hydroxide. Calculate the concentration, in mol dm ⁻³ , of the acid in FA 1 .	
		concentration of the acid in FA 1 = mol dm ⁻³	
	(iii)	Calculate the concentration, in g dm ⁻³ , of the acid used to make solution FA 1 .	
		concentration of the acid in FA 1 = g dm ⁻³	
	(iv)	Using your answers to (ii) and (iii), calculate the relative molecular mass, $M_{\rm r}$, of the acid in FA 1 .	
		$M_{\rm r}$ of the acid =	

For

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(v)	Suggest which of the acids, methanoic, propanoic or pent-2-enoic acid, is present in FA 1 .	For Examiner's Use
	Acid present is	
(vi)	Suggest a test that could be carried out to distinguish pent-2-enoic acid from methanoic acid and propanoic acid. Give the expected result of your test.	
	test	
	expected result[5]	
	[Total: 13]	

2 The formula of hydrated iron(II) sulfate is FeSO₄.**x**H₂O where **x** shows the number of moles of water of crystallisation.

For Examiner's Use

The value of \mathbf{x} can be found by heating solid hydrated iron(II) sulfate to remove the water of crystallisation.

FA 3 is hydrated iron(II) sulfate, FeSO₄.**x**H₂O.

(a) Method

Record all weighings, in an appropriate form, in the space below.

- Weigh and record the mass of the empty crucible.
- Tip the contents of the tube labelled **FA 3** into the weighed crucible. Reweigh and record the mass of the crucible and **FA 3**.
- Use a pipe-clay triangle to support the crucible and contents on a tripod.
- Heat **gently** for about three minutes.
- Leave the crucible to cool for approximately five minutes.

While you are waiting for the crucible to cool, start work on Question 3.

- When cool, reweigh the crucible with the residue.
- Reheat gently for three minutes, cool and reweigh the crucible until you are satisfied
 that all the water of crystallisation has been lost. It should not be necessary to reheat
 the crucible more than three times.

I	
II	
III	
IV	
V	
VI	

[6]

For Examiner's Use

(b)	(i)	Calculate the mass of water lost and the mass of anhydrous iron($\rm II$) sulfate that remains after the heating process.
		mass of water lost = g
		mass of anhydrous iron(II) sulfate = g
	(ii)	Determine the value of $\bf x$ in the formula of hydrated iron(II) sulfate, FeSO ₄ . $\bf x$ H ₂ O. (A_r : H, 1.0; O, 16.0; S, 32.1; Fe, 55.8)
		value of x =[3]
(c)		roup of students carried out this practical and made their measurements correctly. e students calculated a value of 9 for x . The textbook value of x is less than 9.
	(i)	Suggest an error in the practical procedure of the experiment that could account for this result and explain why this gives a value of ${\bf x}$ that is too high.
	(ii)	Suggest a modification that could be made to the experimental procedure to reduce this error. Explain why this modification should give an answer for ${\bf x}$ that is closer to the textbook value.
		[3]
		[Total: 12]

3 Qualitative Analysis

For Examiner's Use

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.

(a)	You are provided with a solid FA 4 . FA 4 is a mixture that contains two cations and two anions.		
	(i)	Place a spatula measure of FA 4 in a hard-glass test-tube. Heat the solid and identify the gas given off. Record all your observations.	
	(ii)	To a spatula measure of FA 4 in a test-tube, add a 1 cm depth of dilute nitric acid. Record your observations.	

For Examiner's Use

(111)	distilled water to make a solution. Divide the solution into two portions.
	To the first portion, add a 1 cm depth of aqueous sodium hydroxide. Record your observations.
	To the second portion, add a few drops of aqueous silver nitrate, then add a 1 cm depth of dilute nitric acid. Record your observations.
(iv)	Use your results from (i) to (iii) to identify two anions and one cation that are present in FA 4.
	anions present
	cation present
(v)	What further test could be carried out on FA 4 to confirm the presence of the cation you suggested in (iv) ? You should state the reagent to be used and the expected result.
	Do not carry out this test.
(vi)	To a spatula measure of ${\bf FA~4}$ in a test-tube, add a 1 cm depth of distilled water to make a solution. To this solution, add a few drops of aqueous barium chloride or barium nitrate.
	Describe the appearance of the precipitate formed and state its identity.
	appearance of precipitate
	identity of precipitate
	[9]

(b) FA 5, FA 6, FA 7 and FA 8 are aqueous solutions each containing one of the ions Al^{3+} , Ca^{2+} , Zn^{2+} and Pb^{2+} .

For Examiner's Use

(i) Carry out the following tests. Record your observations in the spaces provided in the table.

((observations				
test –	FA 5	FA 6	FA 7	FA 8	
To a 1 cm depth of solution in a test-tube, add a few drops of aqueous sodium hydroxide, then					
add excess aqueous sodium hydroxide.					
To a 1 cm depth of solution in a test-tube, add a few drops of aqueous ammonia, then					
add excess aqueous ammonia.					
To a 1 cm depth of solution in a test-tube, add a 1 cm depth of aqueous potassium iodide.					

(ii)	Use the Qualitative Analysis Notes on page 10 to identify the cation present in each
	of the solutions.

FA 5 is	, FA	6 is,	FA 7 is,	FA 8 is
	•	•	•	

[6]

[Total: 15]

Qualitative Analysis Notes

Key: [ppt. = precipitate]

1 Reactions of aqueous cations

,	reaction with			
ion	NaOH(aq)	NH ₃ (aq)		
aluminium, Al³+(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), Cr³+(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), Fe²+(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), Fe³+(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, Zn²+(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.]

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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 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 A <i>l</i> 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 acids)
sulfite, SO ₃ ²⁻ (aq)	SO ₂ liberated with dilute acids; 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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