



Cambridge International Examinations  
Cambridge Pre-U Certificate

CANDIDATE  
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**CHEMISTRY**

Paper 4 Practical

**9791/04**

**May/June 2014**

**2 hours**

Candidates answer on the Question Paper.

Additional Materials: As listed in the Confidential Instructions  
Data Booklet

**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.  
A Data Booklet is provided.

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.

<b>Session</b>
<b>Laboratory</b>

<b>For Examiner's Use</b>	
1	
2	
3	
<b>Total</b>	

This document consists of **9** printed pages and **3** blank pages.

- 1 **FA 1** is a mixture of barium chloride dihydrate,  $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$ , and one other barium salt. When barium chloride dihydrate is heated the anhydrous salt is formed.



You will first measure the mass lost on heating the mixture and so determine the percentage by mass of the barium chloride dihydrate.

You will then identify the anion in the second salt.

**(a) Method**

**Before starting any practical work, read through all the instructions and prepare a table for your results in the space provided.**

**You have been provided with two samples of FA 1.**

1. Weigh a clean dry crucible without a lid and record your reading.
2. Place in the crucible all of the contents of one of the containers of **FA 1**.
3. Reweigh the crucible and record your reading.
4. Support the crucible in the pipe-clay triangle on top of a tripod.
5. Heat the crucible **gently** for about 1 minute and then **strongly** for a further 8 minutes.
6. Allow the crucible to cool.  
**While the crucible is cooling you are advised to start Question 2.**
7. As soon as the crucible is cool enough to touch, reweigh the crucible and its contents and record your reading.
8. Calculate and record the initial mass of the mixture and the mass of water lost.

I	
II	
III	
IV	

[4]

- (b) By performing the following calculations you will determine the percentage by mass of barium chloride dihydrate in the mixture.

**You must show your working in each step of your calculations.**

- (i) Calculate the amount, in mol, of water lost from the sample of **FA 1**.

..... mol

- (ii) Calculate the percentage by mass of barium chloride dihydrate,  $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$ , present in **FA 1**.

I	
II	
III	

..... %  
[3]

- (c) Place approximately half of the second sample of **FA 1** into a test-tube and add cautiously some dilute hydrochloric acid. Do **not** use the hydrochloric acid labelled **FA 3**.

Test any gases that are given off and record all your observations in the space below. Identify the anion present in the second salt.

The anion in the second salt is ..... [3]

- (d) In determining the percentage by mass of barium chloride dihydrate in **FA 1**, it is necessary to assume that the second salt has a particular property.

State what this property is and explain why this is essential for the method to be valid.

.....  
.....  
..... [2]

[Total: 12]

- 2 A student was given a sample containing a mixture of ammonium chloride and one other salt. The student was told to determine the percentage by mass of ammonium chloride in the sample. To do this, the student carried out the following steps.

step 1 1.40 g of the mixture was dissolved in exactly 250.0 cm<sup>3</sup> of 0.200 mol dm<sup>-3</sup> sodium hydroxide, NaOH.

step 2 This solution was transferred to a large conical flask and heated. The sodium hydroxide reacted with the ammonium chloride as shown in the equation.



step 3 The student tested for the ammonia gas given off and when no more gas was being formed, the solution was allowed to cool to room temperature and was labelled **FA 2**.

You are provided with some of this solution, **FA 2**. By titration with hydrochloric acid, HCl, you are to determine the amount of sodium hydroxide left in the solution. This will enable a percentage by mass of the ammonium chloride in the mixture to be determined, assuming that the student's method is valid.

The following reagents are provided.

**FA 2**, the solution prepared by the student  
**FA 3**, 0.100 mol dm<sup>-3</sup> hydrochloric acid, HCl  
 methyl orange indicator

**(a) Method**

**Before starting any practical work, read through all the instructions and prepare a suitable table for your results in the space provided.**

1. Fill a burette with the hydrochloric acid, **FA 3**.
2. Use a pipette to transfer 25.0 cm<sup>3</sup> of **FA 2** into a conical flask.
3. Add 5 drops of methyl orange indicator to the conical flask.
4. Titrate the solution in the flask with **FA 3**.
5. Repeat the titration as many times as you feel are necessary in order to obtain consistent results.
6. Record your results in a suitable form in the space below.
7. Clearly indicate the results you will use to calculate the average titre.

I	
II	
III	
IV	
V	
VI	

[6]

(b) From your titration results calculate the average titre.

25.0 cm<sup>3</sup> of **FA 2** required ..... cm<sup>3</sup> of **FA 3**.  
[1]

(c) You must show your working and use appropriate significant figures in the answer for each step of your calculations.

(i) Calculate the amount, in mol, of hydrochloric acid present in the average titre of **FA 3**.

..... mol

(ii) Calculate the amount, in mol, of sodium hydroxide present in 250.0 cm<sup>3</sup> of **FA 2**.

..... mol

(iii) Calculate the amount, in mol, of ammonium chloride present in the mixture.

..... mol

(iv) Calculate the percentage by mass of ammonium chloride present in the mixture.

I	
II	
III	
IV	
V	

..... %  
[5]

- (d) In the titration you have carried out, volumes were measured using both a burette and a pipette.

Using one of your accurate titration results, determine whether the percentage error is greater or less for this titre value than it is for the volume measured using the pipette.

You should assume that the  $25\text{ cm}^3$  pipette has an uncertainty of  $\pm 0.06\text{ cm}^3$ .

**Show your working.**

[2]

- (e) A second student claimed that the method used by the first student could be improved so that the value obtained for the percentage by mass was more accurate. In the modified method the analysis was carried out as follows.

step 1 1.40 g of the mixture was dissolved in exactly  $250.0\text{ cm}^3$  of  $0.200\text{ mol dm}^{-3}$  sodium hydroxide, NaOH.

step 2 Using a pipette,  $25.0\text{ cm}^3$  of this solution was transferred to a conical flask and heated.

step 3 The student tested for the ammonia gas given off and when no more gas was being formed, the solution was allowed to cool to room temperature.

step 4 The cooled solution was then titrated with  $0.100\text{ mol dm}^{-3}$  hydrochloric acid.

step 5 Steps 2 to 4 were repeated until consistent results were obtained.

Discuss whether you agree that this method would give a more accurate value for the percentage by mass of ammonium chloride. Explain how the percentage you calculated in (c)(iv) would compare with the value determined using this second method.

.....  
.....  
.....  
.....[2]

[Total: 16]

Before starting Question 3, half-fill a 250 cm<sup>3</sup> beaker with water and heat with a Bunsen burner to between 60 and 70 °C. You will use this as a hot water bath in this question. Turn off the Bunsen burner.

3 (a) **FA 4** is a solution of a metal nitrate. The cation present in **FA 4** is listed in the Qualitative Analysis Notes. By carrying out a series of tests you will identify this cation.

(i) Carry out the following tests on **FA 4** and record your observations.

test	observations
To approximately 1 cm depth of <b>FA 4</b> in a test-tube add aqueous sodium hydroxide, then	
add excess sodium hydroxide.	
To approximately 1 cm depth of <b>FA 4</b> in a test-tube add aqueous ammonia, then	
add excess aqueous ammonia.	

[2]

(ii) Suggest possible identities for the cation in **FA 4**.

..... [1]

(iii) State a reagent that would help you to confirm the identity of the cation. Carry out a test on **FA 4** with this reagent and record your observations.

reagent .....

observations ..... [2]

(iv) Identify the cation in **FA 4**.

..... [1]

- (b) **FA 5, FA 6 and FA 7** are samples, one of which is a primary alcohol, one is a tertiary alcohol and one is a solution of glucose. By carrying out the following tests you will assign each of the samples.

For the purposes of safety you must assume all three samples are flammable.

**(i) Tests with Tollens' reagent**

Preparation of Tollens' reagent

1. Pour aqueous silver nitrate into a boiling tube to a depth of approximately 2 cm.
2. Add approximately 0.5 cm depth of aqueous sodium hydroxide.
3. Add aqueous ammonia a little at a time with continuous shaking until the brown precipitate just dissolves. Be careful not to add an excess of ammonia.
4. Use this solution to carry out the following tests.

test	observations		
	FA 5	FA 6	FA 7
To 1 cm depth of each compound in a clean, dry test-tube add a few drops of the Tollens' reagent.  If no reaction is seen, warm the tube in the hot water bath.			
<b>When you have made your observations, without delay rinse out thoroughly all the test-tubes and the boiling tube that contain any Tollens' reagent.</b>			

[2]

**(ii) Tests with acidified potassium manganate(VII)**

test	observations		
	FA 5	FA 6	FA 7
To 1 cm depth of each compound in a clean, dry test-tube add approximately 2 cm depth of dilute sulfuric acid. Then add a few drops of aqueous potassium manganate(VII).  If no reaction is seen, warm the tube in the hot water bath.			

[2]



(iii) Assign each of the samples, **FA 5**, **FA 6** and **FA 7**.

The primary alcohol is .....

The tertiary alcohol is .....

The solution of glucose is ..... [1]

(iv) Discuss how the reactions you have observed for glucose compare to those expected for an aldehyde.

.....

.....[1]

[Total: 12]

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