Surname		Other Names	
Centre Number		Candidate Number	
Candidate Signature			

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General Certificate of Education
June 2002
Advanced Level Examination

CHEMISTRY **CHM6/P**
Unit 6(b) Practical Examination

Wednesday 22 May 2002 9.00 am to 11.00 am

In addition to this paper you will require:
the AQA Periodic Table (Reference CHEM/PT/EX);
a calculator.

Time allowed: 2 hours

Instructions

- Use blue or black ink or ball-point pen.
- Fill in the boxes at the top of this page.
- **Carry out all three exercises**.
- Answer **all** questions in the spaces provided. All working must be shown.
- Do all rough work in this book. Cross through any work you do not want marked.
- Take careful note of all the instructions given in each exercise.

Information

- The use of note books and laboratory books is **not** permitted.
- The maximum mark for this paper is 30.
- The skills which are being assessed are
  Skill 1 Planning (8 marks) Exercise 3
  Skill 2 Implementing (8 marks) Exercise 1
  Skill 3 Analysing (8 marks) Exercise 2
  Skill 4 Evaluating (6 marks) Exercise 2
- This paper carries 5 per cent of the total marks for Advanced Level.
- You will be assessed on your ability to use an appropriate form and style of writing, to organise relevant information clearly and coherently, and to use specialist vocabulary, where appropriate.

Advice

- You are advised to spend approximately 40 minutes on each of the three exercises.
- You are advised to carry out Exercise 1 first.

For Examiner's Use			
Number	Mark	Number	Mark
Skill 1			
Skill 2			
Skill 3			
Skill 4			
Total (Column 1) →			
Total (Column 2) →			
TOTAL			
Examiner's Initials			

NO QUESTIONS APPEAR ON THIS PAGE

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This paper consists of the following.

Exercise 1	**Implementing**	Reactions of some metal ions.
Exercise 2	**Analysing and Evaluating**	Determination of the dissociation constant of a weak acid.
Exercise 3	**Planning**	The preparation of aspirin.

An essential part of any practical work is to plan for the most efficient use of the time available. There is enough time to complete the exercises set provided that a sensible approach is used.

You are advised to spend approximately

40 minutes on Exercise 1
40 minutes on Exercise 2
40 minutes on Exercise 3.

TURN OVER FOR EXERCISE 1

Exercise 1 Reactions of some metal ions

Skill assessed: **Implementing** (8 marks)

Introduction

You are provided with **five** solutions labelled **A**, **B**, **C**, **D** and **E**, respectively. Each solution contains a single type of metal ion. Perform the tests described below on each solution in turn.

Record in **Table 1** exactly what you **observe**.

You are not required to identify any of the reaction products.
Wear safety glasses at all times.
Assume that all of the solutions are toxic and corrosive.

Procedure

Use a separate sample of each solution in each of the following tests.

1. Reaction with sodium hydroxide
   Place about 10 drops of solution **A** into the test tube labelled "A". Add sodium hydroxide solution, dropwise with shaking, until in excess. Record your observations.
   Do not discard this mixture.
   Repeat the above test with separate solutions of **B**, **C**, **D** and **E** instead of solution **A**.
   Retain each mixture for use in Test 2.

2. Heating the mixtures from **Test 1**
   Half fill a 250 cm^3 beaker with boiling water. Stand the test tubes containing the mixtures from Test 1 in the beaker for about ten minutes. Record any further changes that occur.

   While you are waiting, begin Test 3.

3. Reaction with potassium thiocyanate
   Place about 10 drops of solution **A** in a test tube and add 10 drops of potassium thiocyanate solution with shaking. Allow the mixture to stand for a few minutes and record your observations.
   Repeat the above test with separate solutions of **B**, **C**, **D** and **E** instead of solution **A**.

4. Reaction with potassium hexacyanoferrate(II)
   Place about 10 drops of solution **A** in a test tube and add 10 drops of potassium hexacyanoferrate(II) solution with shaking. Record your observations.
   Repeat the above test with separate solutions of **B**, **C**, **D** and **E** instead of solution **A**.

   You should not attempt to identify the ions present in any of the five samples.

For Examiner's use only			
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Table 1

	Observations with Solution **A**	Observations with Solution **B**	Observations with Solution **C**	Observations with Solution **D**	Observations with Solution **E**
1. Reaction with sodium hydroxide solution					
2. Heating the mixture from Test 1					
3. Reaction with potassium thiocyanate solution					
4. Reaction with potassium hexacyanoferrate(II) solution					

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Exercise 2 Determination of the dissociation constant of a weak acid

Skills assessed: **Analysing** (8 marks) and **Evaluating** (6 marks)

Introduction

The pK_a value of a weak monoprotic acid can be determined by adding enough sodium hydroxide solution to a solution of the acid to neutralise exactly half of the acid. The pK_a value of the acid is equal to the pH of this half-neutralised mixture. Such a mixture can be formed during a titration, when alkali is added from a burette to the acid in a conical flask.

A chemist used a pH curve to determine the pK_a value of an unknown weak monoprotic organic acid. The chemist transferred 25.0 cm^3 of a solution of the acid into a conical flask using a pipette, and measured the pH of the acid solution using a pH meter accurate to one decimal place. A solution of sodium hydroxide of concentration 0.100 mol dm^{-3} was added from a burette in small portions. The pH of the mixture was recorded after each addition of the sodium hydroxide solution. The chemist's results are given in **Table 2** below.

Table 2

Volume of sodium hydroxide solution added/cm^3	pH	Volume of sodium hydroxide solution added/cm^3	pH
0.0	2.0	23.0	5.3
2.0	2.4	23.5	6.0
4.0	2.5	24.0	6.4
8.0	2.6	24.5	11.7
12.0	3.0	25.0	12.0
16.0	3.5	26.0	12.2
20.0	4.6	28.0	12.6
22.0	5.4		

Analysis

1. Use the results given in the table above to plot a graph of pH (on the *y*-axis) against volume of sodium hydroxide solution added (on the *x*-axis). Use the points to draw the pH curve in the way you think is most appropriate.

2. Use your graph from part 1 to determine:

   (a) the volume of sodium hydroxide solution at the end-point of the titration .... cm^3

   (b) the volume of sodium hydroxide solution needed to half-neutralise the acid .... cm^3

   (c) the pH of the half-neutralised mixture. ....

EXERCISE 2 CONTINUES ON PAGE 8

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3. Use the pH of the half-neutralised mixture from part 2 (c) to calculate the value of the acid dissociation constant, K_a, of the weak acid. ($pK_a = -\log_{10}K_a$)

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4. The weak acid is known to be one of the following.

Table 3

Acid	K_a/mol dm^{-3}
Trichloroethanoic acid	2.3×10^{-1}
Dichloroethanoic acid	5.0×10^{-2}
Chloroethanoic acid	1.3×10^{-3}
Methanoic acid	1.6×10^{-4}
Ethanoic acid	1.7×10^{-5}

Use your answer from part 3 and the data in **Table 3** to identify the unknown acid.

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5. Assume that the maximum errors for the apparatus used in this experiment are

pipette	$\pm$ 0.05 cm^3	
burette total error	$\pm$ 0.15 cm^3	(from two readings and an end-point error)
pH meter	$\pm$ 0.1 pH units	

Estimate the percentage maximum error in using each piece of apparatus and, hence, the maximum overall apparatus error. Use the volume of sodium hydroxide at the end-point to estimate the error in using the burette, and the pH of the half-neutralised mixture to estimate the error in using the pH meter.

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Evaluation

1. Calculate the difference between the value of K_a obtained from the graph and the value of K_a for the acid given in **Table 3**. Express this difference as a percentage of the value given in **Table 3**.

   If you could not complete part 3 of the Analysis section, you should assume that the value of K_a determined from the graph is 1.6×10^{-3}. (This is **not** the correct answer.)

   ....

   ....

2. Comment on the magnitude of the difference between the value of K_a obtained from the graph and the value of K_a for the acid given in **Table 3**.

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3. State **two** ways in which the chemist's method of performing the experiment could be improved.

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Exercise 3 The preparation of aspirin

Skill assessed: **Planning** (8 marks)

Write your answer to this exercise in the space provided on pages 11 to 16 of this booklet.

Introduction

Aspirin was the first drug to be produced synthetically. It is a white solid prepared by the reaction between 2-hydroxybenzenecarboxylic acid and ethanoic anhydride.

HOC_6H_4COOH	$(CH_3CO)_2O$	$CH_3COOC_6H_4COOH$
2-hydroxybenzenecarboxylic acid	ethanoic anhydride	aspirin
($M_r = 138$)	($M_r = 102$)	($M_r = 180$)

The other product of the reaction is ethanoic acid.

2-Hydroxybenzenecarboxylic acid is a crystalline solid, and ethanoic anhydride is a corrosive liquid.

This reaction is an example of acylation, and involves the following practical details. Equimolar amounts of 2-hydroxybenzenecarboxylic acid and ethanoic anhydride are used, and a few drops of 85% phosphoric acid are added to catalyse the reaction. The reagents must be mixed with care, as the reaction can initially be violent.

To complete the reaction, the mixture is refluxed for about fifteen minutes. A few cm^3 of water are added to the hot mixture to decompose any unreacted ethanoic anhydride; this causes the mixture to boil. When the reaction has subsided, the reaction mixture is added to about 40 cm^3 of cold water to precipitate the aspirin. The crude product is purified by recrystallisation from water. A typical yield, based on 2-hydroxybenzenecarboxylic acid, is 75%.

Question

Using the information above, describe how you would obtain 5 g of pure crystalline aspirin, starting from 2-hydroxybenzenecarboxylic acid and ethanoic anhydride.

Your answer must include

1. A balanced equation for the reaction taking place and calculations of:
   - (a) the theoretical mass of 2-hydroxybenzenecarboxylic acid needed to form 5 g of aspirin
   - (b) the minimum mass of 2-hydroxybenzenecarboxylic acid needed to form 5 g of aspirin, bearing in mind that you will only obtain 75% of the theoretical yield of product
   - (c) the likely mass of ethanoic anhydride needed in the reaction.

2. A full description of the preparation of aspirin and its purification. Include details of the apparatus you would use.

3. Details of potential hazards and the relevant safety precautions you would use.

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