Surname		Other Names	
Centre Number			Candidate Number									
Candidate Signature												

For Examiner's Use

General Certificate of Education
June 2007
Advanced Level Examination

ASSESSMENT and QUALIFICATIONS ALLIANCE

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

Friday 25 May 2007 9.00 am to 11.00 am

For this paper you must have
- 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.
- Answer 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 to be marked.
- Take careful note of all the instructions given in each exercise.
- The Periodic Table/Data Sheet is provided on pages 3 and 4. Detach this perforated sheet at the start of the examination.

Information
- You must not use note books and laboratory books.
- The maximum mark for this paper is 30.
- The skills which are being assessed are
  Skill 1 Planning (8 marks)
  Skill 2 Implementing (8 marks)
  Skill 3 Analysing (8 marks)
  Skill 4 Evaluating (6 marks)
- 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 about 40 minutes on each of the three exercises.
- You are advised to carry out Exercise 1 first.

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

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This paper consists of the following.

Exercise 1	**Implementing**	Titration of a solution of potassium iodate(V)
Exercise 2	**Analysing and Evaluating**	Determination of the dissociation constant of a weak acid
Exercise 3	**Planning**	Preparation of phenyl benzenecarboxylate

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

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The Periodic Table of the Elements

■ The atomic numbers and approximate relative atomic masses shown in the table are for use in the examination unless stated otherwise in an individual question.

I	II										III	IV	V	VI	VII	0
1.0 **H** Hydrogen 1																4.0 **He** Helium 2
6.9 **Li** Lithium 3	9.0 **Be** Beryllium 4										10.8 **B** Boron 5	12.0 **C** Carbon 6	14.0 **N** Nitrogen 7	16.0 **O** Oxygen 8	19.0 **F** Fluorine 9	20.2 **Ne** Neon 10
23.0 **Na** Sodium 11	24.3 **Mg** Magnesium 12										27.0 **Al** Aluminium 13	28.1 **Si** Silicon 14	31.0 **P** Phosphorus 15	32.1 **S** Sulphur 16	35.5 **Cl** Chlorine 17	39.9 **Ar** Argon 18
39.1 **K** Potassium 19	40.1 **Ca** Calcium 20	45.0 **Sc** Scandium 21	47.9 **Ti** Titanium 22	50.9 **V** Vanadium 23	52.0 **Cr** Chromium 24	54.9 **Mn** Manganese 25	55.8 **Fe** Iron 26	58.9 **Co** Cobalt 27	58.7 **Ni** Nickel 28	63.5 **Cu** Copper 29	65.4 **Zn** Zinc 30	69.7 **Ga** Gallium 31	72.6 **Ge** Germanium 32	74.9 **As** Arsenic 33	79.0 **Se** Selenium 34	79.9 **Br** Bromine 35	83.8 **Kr** Krypton 36
85.5 **Rb** Rubidium 37	87.6 **Sr** Strontium 38	88.9 **Y** Yttrium 39	91.2 **Zr** Zirconium 40	92.9 **Nb** Niobium 41	95.9 **Mo** Molybdenum 42	98.9 **Tc** Technetium 43	101.1 **Ru** Ruthenium 44	102.9 **Rh** Rhodium 45	106.4 **Pd** Palladium 46	107.9 **Ag** Silver 47	112.4 **Cd** Cadmium 48	114.8 **In** Indium 49	118.7 **Sn** Tin 50	121.8 **Sb** Antimony 51	127.6 **Te** Tellurium 52	126.9 **I** Iodine 53	131.3 **Xe** Xenon 54
132.9 **Cs** Caesium 55	137.3 **Ba** Barium 56	138.9 **La** Lanthanum 57 *	178.5 **Hf** Hafnium 72	180.9 **Ta** Tantalum 73	183.9 **W** Tungsten 74	186.2 **Re** Rhenium 75	190.2 **Os** Osmium 76	192.2 **Ir** Iridium 77	195.1 **Pt** Platinum 78	197.0 **Au** Gold 79	200.6 **Hg** Mercury 80	204.4 **Tl** Thallium 81	207.2 **Pb** Lead 82	209.0 **Bi** Bismuth 83	210.0 **Po** Polonium 84	210.0 **At** Astatine 85	222.0 **Rn** Radon 86
223.0 **Fr** Francium 87	226.0 **Ra** Radium 88	227 **Ac** Actinium 89 †															

Key

relative atomic mass — 6.9 **Li** Lithium

atomic number — 3

* **58 – 71** Lanthanides	140.1 **Ce** Cerium 58	140.9 **Pr** Praseodymium 59	144.2 **Nd** Neodymium 60	144.9 **Pm** Promethium 61	150.4 **Sm** Samarium 62	152.0 **Eu** Europium 63	157.3 **Gd** Gadolinium 64	158.9 **Tb** Terbium 65	162.5 **Dy** Dysprosium 66	164.9 **Ho** Holmium 67	167.3 **Er** Erbium 68	168.9 **Tm** Thulium 69	173.0 **Yb** Ytterbium 70	175.0 **Lu** Lutetium 71
† **90 – 103** Actinides	232.0 **Th** Thorium 90	231.0 **Pa** Protactinium 91	238.0 **U** Uranium 92	237.0 **Np** Neptunium 93	239.1 **Pu** Plutonium 94	243.1 **Am** Americium 95	247.1 **Cm** Curium 96	247.1 **Bk** Berkelium 97	252.1 **Cf** Californium 98	(252) **Es** Einsteinium 99	(257) **Fm** Fermium 100	(258) **Md** Mendelevium 101	(259) **No** Nobelium 102	(260) **Lr** Lawrencium 103

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Gas constant $R = 8.31\ \text{J K}^{-1}\ \text{mol}^{-1}$

Table 1
Proton n.m.r chemical shift data

Type of proton	**δ/ppm**
RCH_3	0.7–1.2
R_2CH_2	1.2–1.4
R_3CH	1.4–1.6
$RCOCH_3$	2.1–2.6
$ROCH_3$	3.1–3.9
$RCOOCH_3$	3.7–4.1
ROH	0.5–5.0

Table 2
Infra-red absorption data

Bond	**Wavenumber/cm^{-1}**
C—H	2850–3300
C—C	750–1100
C═C	1620–1680
C═O	1680–1750
C—O	1000–1300
O—H (alcohols)	3230–3550
O—H (acids)	2500–3000

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Exercise 1 Titration of a solution of potassium iodate(V)

Skill assessed **Implementing** (8 marks)

Introduction

You are provided with an aqueous solution of potassium iodate(V). You will add to it an excess of potassium iodide and dilute sulphuric acid and then titrate the iodine formed with a solution of sodium thiosulphate. Sodium thiosulphate reacts with the brown solution of iodine and decolourises it.

Wear eye protection at all times.
Assume that all reagents are toxic and corrosive.

Procedure

1 Rinse the burette with the sodium thiosulphate solution provided. Set up the burette and, using a funnel, fill it with the sodium thiosulphate solution. Record the initial burette reading in the table below.

2 Rinse the pipette with the potassium iodate(V) solution provided. Using this pipette and a pipette filler, transfer 25.0 cm^3 of the potassium iodate(V) solution to a 250 cm^3 conical flask.

3 Using the measuring cylinder, transfer 10 cm^3 of dilute sulphuric acid to the conical flask.

4 Add one of the samples of solid potassium iodide provided to the conical flask and swirl the mixture until the solid dissolves. Iodine is formed which makes the solution appear brown in colour.

5 Add the sodium thiosulphate solution from the burette until the mixture in the conical flask becomes pale yellow in colour. At this point add approximately 2 cm^3 of starch solution to the conical flask. Starch reacts with the iodine to form a dark blue solution. Continue titrating until the blue colour just disappears. Record the final burette reading in the table below.
(NB: the blue colour may return after a few minutes. You should ignore this.)

6 Rinse the conical flask thoroughly with water and repeat the titration until you obtain **two** titres which are within 0.10 cm^3 of each other. (You should do no more than five titrations.)
Have one of your final burette readings checked by your supervisor.

7 Calculate and record the average titre.

Results

Final burette reading/cm^3					
Initial burette reading/cm^3					
Volume of sodium thiosulphate used/cm^3					
Tick the titres to be used in calculating the average titre					

Average titre = …… cm^3

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M		C		P	
T		A			

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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 using the pH curve obtained when the acid is titrated against sodium hydroxide. The pH of the solution formed when exactly half of the acid has been neutralised is equal to the pK_a value of the acid.

A chemist used a pH curve to determine a pK_a value of an unknown weak monoprotic 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 which can be read to one decimal place. A solution of sodium hydroxide of concentration of 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 3** below.

Table 3

Volume of sodium hydroxide solution added / cm^3	pH	Volume of sodium hydroxide solution added / cm^3	pH
0.0	2.9	21.5	5.0
2.0	3.4	22.0	5.4
4.0	3.6	22.5	11.7
8.0	3.8	23.0	12.0
12.0	4.0	24.0	12.2
16.0	4.3	25.0	12.3
20.0	4.2	28.0	12.4
21.0	4.8	30.0	12.4

Analysis **Full marks can only be scored in calculations if you show all your working**.

1 Use the results given in the table above to plot a graph of pH (*y*-axis) against volume of sodium hydroxide solution added. Use the points to draw the pH curve.

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 neutralise half of the acid …… cm^3

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

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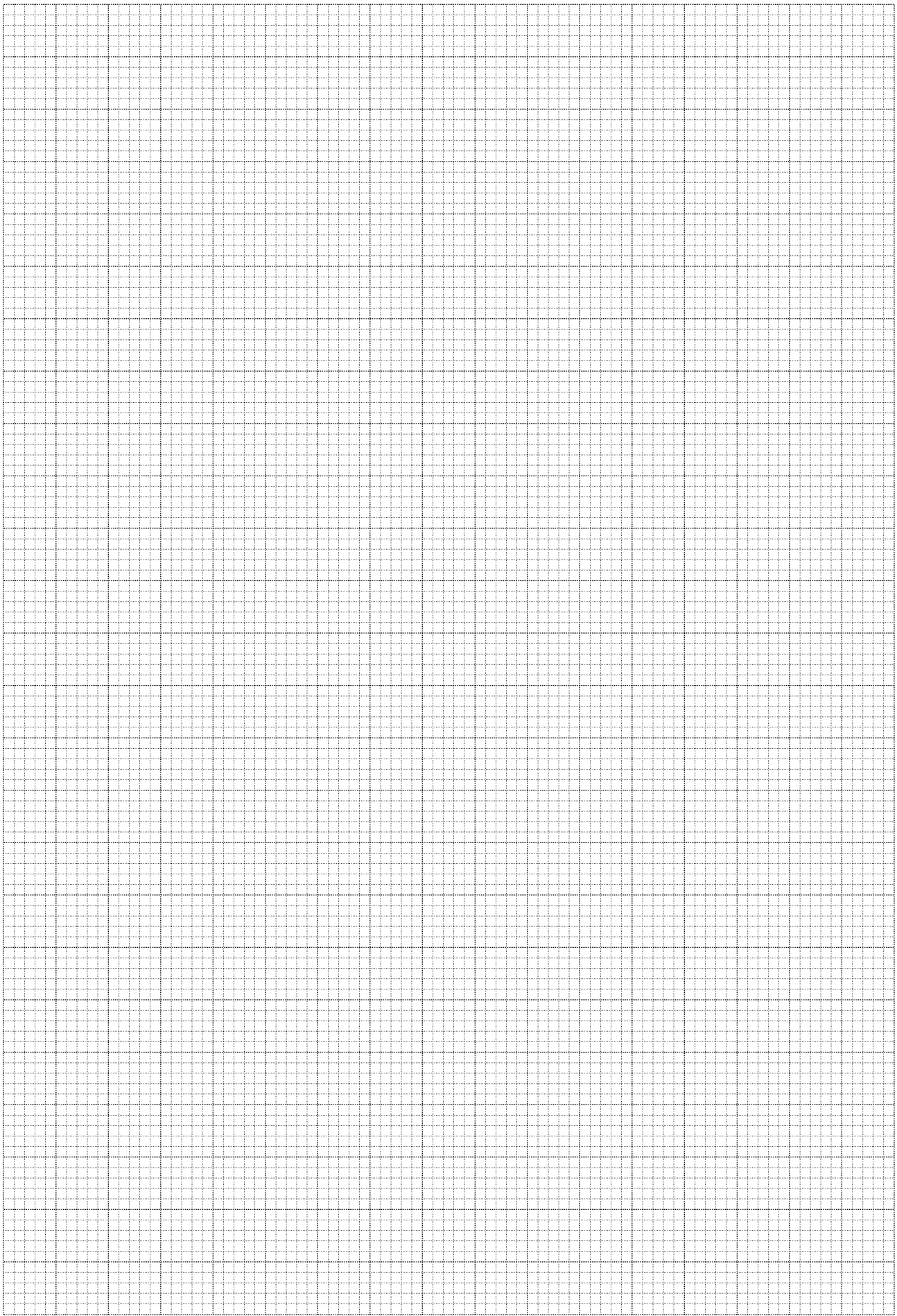

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3 Use the pH of the half-neutralised mixture from part 2(c) to calculate the value of the acid dissociation constant, K_a, of the weak acid.

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

Table 4

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 4** to identify the unknown acid.

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5 For the pipette and the burette, the maximum total errors are shown below. These errors take into account multiple measurements.

pipette	± 0.05 cm^3
burette total error	± 0.15 cm^3

Estimate the maximum percentage error in using these pieces of apparatus and, hence, estimate their combined error.
You should use the volume of sodium hydroxide at the end-point to estimate the percentage error in using the burette.

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Evaluation **Full marks can only be scored in calculations if you show all your working.**

1 Calculate the difference between the K_a value obtained from the graph and the K_a value of the acid you identified in **Table 4** as the unknown acid. Express this difference as a percentage of the value given in **Table 4**.

(If you could not complete part 2 of the Analysis section, you should assume that the K_a value determined from the graph is 1.9×10^{-4} mol dm^{-3}. This is not the correct value.)

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2 The overall percentage apparatus error for this experiment, including the errors in using the pH meter, was found to be 25 %. Comment on the magnitude of the difference between the K_a value obtained from the graph and the K_a value of the acid you identified in **Table 4** as the unknown acid.

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3 State **two** ways in which the accuracy of the pH readings could be improved.

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Exercise 3 Preparation of phenyl benzenecarboxylate

Skill assessed **Planning** (8 marks)

Introduction

Phenyl benzenecarboxylate is a white solid which can be prepared by a reaction between phenol and benzenecarbonyl chloride. Hydrogen chloride is also formed in this reaction.

C_6H_5OH	C_6H_5COCl	$C_6H_5COOC_6H_5$
phenol	benzenecarbonyl chloride	phenyl benzenecarboxylate
(M_r = 94)	(M_r = 141)	(M_r = 198)

The crude phenyl benzenecarboxylate produced in the reaction can be purified by recrystallisation from ethanol. A typical yield, based on benzenecarbonyl chloride, is 70 %. The purity of the recrystallised product can be confirmed by determining its melting point.

Phenol is a corrosive, toxic solid. Benzenecarbonyl chloride is a liquid with an irritating vapour. Ethanol is a flammable liquid.

Using the information above, answer the following questions

1 (a) Write a balanced equation for the reaction taking place.
(b) Calculate the theoretical mass of benzenecarbonyl chloride needed to form 5 g of phenyl benzenecarboxylate.
(c) In this experiment only 70 % of the benzenecarbonyl chloride is converted into phenyl benzenecarboxylate. Calculate the minimum mass of benzenecarbonyl chloride needed to form 5 g of phenyl benzenecarboxylate.
(d) Calculate the minimum mass of phenol needed in the reaction.

2 Write a full description of the purification of the crude phenyl benzenecarboxylate.
You do not have to describe the preparation of phenyl benzenecarboxylate.

3 Write a brief description showing how you would use the melting point of the purified product to confirm its purity.
You do not have to describe how you would obtain the melting point.

4 Give details of the potential hazards of this experiment, and the relevant safety precautions you would take.

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END OF QUESTIONS

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