Surname		Other Names	
Centre Number			Candidate Number									
Candidate Signature												

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General Certificate of Education
June 2003
Advanced Level Examination

ASSESSMENT and QUALIFICATIONS ALLIANCE

CHEMISTRY CHM6/P
Unit 6(b) Practical Examination

Wednesday 21 May 2003 9.00 am to 11.00 am

In addition to this paper you will require:
- a calculator.

Time allowed: 2 hours

The Instructions to Supervisors are attached

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.
- The Periodic Table/Data Sheet is provided on pages 3 and 4. Detach this perforated sheet at the start of the examination.

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)
  Skill 2 Implementing (8 marks)
  Skill 3 Analysing (8 marks)
  Skill 4 Evaluating (6 marks)
- 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			

This paper consists of the following.

Exercise 1	**Implementing**	The titration of a solution of potassium iodate(V).
Exercise 2	**Analysing and Evaluating**	The analysis of a mixture of iron(II) and iron(III) ions.
Exercise 3	**Planning**	The preparation of (4-methylphenyl)phenylmethanone.

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.

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

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
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

* **58 – 71** Lanthanides

† **90 – 103** Actinides

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

Exercise 1 The 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). Determine the exact concentration of this solution by

(a) adding an excess of potassium iodide and dilute sulphuric acid and
(b) titrating the liberated iodine against a 0.100 mol dm^{-3} solution of sodium thiosulphate solution.

Wear safety glasses at all times.
Assume that all solutions are toxic and corrosive.

Procedure

1. Rinse a burette with the sodium thiosulphate solution provided. Set up the burette and, using a funnel, fill it with the sodium thiosulphate solution provided. Record the initial burette reading.
2. Rinse a 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 a measuring cylinder, add approximately 10 cm^3 of dilute sulphuric acid to the conical flask.
4. Add one of the samples of potassium iodide provided to the conical flask and swirl the mixture to dissolve the crystals.
5. Add the sodium thiosulphate solution from the burette until the mixture in the conical flask is pale yellow in colour. Add approximately 2 cm^3 of starch solution to the conical flask. Continue titrating until the blue colour just disappears. Record the final burette reading in the table below.
(NB: the blue colour may return on standing. You should ignore this.)
6. Rinse the conical flask 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. Tick the titres you will be using in calculating the mean titre. Calculate and record the mean titre.

Final burette reading / cm^3					
Initial burette reading / cm^3					
Volume of thiosulphate solution used / cm^3					
Tick the titres you used in calculating the mean					

Mean titre = ... cm^3

For Examiner's use only					
M		C		P	
T		A			

Turn over ▶

Exercise 2 The analysis of a mixture of iron(II) and iron(III) ions.

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

The concentration of iron(II) ions can be determined by titration using a solution of potassium dichromate(VI) in the presence of dilute sulphuric acid. Half-equations for the redox reactions occurring are given below.

$$Fe^{2+} \longrightarrow Fe^{3+} + e^-$$

$$Cr_2O_7^{2-} + 14H^+ + 6e^- \longrightarrow 2Cr^{3+} + 7H_2O$$

A mixture of iron(II) and iron(III) ions in solution was analysed by the following procedure.

A 25.0 cm^3 portion of the iron(II) / iron(III) solution was measured by pipette. This solution was acidified with dilute sulphuric acid and titrated with a 0.0170 mol dm^{-3} solution of potassium dichromate(VI). In this titration the dichromate(VI) ions reacted with the iron(II) ions present.

After the titration was complete, all of the iron in the portion was present as iron(III). An excess of zinc was then added to reduce all the iron(III) in the mixture to iron(II).

$$Zn + 2Fe^{3+} \longrightarrow Zn^{2+} + 2Fe^{2+}$$

After filtering to remove the unreacted zinc, the resulting solution of iron(II) was acidified with dilute sulphuric acid and again titrated with the 0.0170 mol dm^{-3} solution of potassium dichromate(VI). In this second titration, the dichromate(VI) ions reacted with all of the iron ions present in the 25.0 cm^3 portion of the original mixture.

The experiment was repeated three times and the results for all four experiments are shown in **Tables 1** and **2**.

Table 1

Experiment	1	2	3	4
Titre for original portion / cm^3	17.50	17.45	17.75	17.55

Table 2

Experiment	1	2	3	4
Titre after reaction with zinc / cm^3	23.70	23.65	23.75	23.70

Analysis

1. Write an equation for the reaction between iron(II) ions and dichromate(VI) ions in the presence of dilute sulphuric acid.

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2. Identify all the concordant results in **Table 1** and use these to determine a mean titre for the original solution.

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3. Use your answer from part 2 above to calculate the number of moles of iron(II) ions in 25.0 cm^3 of the original solution.

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4. (a) Identify all the concordant results in **Table 2** and use these to determine a mean titre for the solution after reaction with zinc.

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(b) Calculate the total number of moles of iron(II) and iron(III) ions present in 25.0 cm^3 of the original solution.

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5. Use your answers from part 3 and part 4(b) to calculate the percentage of iron present as iron(II) ions in the original solution.

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6. Assume that the maximum errors for the apparatus used in this experiment were

pipette	± 0.05 cm^3	
burette total error	± 0.15 cm^3	(from two readings and an end-point error)

Calculate the maximum percentage error in using each piece of apparatus, and hence the maximum overall apparatus error. Use the mean titre for the original mixture to calculate the error in using the burette.

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Turn over ▶

Evaluation

1. Comment on the consistency of the titration results given in **Tables 1** and **2**.

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2. According to the supplier, the original solution contained iron(II) ions as 78.0 % of the total iron present. Calculate the difference between the value calculated in part 5 of the **Analysis** section and the supplier's value. Express this difference as a percentage of the supplier's value. (If you could not complete the calculation in part 5 of the **Analysis** section, you should assume a value of 82.2 %. This is **not** the correct value).

   Difference .....

   Percentage .....

3. Explain why it is necessary to filter off unreacted zinc.

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4. Explain how errors could result from filtration process.

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6

Exercise 3 The preparation of (4-methylphenyl)phenylmethanone.

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

Write your answer to this exercise in the space provided on pages 10 to 14 of this booklet.

Introduction

(4-Methylphenyl)phenylmethanone is a pale yellow solid which can be prepared from methylbenzene and benzenecarbonyl chloride by a Friedel–Crafts reaction. Aluminium chloride is used as a catalyst.

$C_6H_5CH_3$	C_6H_5COCl	$C_6H_5COC_6H_4CH_3$	$AlCl_3$
methylbenzene ($M_r = 92.0$)	benzenecarbonyl chloride ($M_r = 140.5$)	(4-methylphenyl)phenylmethanone ($M_r = 196.0$)	aluminium chloride ($M_r = 133.5$)

This reaction is an example of acylation; the other product of the reaction is hydrogen chloride. Equimolar amounts of benzenecarbonyl chloride and aluminium chloride are used together with a large excess of methylbenzene.

A typical yield, based on benzenecarbonyl chloride, is 65%.

The crude solid product can be purified by recrystallisation from methylbenzene.

Methylbenzene is a toxic flammable liquid.

Questions

1. (a) Write an equation for the reaction taking place.

   (b) Calculate the theoretical mass of benzenecarbonyl chloride needed to form 5 g of (4-methylphenyl)phenylmethanone.

   (c) Calculate the mass of benzenecarbonyl chloride needed to form 5 g of (4-methylphenyl)phenylmethanone, bearing in mind that you will only obtain 65% of the theoretical yield of the product.

   (d) Calculate the mass of aluminium chloride needed in the reaction.

2. Give a full description of the procedure for purifying the crude product by recrystallisation, including details of the apparatus you would use.
   You are **not** required to describe the preparation of the crude product.

3. For the recrystallisation, give details of potential hazards and relevant safety precautions.

8

Turn over ▶

General Certificate of Education
June 2003
Advanced Level Examination

ASSESSMENT and QUALIFICATIONS ALLIANCE

CHEMISTRY PRACTICAL EXAMINATION **CHM6/PTN**
Instructions to Supervisors

CONFIDENTIAL

1 The practical examination will be held on Wednesday 21 May 2003, 9.00 am to 11.00 am.

Centres are permitted to run more than one session for the Practical Examination provided that the following conditions are met:

- all candidates to be examined must be present in the centre by 9.30 am at the latest;
- all candidates who are waiting to be examined must be supervised until their session begins;
- candidates who are released at the end of their session must have no contact with any candidate yet to be examined.

2 **The strictest possible precautions are to be taken to prevent these exercises becoming known to the candidates in advance, either directly or indirectly. AQA emphasises the need to preserve the absolute fairness and integrity of this examination. This copy of Instructions to Supervisors is to be kept at the centre under secure conditions when not in use; it is not to be removed from the centre.**

3 A combined question paper/answer book will be supplied. If an answer book is badly damaged, e.g. by spillage, a candidate may be given a fresh book, **but both books must be sent to the Examiner**, together with a statement of the reasons for issuing a duplicate answer book. The damaged book must be sealed in a polythene bag.

The Periodic Table/Data Sheet will be provided as a perforated sheet on pages 3 and 4 of the question paper/answer book. Candidates will be instructed to detach this sheet at the start of the examination.

4 The use of books and laboratory notebooks is **not** permitted.

5 The attention of candidates must be drawn to the requirement that all rough work must be done in the answer book. **Extra paper is not to be supplied for this purpose**. Candidates' attention should also particularly be drawn to the instructions contained in the question paper.

6 As far as possible, apparatus and special materials should not be put away until the end of the examination period; an Inspector who arrives late will thus be able to see the preparations that have been made.

7 If a candidate fails with the material allotted to him/her and asks to be allowed a second opportunity, he/she may be allowed it at the discretion of the Supervisor. **Under no circumstances may materials from other sources be used**. Supervisors should bear this in mind as well as the availability of apparatus and the amount of time remaining when exercising this discretion. No extra time is to be allowed to such a candidate and he/she must hand in his/her script at the same time as other candidates at the centre. A full report, in writing, of any such incident must be sent to the Examiner together with the scripts. **Supervisors must not allow extra time to candidates** unless specific permission is given by AQA. Any circumstance which leads to a shortage of time should be reported to the Examiner.

8 A Supervisor must not give any advice to candidates about the way they are conducting experiments unless it is to prevent personal injury to the candidates or damage to apparatus. If any such incident occurs, the Supervisor should report details, in writing, to the Examiner when scripts are sent. Unless specific mention to the contrary is made in the instructions, Supervisors must not give any advice or information to candidates, whether it is asked for or not.

APPARATUS AND MATERIALS

Exercise 1

This exercise involves the reaction of a solution of potassium iodate(V) with potassium iodide, and the titration of the liberated iodine with standard sodium thiosulphate solution.

Materials

Each candidate will require two volumetric solutions:

1 (a) A standard **sodium thiosulphate** solution of concentration between 0.090 and 0.110 mol dm^{-3}.

This solution may be made up in the centre or purchased from a reputable manufacturer at the discretion of the centre. Wherever possible, the centre should prepare one bulk batch only of this solution. It is essential that the concentration of this solution should be in the range specified. It must be stressed that the accuracy of this solution is the responsibility of the centre **alone**.

Each candidate will require 200 cm^3 of this solution, in a closed container labelled sodium thiosulphate.

(b) A solution of **potassium iodate(V)**, of concentration between 0.0150 and 0.0170 mol dm^{-3}.

This solution may be made up in the centre. Wherever possible, the centre should prepare one bulk batch only of this solution. It is essential that the concentration of this solution should be within the range specified. It must be stressed that the accuracy of this solution is the responsibility of the centre **alone**.

Each candidate will require 150 cm^3 of this solution, in a closed container labelled potassium iodate(V).

2 Each candidate will require 100 cm^3 of **sulphuric acid**, of concentration between 0.9 mol dm^{-3} and 1.1 mol dm^{-3}. This solution may be made up in the centre. Wherever possible, the centre should prepare one bulk batch only of this solution. It is essential that the concentration of this solution should be within the range specified. It must be stressed that the accuracy of this solution is the responsibility of the centre **alone**.

3 Each candidate will require five separate samples of **potassium iodide**. Each sample must be labelled potassium iodide and weigh at least 0.50 g.

4 Each candidate will require access to a solution of **starch** as indicator. It is not essential to provide individual supplies of the indicator.

5 Reagents of good analytical quality should be used in preparing the solutions, and they should be carefully stored in bottles fitted with air-tight stoppers. Great care must be taken in the storage and dispensing of each solution to ensure that its concentration is unaltered. Wherever possible, centres are advised to check that the reagents used do work.

6 Supervisors are required in every instance to carry out the volumetric exercise and to report the results to the Examiner on the form provided on page 5 of this booklet. A Supervisor result is required for **each** group of candidates. The Supervisor results must be entered with the list of candidates supervised in the group on the form provided. The accuracy of the candidates' results will be assessed against the Supervisor's results for the titration. Supervisors must **not** carry out the exercise in the presence of the candidates.

Supervisors are also required to keep a sample (not less than 100 cm^3) of each volumetric solution used in a small stoppered bottle. These samples should be kept for a period of four weeks after the examination and should be available to the Examiners if called for.

It is essential that orders for solutions which are not to be made up in the centre should be placed without delay.

Spare supplies of all solutions specified in these instructions must be available.

7 Supervisors are required to assess the manipulative skills of candidates and to complete the grid on page 5 of this booklet. This form must be sent to the Examiner with the scripts.

If a centre needs to conduct the examination in two or more separate sessions, the form on page 5 must be completed and sent to the Examiner with each group of scripts. This form may be photocopied if centres have large numbers of candidates.

Apparatus

The apparatus specified below represents the minimum requirement. Candidates will be advised to carry out Exercise 1 first.

Each candidate will require:

one 50 cm^3 burette and stand
one funnel
one 25 cm^3 pipette
one pipette filler
one measuring cylinder (10 cm^3 or 25 cm^3 or 50 cm^3)
two or more 250 cm^3 conical flasks
one dropping pipette or one measuring cylinder (5 cm^3 or 10 cm^3) for adding starch solution
one wash bottle
a plentiful supply of purified water (either distilled or de-ionised).

CHEMISTRY PRACTICAL EXAMINATION CHM6/P June 2003

Centre Number _____

Supervisor _____ Date _____

Supervisor's average titre _____ cm^3

Name of Candidate	1 Pipette empties under gravity	2 Transfers from pipette without spillage	3 Touches surface with pipette	4 Uses thio in burette & iodate(V) in pipette	5 Removes funnel before titrating	6 Dropwise addition near end-point	7 Swirls mixture	8 Reads burette correctly	9 Does not need additional reagent		TOTAL (9)

Notes for the assessment of Manipulative Skills listed 1–9 above.

1–3 The Supervisor should observe the candidate in the use of the pipette at an appropriate time during the titration. The candidate scores the mark if the correct technique is used **once**.

4 The mixture in the flask at the beginning of the titration should be red-brown.

5–8 The Supervisor should observe the candidate in the use of the burette at an appropriate time during the titration. The candidate scores the mark if the correct technique is used **once**.

9 The candidate loses this mark if an extra supply of sodium thiosulphate, potassium iodide or potassium iodate(V) is needed.

This sheet may be photocopied