Surname		Other Names	
Centre Number			Candidate Number	
Candidate Signature			

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General Certificate of Education
June 2004
Advanced Level Examination

ASSESSMENT and QUALIFICATIONS ALLIANCE

CHEMISTRY CHM6/P
Unit 6(b) Practical Examination

Friday 28 May 2004 9.00 am to 11.00 am

In addition to this paper you will require:
- 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.
- 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 iron(II) sulphate.
Exercise 2	**Analysing and Evaluating**	The reaction between hydrogen peroxide and iodide ions.
Exercise 3	**Planning**	Determining the dissociation constant, K_a, of a weak acid.

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 iron(II) sulphate.

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

Introduction

You are provided with an aqueous solution of iron(II) sulphate of approximate concentration $0.1\,mol\,dm^{-3}$. Determine the exact concentration by titrating this solution, after acidification, against potassium manganate(VII) solution of concentration $0.0200\,mol\,dm^{-3}$.

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

Procedure

1. Rinse the burette with the potassium manganate(VII) solution provided. Set up the burette and, using a funnel, fill it with the potassium manganate(VII) solution. Record the initial burette reading.

2. Rinse a pipette with the iron(II) sulphate solution provided. Using this pipette and a pipette filler, transfer $25.0\,cm^3$ of the iron(II) sulphate solution to a $250\,cm^3$ conical flask.

3. Using a measuring cylinder, transfer approximately $20\,cm^3$ of dilute sulphuric acid to the conical flask.

4. Add the potassium manganate(VII) solution from the burette into the conical flask containing the iron(II) sulphate solution until a permanent pink colour is seen. Record your burette readings in the table below.

5. 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.

6. Tick the titres you will use to calculate the average. Calculate and record the average titre.

Final burette reading/cm^3					
Initial burette reading/cm^3					
Volume of potassium manganate(VII) solution used/cm^3					
Tick the titres you used in calculating the average					

Average titre = ... cm^3

8

Exercise 2 The reaction between hydrogen peroxide and iodide ions.

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

Introduction

In aqueous solution, and in the presence of dilute sulphuric acid, hydrogen peroxide reacts with iodide ions to form iodine according to the following equation.

$$H_2O_2(aq) + 2H^+(aq) + 2I^-(aq) \longrightarrow I_2(aq) + 2H_2O(l)$$

Sodium thiosulphate solution and starch solution are added to an acidified mixture of hydrogen peroxide and potassium iodide. The **initial rate** of this reaction is investigated by measuring the time taken to liberate sufficient iodine to react with the thiosulphate ions present, and then produce a blue colour with starch solution.

A series of experiments is carried out in which the concentration of iodide ions is varied, while keeping the concentrations of all of the other reagents the same. In each experiment the time taken for the reaction mixture to turn blue is recorded. The results obtained are used to determine the order of reaction with respect to iodide ions.

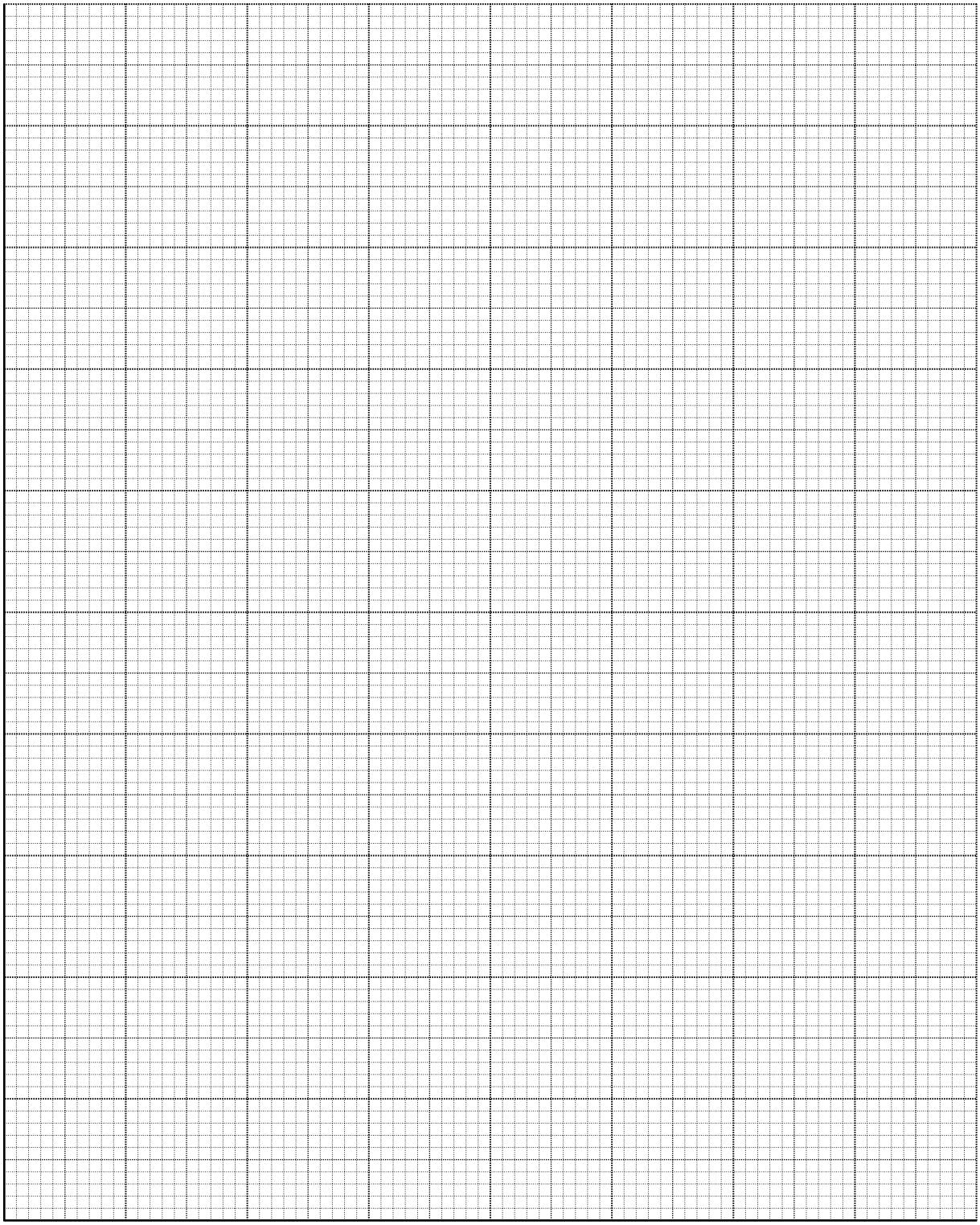
The rate of the reaction can be represented as (1/time), and the concentration of iodide ions can be represented by the volume of potassium iodide solution used. A graph of $\log_{10}$(1/time) on the y axis against $\log_{10}$(volume of KI) is a straight line with gradient equal to the order of the reaction with respect to iodide ions.

A set of results is given in the table below. The volumes of potassium iodide solution were measured using a measuring cylinder. The times taken for the mixture to turn blue were recorded on a stopclock graduated in seconds.

Expt	Volume of potassium iodide solution/cm^3	$\log_{10}$(volume of KI)	time/s	$\log_{10}$(1/time)
1	5	0.70	68	–1.83
2	8	0.90	45	–1.65
3	10	1.00	36	–1.56
4	15	1.18	25	–1.40
5	20	1.30	22	–1.34
6	25	1.40	16	–1.20

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

1. Use the results given in the table to plot a graph of $\log_{10}$(1/time) on the y axis against $\log_{10}$ (volume of KI). Draw a straight line of best fit on the graph, ignoring any anomalous points.



Turn over ▶

2. Determine the gradient of the graph.

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

measuring cylinder	$\pm$ 0.5 cm^3
clock	$\pm$ 1 second

Calculate the maximum percentage error in using each piece of apparatus in Experiment 3. Hence, calculate the maximum overall percentage error in Experiment 3.

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Evaluation

1. Consider your graph and comment on the results obtained from the investigation. Is your line of best fit good enough for you to deduce an order with confidence? Identify any anomalous results.

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2. State **two** ways in which the method of performing this experiment could be improved, other than by repeating the experiment. In each case, explain why the accuracy of the experiment would be improved.

Improvement 1

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Explanation

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Improvement 2

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Explanation

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6

Exercise 3 Determining the dissociation constant, K_a, of a weak acid.

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

Introduction

A suitable method for determining the dissociation constant, K_a, of a weak acid involves measuring the pH values of the solution obtained as aqueous alkali is added to a known volume of the aqueous acid, until the alkali is present in considerable excess.

A pH curve can be plotted and used to determine the volume of alkali needed at the equivalence point (end-point). The pH when half of this volume of alkali has been added is equal to the pK_a of the acid.

Question

Describe how you could determine the dissociation constant, K_a, of the acid.

You are provided with a crystalline sample of the weak monoprotic acid HA and a 0.100 mol dm^{-3} solution of sodium hydroxide. The M_r of the acid is 150 and the acid is soluble in water.

Your answer must include

1. Calculations to determine the scale you would choose for the experiment and the mass of acid you would need to prepare your standard solution.

2. A description of the experiment you would perform and details of the apparatus you would use. You do **not** need to describe how you would prepare the acid solution.

3. A sketch of the pH curve and a clear explanation of how you would use the results of your experiment to determine the dissociation constant, K_a, of the acid.

4. Details of potential hazards and the relevant safety precautions you would take.

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General Certificate of Education
June 2004
Advanced Level Examination

CHEMISTRY PRACTICAL EXAMINATION **CHM6/P/TN**
Instructions to Supervisors

CONFIDENTIAL

1 The practical examination will be held on Friday 28 May 2004, 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

In this exercise candidates are instructed to titrate an acidified solution of iron(II) sulphate against a potassium manganate(VII) solution of known concentration. However, as iron(II) sulphate solution slowly oxidises in air, candidates are to be given a solution of ammonium iron(II) sulphate, labelled as iron(II) sulphate, since this is less susceptible to oxidation.

Materials

Each candidate will require two volumetric solutions:

1 (a) A standard **potassium manganate(VII)** solution of concentration between 0.0190 and 0.0210 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 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 **potassium manganate(VII)**.

(b) A solution of **ammonium iron(II) sulphate** of concentration between 0.090 and 0.110 mol dm^{-3}.

This solution may be made up in the centre. For maximum resistance to oxidation the centre should make up the solution in sulphuric acid, of approximate concentration 1 mol dm^{-3}. 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 **iron(II) sulphate**. It is recommended that the centre includes a warning that the solution contains 1 mol dm^{-3} sulphuric acid.

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 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.

4 Supervisors are required in every instance to carry out the volumetric exercise and to report the result 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 result 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 result for the titration. Supervisors must **not** carry out the exercise in the presence of the candidates.

Supervisors are also asked 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.

5 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, a copy of 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, small enough to fit into the burette
one 25 cm^3 pipette
one pipette filler
one measuring cylinder (25 cm^3 or 50 cm^3)
two or more 250 cm^3 conical flasks
one wash bottle
a plentiful supply of purified water (either distilled or de-ionised)
suitable eye protection.

CHEMISTRY PRACTICAL EXAMINATION CHM6/P June 2004

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 $KMnO_4$ in burette	5 Removes funnel before titrating	6 Dropwise addition near end-point	7 Swirls mixture	8 Reads burette correctly	9 Doesn't need additional reagent	10 Works safely	TOTAL (10)

Notes for the Assessment of Manipulative Skills listed 1–10 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 pale green/colourless.

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 potassium manganate(VII) or ammonium iron(II) sulphate is needed, when the reason for the additional sample is clearly the fault of the candidate e.g. spillage, careless measurement of volumes or using the wrong solution.

10 The candidate loses this mark if he/she does not wear eye protection, or does anything which the supervisor regards as potentially hazardous.

This sheet may be photocopied

THERE ARE NO INSTRUCTIONS PRINTED ON THIS PAGE

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