

This paper consists of the following.

Exercise 1	Implementing	Titration of a sample of hydrochloric acid
Exercise 2	Analysing and Evaluating	Identification of a Group II carbonate
Exercise 3	Planning	Determination of an enthalpy change of neutralisation

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.

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 Titration of a sample of hydrochloric acid

Skill assessed **Implementing** (8 marks)

Introduction

You are provided with a sample of hydrochloric acid whose concentration is approximately 0.1 mol dm^{-3} . Titrate the hydrochloric acid with a $0.100 \text{ mol dm}^{-3}$ solution of sodium hydroxide.

Wear suitable eye protection.

Procedure

- 1 Rinse the burette with the hydrochloric acid provided. Set up the burette and, using a funnel, fill it with the hydrochloric acid provided. Record the initial burette reading.
- 2 Rinse a pipette with the sodium hydroxide solution provided. Using this pipette, transfer 25.0 cm^3 of the sodium hydroxide solution to a 250 cm^3 conical flask.
- 3 Add 3 or 4 drops of **phenol red** indicator to the conical flask. This indicator changes from **pink** in alkaline solution to **yellow** in acid solution. The end-point has been reached when the solution just turns yellow.
- 4 Add the acid from the burette until the mixture in the conical flask just changes colour. Record your burette reading in the table below.
- 5 Rinse the conical flask 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.
- 6 Calculate and record the mean titre.

Final burette reading/ cm^3					
Initial burette reading/ cm^3					
Volume of hydrochloric acid used/ cm^3					
Tick the titres to be used in calculating the mean					

Mean titre = cm^3

For Examiner's use only					
M		C		P	
T		A			

8

Turn over

Exercise 2 Identification of a Group II carbonate**Skill assessed** **Analysing** (8 marks) **and Evaluating** (6 marks)**Introduction**

A pure compound was known to be the carbonate of a Group II metal. A student was asked to identify this carbonate by titration of a sample of the compound with hydrochloric acid.

The method chosen by the student involved rinsing a weighing bottle with pure water and then adding 1.00 g of the carbonate to the bottle. The contents of the weighing bottle were then transferred to a conical flask. About 20 cm³ of pure water were added to the conical flask.

The student filled a burette with 1.00 mol dm⁻³ hydrochloric acid. The sample was titrated with the acid solution, using methyl orange indicator. The student then repeated the titration using further 1.00 g samples of the carbonate. The following results were obtained.

Titration number	1	2	3	4	5
Final burette reading/cm ³	19.60	19.20	19.35	19.15	25.85
Initial burette reading/cm ³	0.05	0.05	0.05	0.10	6.75
Titre per 1.00 g of carbonate/cm ³					

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

- 1 In this titration, hydrochloric acid and the metal carbonate react in a 2:1 mol ratio. Write an equation for the reaction, representing the metal carbonate as MCO₃

.....

- 2 Use all of the concordant results in the table above to determine the mean titre.

.....

.....

- 3 Use the mean titre to calculate the number of moles of the metal carbonate present in 1.00 g of the sample.

.....

.....

.....

4 Using your result from part 3, determine the relative formula mass, M_r , of the metal carbonate.

.....
.....
.....

5 Use your result from part 4 to show that the Group II metal present in the carbonate is calcium.

.....
.....
.....
.....

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

- balance total error ± 0.01 g
- burette total error ± 0.15 cm³ (from two readings and an end-point error)

Calculate the maximum percentage errors in using the balance and in using the burette in this experiment, and hence the overall maximum percentage error.

.....
.....
.....
.....

8

Turn over 

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

1 Comment on the consistency of the titres. Suggest **one** possible reason for an anomalous result.

.....
.....
.....
.....

2 Calculate the difference between the experimental M_r value determined by the student and the actual M_r value of calcium carbonate. Express this difference as a percentage of the actual M_r value of calcium carbonate.

(If you could not complete the calculation in part 4 of the Analysis section, you should assume that the experimental M_r value is 106. This is not the correct answer.)

Difference

Percentage

3 Comment on the significance of the difference between the actual M_r of calcium carbonate and your calculated value. Assume that this difference is not due to impurities.

.....
.....

4 State **two** ways of improving the student's method of weighing the metal carbonate. In each case explain why the accuracy of the experiment would be improved.

Improvement 1

.....

Explanation

.....

Improvement 2

.....

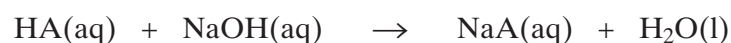
Explanation

.....

6

Exercise 3 Determination of an enthalpy change of neutralisation**Skill assessed** **Planning** (8 marks)**Introduction**

When a monobasic acid, HA, in aqueous solution is neutralised by a solution of sodium hydroxide, the following exothermic reaction occurs.



When equal volumes of a 0.2 mol dm^{-3} solution of HA and a 0.2 mol dm^{-3} solution of sodium hydroxide react together, the temperature rises by about 1°C .

Question

Using the information above, describe how you would measure the enthalpy change of the reaction between the unknown acid, HA, and sodium hydroxide. Assume that you have access to solutions of HA and of sodium hydroxide of the concentrations you choose.

Your answer must include the following

- 1 The volumes and concentrations of the solutions of the unknown acid, HA, and of sodium hydroxide to be used, and your reasons for choosing these concentrations.
- 2 A description of the experiment you would perform. Include details of the apparatus you would use and the precautions you would take to minimise heat loss. A diagram is not essential but may help your answer.
- 3 A clear explanation, including a sketch graph of temperature against time, showing how you would use your results to calculate, in kJ mol^{-1} , the enthalpy change of neutralisation of the unknown acid, HA.
- 4 Details of the potential hazards, and the relevant safety precautions you would take.

.....

.....

.....

.....

.....

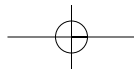
.....

.....

.....

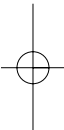
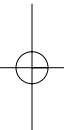
8

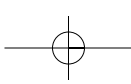
Turn over 



LEAVE
MARGIN
BLANK

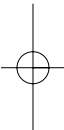
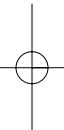
A large rectangular area containing 25 horizontal dotted lines, intended for writing.



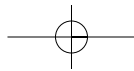


LEAVE
MARGIN
BLANK

A large rectangular area containing 25 horizontal dotted lines, intended for writing.

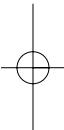
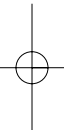


Turn over 



LEAVE
MARGIN
BLANK

A large rectangular area containing 25 horizontal dotted lines, intended for writing.



General Certificate of Education
June 2005
Advanced Subsidiary Examination



CHEMISTRY PRACTICAL EXAMINATION **CHM3/P/TN**
Instructions to Supervisors

CONFIDENTIAL

- 1 The practical examination will be held on Thursday 19 May 2005, 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 AQA. **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 AQA.

- 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 hydrochloric acid with sodium hydroxide solution.

Materials

Each candidate will require two volumetric solutions:

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

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 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 **sodium hydroxide**.

- (b) A solution of **hydrochloric acid** of concentration between 0.090 and $0.100 \text{ mol dm}^{-3}$.

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 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 150 cm^3 of this solution, in a closed container labelled **hydrochloric acid**.

- 2 Each candidate will require access to a solution of **phenol red** as indicator. It is not essential to provide individual supplies of the indicator.
- 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 results for the titration. Supervisors must **not** carry out the exercises in the presence of the candidates.

Supervisors are also asked to keep a sample (at least 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.

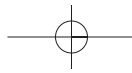
If a centre needs to conduct the examination in two or more separate sessions, the form on page 5 must be completed and sent to the Examiner with each group of scripts. This form may be photocopied if centres have large numbers of candidates.

Apparatus

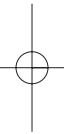
The apparatus specified below represents the minimum requirement. Candidates will be advised to carry out Exercise 1 first.

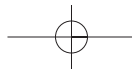
Each candidate will require:

one 50 cm³ burette and stand
one funnel
one 25 cm³ pipette
one pipette filler
two or more 250 cm³ conical flasks
one wash bottle
a plentiful supply of purified water (either distilled or de-ionised)
suitable eye protection.

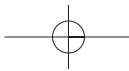


THERE ARE NO INSTRUCTIONS PRINTED ON THIS PAGE





THERE ARE NO INSTRUCTIONS PRINTED ON THIS PAGE



THERE ARE NO INSTRUCTIONS PRINTED ON THIS PAGE

