Centre Number	Candidate Number	Name

UNIVERSITY OF CAMBRIDGE INTERNATIONAL EXAMINATIONS
General Certificate of Education
Advanced Subsidiary Level and Advanced Level

CHEMISTRY **9701/31**

Paper 31 Advanced Practical Skills

For Examination from 2007

SPECIMEN PAPER

2 hours

Candidates answer on the Question Paper.
Additional Materials: As listed in Instructions to Supervisors.

READ THESE INSTRUCTIONS FIRST

Write your Centre number, candidate number and name on all the work you hand in.
Give details of the practical session and laboratory where appropriate, in the boxes provided.
Write in dark blue or black pen.
You may use a soft pencil for any diagrams, graphs or rough working.
Do not use staples, paper clips, highlighters, glue or correction fluid.

Answer **all** questions.
You are advised to show all working in calculations.
Use of a Data Booklet is unnecessary.

Qualitative Analysis Notes are printed on pages 9 and 10.

At the end of the examination, fasten all your work securely together.
The number of marks is given in brackets [ ] at the end of each question or part question.

Session
Laboratory

For Examiner's Use	
1	
2	
TOTAL	

This document consists of **10** printed pages.

For Examiner's Use

1 **FA 1** is 2.00 mol dm^{-3} sodium hydroxide, NaOH.
FA 2 is **approximately** 0.75 mol dm^{-3} sulphuric acid, H_2SO_4.

A student suggests that the concentration of the sulphuric acid can be determined by measuring the temperature of the solution as the acid is added in small amounts to a known volume of **FA 1** in a plastic cup.

The student proposes the following hypothesis.

As the acid is added to the alkali the temperature rise will be directly proportional to the volume of acid added until the end-point of the reaction is reached. Upon further addition of acid there will be a reduction in the temperature of the solution in the cup as the acid added is not reacting and is at a lower temperature than the solution in the plastic cup.

$$2NaOH(aq) + H_2SO_4(aq) \rightarrow Na_2SO_4(aq) + 2H_2O(l)$$

(a) Use the equation for the reaction to estimate the volume of approximately 0.75 mol dm^{-3} H_2SO_4 that will neutralise 25.0 cm^3 of 2.00 mol dm^{-3} NaOH.

[1]

(b) In the experiment you will add **FA2** from the burette to 25.0 cm^3 of **FA1** in a plastic cup. You will measure the temperature of the solution after each addition of acid. You will be required to plot a graph of the temperature before and after the end-point in order to determine the end-point accurately and consequently calculate the precise concentration of H_2SO_4 in **FA2**.

In order to obtain precise information about the end-point of the reaction, you will need to decide

how many additions of H_2SO_4 are to be made,
the volume of acid to be added each time.

number of additions of H_2SO_4 .....

volume of acid added each time ..... [2]

(c) In the space below you should record the results of your experiment, including
the initial temperature of **FA 1** in the plastic cup when no acid has been added,
the total volume of **FA 2** added at each stage of the experiment,
the temperature of the solution in the plastic cup after each addition of acid,
the temperature rise, ΔT.

[ΔT = temperature of the solution after each addition of acid – initial temperature of **FA 1**] [5]

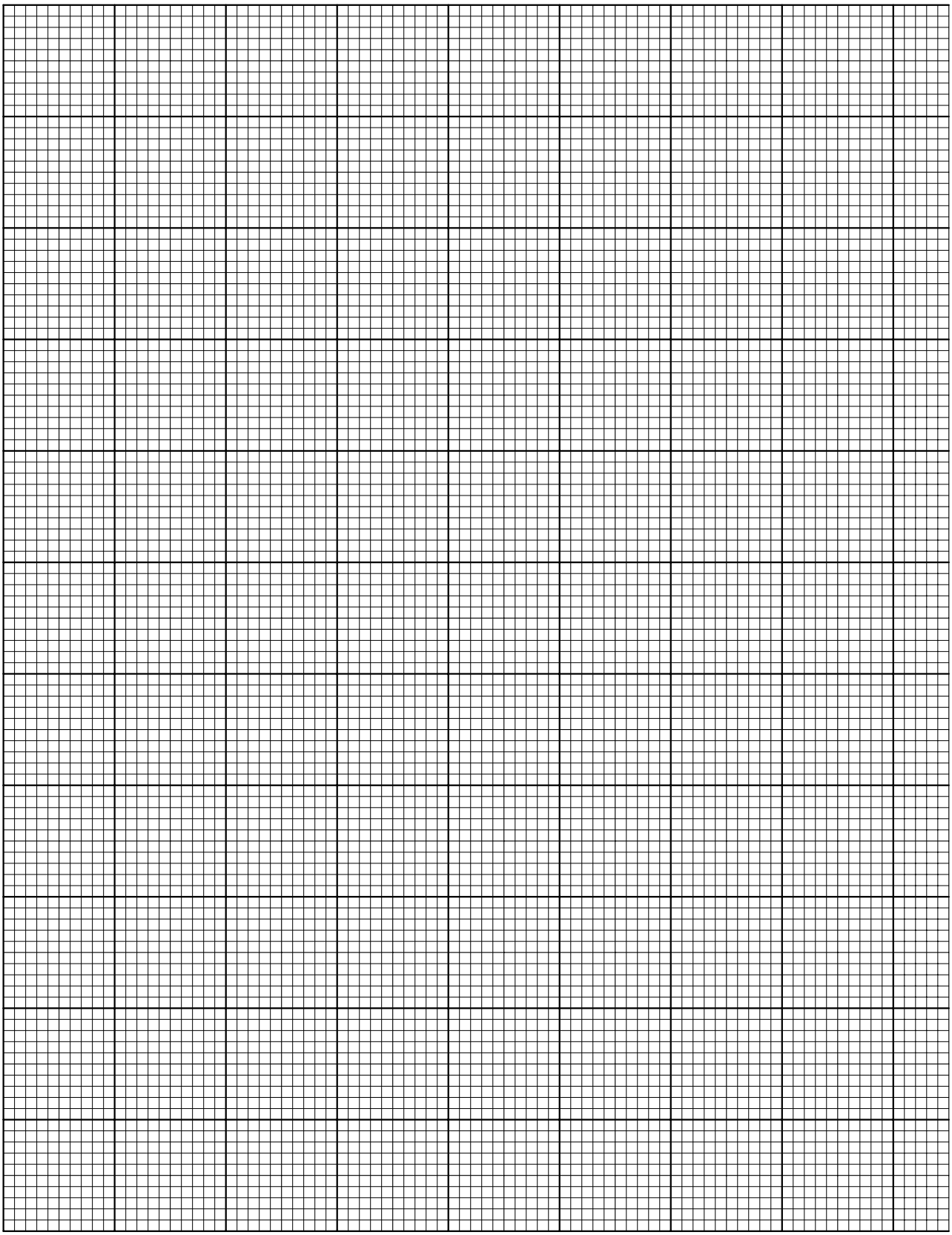
You must decide how best to record and present your measurements. [3]

Experimental procedure:
Fill the burette with **FA 2**.
Support the plastic cup in the 250 cm^3 beaker and pipette 25.0 cm^3 of **FA 1** into the plastic cup. Measure and record the steady temperature of **FA 1**.
Run into the cup the first volume of **FA2** you have selected, stir and record the highest temperature observed.
Immediately add a second volume of **FA2** from the burette and repeat the temperature measurement. Continue until you have added all of the planned volume of **FA2**.

Results

(d) Plot a graph of ΔT against volume of **FA 2** added.

Draw two distinct and intersecting lines, one for the increasing and one for the decreasing temperatures.



[4]

(e) The lines intersect at ..... cm^3 of H_2SO_4 [1]

(f) Use the results of your experiment to calculate the exact concentration of H_2SO_4. Show all your working and explain the steps in your calculation.

[2]

(g) Explain how the results of your experiment support or do not support each part of the hypothesis proposed by the student.

.....

.....

.....

.....

..... [3]

(h) From the points plotted and the shape of your graph suggest and explain a possible error in the measurements taken in this experiment.

.....

.....

.....

..... [2]

(i) From what you know about the accuracy of your apparatus, calculate the % error in the volume of acid added at the end-point.

[1]

(j) Another student puts forward the hypothesis that the heat energy produced in the reaction, rather than the temperature rise, is proportional to the volume of acid added.

How could the data collected in the experiment be converted into heat energy produced?
[4.3 J raises the temperature of 1 cm^3 of solution by 1 ^oC]

[1]

For Examiner's Use

2 Labels have become detached from two bottles of chemicals, each containing a white powder. One of these is believed to be barium iodide and the other a metal nitrate. A solution has been prepared from each of the solids and these solutions are labelled **FA 3** and **FA 4**.

(a) By selecting a suitable reagent from those listed in the reactions of anions on page 10, you should carry out a test to establish which of the solutions contains the iodide ion.

Record details of the test performed and the observations obtained in the test in the space below.

From this test, solution **FA** ……. contains the iodide ion. [2]

(b) By selecting another suitable reagent, carry out a test on the solution you have chosen, to confirm the presence of the iodide ion.

Record details of the test performed and the observations obtained in the test in the space below. State, with reasons, whether or not this confirms your choice above.

[2]

You are to perform the tests given in the table opposite on each of **FA 3** and **FA 4** to

(i) confirm the presence of the nitrate ion in the solution **not** chosen above,

(ii) identify and confirm the **cation** in each solution.

Record details of colour changes seen, the formation of any precipitate and the solubility of any such precipitate in an excess of the reagent added.
Where gases are released they should be identified by an appropriate test which you should describe in your observations.
You should indicate clearly at what stage in a test a change occurs.

Marks are **not** given for chemical equations.
No additional tests for ions present should be attempted.

[6]

test	observations with **FA3**	observations with **FA4**
(c) To 1 cm depth of solution in a boiling-tube, add aqueous sodium hydroxide drop-wise until it is in excess; then		
add a piece of aluminium foil and **warm** the tube. **Care** – solutions containing sodium hydroxide can "bump" when heated and eject the hot alkali from the tube.		
(d) To a 1cm depth of solution in a test-tube, add aqueous ammonia drop-wise until it is in excess.		
(e) To 3 cm depth of solution in a test-tube, add 2 cm depth of dilute sulphuric acid.		
(f) To 3 cm depth of solution in a test-tube, add 2 cm depth of aqueous potassium chromate(VI); then		
add 2 cm depth of dilute hydrochloric acid.		

For Examiner's Use

(g) Explain how your observations identify and confirm the presence of barium ions in the solution that contained the iodide ion.

.....

.....

..... [1]

The observations made with aqueous sodium hydroxide and aqueous ammonia should have indicated either of two possible cations in the other solution.

Identify these cations; explain the observations and explain how other tests carried out eliminate one of these cations.

.....

.....

.....

.....

..... [2]

(h) The test that indicates the presence of a nitrate would also give a positive result with nitrite. Suggest a test to distinguish between these two ions. Do **not** carry out this test.

.....

.....

..... [1]

Qualitative Analysis Notes

Key: [ppt. = precipitate.]

1 Reactions of aqueous cations

	reaction with	
	NaOH(aq)	NH_3(aq)
aluminium, Al^{3+}(aq)	white ppt. soluble in excess	white ppt. insoluble in excess
ammonium, NH_4^+(aq)	ammonia produced on heating	
barium, Ba^{2+}(aq)	no ppt. (if reagents are pure)	no ppt.
calcium, Ca^{2+}(aq)	white ppt. with high [Ca^{2+}(aq)]	no ppt.
chromium(III), Cr^{3+}(aq)	grey-green ppt. soluble in excess giving dark green solution	grey-green ppt. insoluble in excess
copper(II), Cu^{2+}(aq)	pale blue ppt. insoluble in excess	blue ppt. soluble in excess
iron(II), Fe^{2+}(aq)	green ppt. insoluble in excess	green ppt. insoluble in excess
iron(III), Fe^{3+}(aq)	red-brown ppt. insoluble in excess	red-brown ppt. insoluble in excess
lead(II), Pb^{2+}(aq)	white ppt. soluble in excess	white ppt. insoluble in excess
magnesium, Mg^{2+}(aq)	white ppt. insoluble in excess	white ppt. insoluble in excess
manganese(II), Mn^{2+}(aq)	off-white ppt. insoluble in excess	off-white ppt. insoluble in excess
zinc, Zn^{2+}(aq)	white ppt. soluble in excess	white ppt. soluble in excess

[Lead(II) ions can be distinguished from aluminium ions by the insolubility of lead(II) chloride.]

2 Reactions of anions

ion	*reaction*
carbonate, CO_3^{2-}	CO_2 liberated by dilute acids
chromate(VI), CrO_4^{2-} (aq)	yellow solution turns orange with H^+(aq); gives yellow ppt. with Ba^{2+}(aq); gives bright yellow ppt. with Pb^{2+}(aq)
chloride, Cl^- (aq)	gives white ppt. with Ag^+(aq) (soluble in NH_3(aq)); gives white ppt. with Pb^{2+}(aq)
bromide, Br^- (aq)	gives cream ppt. with Ag^+(aq) (partially soluble in NH_3(aq)); gives white ppt. with Pb^{2+}(aq)
iodide, I^- (aq)	gives yellow ppt. with Ag^+(aq) (insoluble in NH_3(aq)); gives yellow ppt. with Pb^{2+}(aq)
nitrate, NO_3^- (aq)	NH_3 liberated on heating with OH^-(aq) and Al foil
nitrite, NO_2^- (aq)	NH_3 liberated on heating with OH^-(aq) and Al foil, NO liberated by dilute acids (colourless NO → (pale) brown NO_2 in air)
sulphate, SO_4^{2-} (aq)	gives white ppt. with Ba^{2+}(aq) or with Pb^{2+}(aq) (insoluble in excess dilute strong acids)
sulphite, SO_3^{2-} (aq)	SO_2 liberated with dilute acids; gives white ppt. with Ba^{2+}(aq) (soluble in excess dilute strong acids)

3 Tests for gases

gas	*test and test result*
ammonia, NH_3	turns damp red litmus paper blue
carbon dioxide, CO_2	gives a white ppt. with limewater (ppt. dissolves with excess CO_2)
chlorine, Cl_2	bleaches damp litmus paper
hydrogen, H_2	"pops" with a lighted splint
oxygen, O_2	relights a glowing splint
sulphur dioxide, SO_2	turns potassium dichromate(VI) (aq) from orange to green

UNIVERSITY OF CAMBRIDGE INTERNATIONAL EXAMINATIONS
General Certificate of Education
Advanced Subsidiary Level and Advanced Level

CHEMISTRY **9701/31**

Paper 31 Practical Test

For Examination from 2007

SPECIMEN CONFIDENTIAL INSTRUCTIONS

Great care should be taken to ensure that any information given does not reach the candidates either directly or indirectly.

READ THESE INSTRUCTIONS FIRST

1 **Access to the examination paper is not permitted before the examination.**

Supervisors are asked to carry out any confirmatory tests included in these instructions to ensure the materials supplied are appropriate.

The 'General Apparatus' requirements and the 'Particular Requirements' are printed separately. It is *especially important* that the details on page 4 are kept secure.

2 Supervisors are advised to remind candidates that **all** substances in the examination should be treated with caution. Only those tests described in the question paper should be attempted. Please also see under 'General Apparatus' on the use of pipette fillers and safety goggles.

In accordance with COSHH (Control of Substances Hazardous to Health) Regulations, operative in the UK, a hazard appraisal of the examination has been carried out.

Attention is drawn, in particular, to certain materials used in the examination. The following codes are used where relevant.

C = corrosive substance

F = highly flammable substance

H = harmful or irritating substance

O = oxidising substance

T = toxic substance

The Supervisor's attention is drawn to the form on page 7 which must be completed and returned with the scripts.

If you have any problems or queries regarding these instructions, please contact CIE
by e-mail: International@cie.org.uk
by phone: +44 1223 553554
by fax: +44 1223 553558
stating the Centre number, the nature of the query and the syllabus number quoted above.

[Turn over

Safety

The attention of Supervisors is drawn to any local regulations relating to safety and first-aid.
'Hazard Data Sheets', relating to materials used in this examination, should be available from your chemical supplier.

General Apparatus and Materials

1 In addition to the fittings and reagents ordinarily contained in a chemical laboratory, the apparatus and materials specified below will be necessary.

2 Pipette fillers and safety goggles should be used where necessary.

3 **It is assumed that common bench reagents and reagents/materials for testing gases listed in the syllabus are available.**

[H] 2 mol dm^{-3} hydrochloric acid
[C] 2 mol dm^{-3} nitric acid
[H] 1 mol dm^{-3} sulphuric acid

[T] 0.1 mol dm^{-3} potassium chromate(VI)
wooden splints
red and blue litmus indicator papers

[C] 2 mol dm^{-3} sodium hydroxide
[H] 2 mol dm^{-3} aqueous ammonia

limewater (a saturated solution of calcium hydroxide) and the associated apparatus

For each candidate

1 x plastic cup (expanded polystyrene/foamed plastic)
1 x 250 cm^3 beaker (to support the plastic cup)
1 x 50 cm^3 burette
1 x stand and burette clamp
1 x small funnel for filling burette
1 x 25 cm^3 pipette
1 x pipette filler
1 x thermometer, -10 °C to 110 °C by 1 °C
6 test-tubes
1 boiling-tube
1 x test-tube rack
1 x Bunsen burner
1 x heat proof mat
2 x teat/squeeze pipettes
paper towels

Particular Requirements

1 As a possible aid to maintaining security, the descriptions of the particular chemicals required are given under two headings:

(a) overall specifications are given on page 3;

(b) the actual identities are given on page 4.

2 Materials with an **FA** code number should be so labelled for the candidates' benefit, **without** the identities being included on the label - where appropriate, the identity of an **FA** coded chemical is given in the question paper itself.

Chemicals Required

1 The chemicals required per question are described in general terms below.

2 Where quantities are specified for each candidate, they are sufficient for the experiments described in the question paper to be completed.

In preparing materials, the bulk quantity for each substance should be increased by 25% as spare material should be available to cover accidental loss.

More material may be supplied if requested by candidates, without penalty.

3 The additional qualitative analysis reagents needed for Question 2 are identified on page 4.

4 **For Question 1**

[C]	Solution **FA 1**	50 cm^3
	FA 1 *should be supplied in stoppered bottles or in beakers covered with "cling-film" or "gladwrap".*	
[H]	Solution **FA 2**	70 cm^3

For Question 2

[H]	Solution **FA 3**	30 cm^3
[T]	Solution **FA 4**	30 cm^3

Detailed Identities of Chemicals Required

1 It is *especially important* that great care is taken that the confidential information given below does not reach the candidates either directly or indirectly.

2 The identities of the chemicals with an **FA** code number are as follows.

Question 1

[C] **FA 1** is 2.00 mol dm^{-3} sodium hydroxide, NaOH, containing 80.00 g dm^{-3} of NaOH.
This solution should be kept covered before and after issue to candidates to prevent absorption of carbon dioxide from the atmosphere.

[H] **FA 2** is 0.75 mol dm^{-3} sulphuric acid, H_2SO_4. Prepare this solution by carefully adding 41.0 cm^3 of concentrated (95%) sulphuric acid to distilled water and diluting the resulting solution to 1 dm^3.

The concentration of **FA 1** *should be checked by titrating a 25.0 cm^3 portion of* **FA 2** *against* **FA 1**. *Adjust the concentration of* **FA 2** to give a titre of 18.75 ± 0.20 cm^3.
It is essential that the FA 1 solution is kept securely stoppered until the time of the examination to prevent absorption of carbon dioxide.
It should be issued to candidates just before the start of the examination – see p3.

Solutions FA 1 and FA 2 should be prepared at least 24 hours before the examination and allowed to stand in the laboratory to equalise their temperature.

[H] **FA 3** is 0.1 mol dm^{-3} aluminium nitrate containing 37.5 g of $Al(NO_3)_3.9H_2O$ in each dm^3 of solution.

[T] **FA 4** contains 0.1 mol dm^{-3} barium chloride **and** 0.1 mol dm^{-3} potassium iodide. Dissolve 24.5 g of $BaCl_2.2H_2O$ **and** 16.6 g of KI in distilled water and make up to 1 dm^3.

3 In addition to those listed on page 2, the qualitative analysis reagents specifically required are set out below. If necessary, they may be made available from a communal supply: however, the attention of the Invigilators should be drawn to the fact that such an arrangement may enhance the opportunity for malpractice between candidates

aluminium foil

[C] 0.05 mol dm^{-3} silver nitrate, $AgNO_3$, 8.5 g dm^{-3}

[T] 0.10 mol dm^{-3} lead(II) nitrate, $Pb(NO_3)_2$, 33.0 g dm^{-3}

COLOUR BLINDNESS

With regard to colour-blindness – a minor handicap, relatively common in males – it is permissible to advise candidates who request assistance on colours of, for example precipitates and solutions (especially titration end-points). Please include with the scripts a note of the index numbers of such candidates.

Experience suggests that candidates who are red/green colour-blind – the most common form – do not generally have significant difficulty. Reporting such cases with the scripts removes the need for a 'Special Consideration' application for this handicap.

Accuracy of Solutions

1 **All the solutions are to be labelled as shown and they should be bulked and mixed thoroughly before use to ensure uniformity.**

2 **Every effort should be made to keep the concentrations accurate to within one part in two hundred of those specified.**

3 If the concentrations differ slightly from those specified, the Examiners will make the necessary allowance. They should be informed of the exact concentrations.

4 It should also be noted that descriptions of solutions given in the question paper may not correspond exactly with the specification in these Instructions. **The candidates must assume the descriptions given in the question paper**.

5 In view of the difficulty of the preparation of large quantities of solution of uniform concentration, it is recommended that the maximum number of candidates per group be 30 and that separate supplies of solutions be prepared for each group.

Responsibilities of the Supervisor

(i) The Supervisor, or other competent chemist **must carry out the experiments in question 1** and complete the table of readings on a spare copy of the question paper which should be labelled 'Supervisor's Results'.

This should be done for:
each session held and each laboratory used in that session, and
each set of solutions supplied.

N.B. The question paper cover requests the candidate to fill in details of the examination session and the laboratory used for the examination.

It is essential that each packet of scripts contains a copy of the applicable Supervisor's Results as the candidates' work cannot be assessed accurately without such information.

(ii) The Supervisor must complete the Report Form on page 7 to show which candidates attended each session. If all candidates took the examination in one session, please indicate this on the Report Form. A copy of the Report Form must accompany each copy of the Supervisor's Results in order for the candidates' work to be assessed accurately.

(iii) The Supervisor must give details on page 8 of any particular difficulties experienced by a candidate, especially if the Examiner would be unable to discover this from the written answers.

Each envelope returned to Cambridge must contain the following items.

1 The scripts of those candidates specified on the bar code label provided.

2 A copy of the Supervisor's Report relevant to the candidates in **1**.

3 A copy of the Report Form, including details of any difficulties experienced by candidates (see pages 7 and 8).

4 The Attendance Register.

5 **A Seating Plan for each session/laboratory**.

Failure to provide appropriate documentation in each envelope may cause candidates to be penalised.

REPORT FORM

This form must be completed and sent to the Examiner in the envelope with the scripts.

Centre Number …… Name of Centre ……

1 Supervisor's Results

Please submit details of the readings obtained in **Question 1** on a spare copy of the question paper clearly marked 'Supervisor's Results' **and showing the Centre number and appropriate session/laboratory number**.

2 The index numbers of candidates attending each session were:

First Session	*Second Session*

3 The Supervisor is required to give details overleaf of any difficulties experienced by particular candidates, giving names and index numbers. These should include reference to:

(a) any general difficulties encountered in making preparation;

(b) difficulties due to faulty apparatus or materials;

(c) accidents with apparatus or materials;

(d) assistance with respect to colour-blindness.

Other cases of hardship, e.g. illness, temporary disability, should be reported direct to CIE on the normal 'Application for Special Consideration' form.

4 A plan of work benches, giving details by index numbers of the places occupied by the candidates for each experiment for each session, must be enclosed with the scripts.

Report on any difficulties experienced by candidates.